DOCTORAL THESIS

Development of Sustainable Polypropylene Based Composites

Abrar Hussain

TALLINNA TEHNIKAÜLIKOOL
TALLINN UNIVERSITY OF TECHNOLOGY
TALLINN 2023

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64/2023

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This dissertation was accepted for the defence of the degree 08/11/2023

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Declaration:
Hereby I declare that this doctoral thesis, my original investigation, and achievement, submitted for the doctoral degree at Tallinn University of Technology has not been submitted for doctoral or equivalent academic degree.

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ISSN 2585-6898 (publication)
ISBN 978-9916-80-077-5 (publication)
ISSN 2585-6901 (PDF)
ISBN 978-9916-80-078-2 (PDF)
Printed by Koopia Niini & Rauam

TALLINNA TEHNIKAÜLIKOOL
DOKTORITÖÖ
64/2023

Polüpropeeni baasil jätkusuutlike komposiitide arendus

ABRAR HUSSAIN

TALTECH KIRJASTUS

Contents

List of publications

The list of author’s publications based on which the thesis has been prepared:

I **Abrar Hussain**, Kamboj, N., Podgursky, V., Antonov, M. and Goljandin, D., 2021. Circular economy approach to recycling technologies of postconsumer textile waste in Estonia: a review. Proceedings of the Estonian Academy of Sciences 70 (1), 80–90, doi.org/10.3176/proc.2021.1.07. (1.07 IF)

II **Abrar Hussain,** Podgursky, V., Viljus, M., Ahmad, T., Awan, M.R, 2022, The role of paradigms and technical strategies for implementation of the circular economy in polymer and composites recycling industries. Advanced Industrial and Engineering Polymer Research, 71 (2), 186–193, doi.org/10.1016/j.aiepr.2022.10.001. (16 IF)

III **Abrar Hussain,** Podgursky V, Goljandin D., Antonov M., Viljus M., Krasnou I., 2023. Sustainable fabrication of polypropylene-postconsumer cotton composite materials: Circularity, characterization, mechanical testing and tribology. Materials Today Sustainability, 22 (1), 100344, doi.org/10.1016/j.mtsust.2023.100344. (8 IF)

IV **Abrar Hussain**, Podgursky V., Goljandin D., Antonov, M., Sergejev F., Krasnou I. Circular Production, Designing and Mechanical Testing of Polypropylene-Based Reinforced Composite Materials: Statistical Analysis for Potential Automotive and Nuclear Applications. Polymers, 2023, 15, 3410. doi.org/10.3390/polym15163410. (5 IF)

V **Abrar Hussain,** Podgursky V., Goljandin D., Antonov M., 2023. Industrial approach to circularity of polymer composites: Processing, characterization, mechanical testing and wear regression. Journal of Reinforced Plastics and Composites, 0 (0), 1–17, DOI: 10.1177/07316844231164563. (4 IF)

VI **Abrar Hussain**, Goljandin, D., Podgursky, V., Abbas, M. M., & Krasnou, I. (2023). Experimental mechanics analysis of recycled polypropylene-cotton composites for commercial applications. Advanced Industrial and Engineering Polymer Research, 6(3), 226–238. doi.org/10.1016/j.aiepr.2022.11.001. (16 IF)

Author's contribution to the publications

Contribution to the papers in this thesis are:

I First and corresponding author, tribological tests, surface evaluation using mechanical and optical profilometer, processing of data, calculations and text writing.

II First and corresponding author, models development, tribological tests, mechanical evaluations, SEM characterization, manuscript writing and article drafting.

III First and corresponding author, composite fabrication, SEM characterization, tensile, bending and impact analysis, abrasion testing, erosion investigation, recycling-circularity quantitative relationship, comparison of injection and compression molding techniques, manuscript writing and article drafting.

IV First and corresponding author, composite fabrication, SEM characterization, tensile, bending and impact analysis, abrasion testing, erosion investigation, recycling-circularity quantitative relationship, comparison of mechanical, surface, thermal, tribological properties. Implementation of ANOVA analysis for prediction of mechanical properties of composites, manuscript writing and article drafting.

V First and corresponding author, composite processing, SEM characterization, mechanical testing, tribological investigations, circularity, ANOVA statistical analysis, wear regression, manuscript writing and article drafting.

VI First and corresponding author, composite processing, SEM characterization, mechanical testing, experimental dynamic analysis of composite materials, development of theoretical models for ductile and brittle materials evaluations, circularity and fracture relationship for commercial applications.

Introduction

The global demand for polymeric and composite materials rises permanently [1]. Natural and synthetic polymers like cotton, silk, wool, polypropylene, synthetic and polyethylene terephthalate polyesters are commercial materials utilized extensively for automotive, medical, electronic, nuclear, aerospace applications, as well as in food and fashion industries [2]. The utilization of natural and synthetic polymeric materials in different applications produces huge waste. The pre-consumer, post-consumer, end-waste and end-of-life waste exist with purity of 99%, 85-98%, 60-85% and < 60% of valuable materials, respectively. Within the concept of circular economy, the transformation of waste into green and sustainable products can be carried out. The waste can be processed using primary, secondary, tertiary recycling technology and incineration techniques [3].

The concept of circularity was introduced for the first time in the European Union in 1962. The circular economy is a transition from open into closed-loop product manufacturing, i.e. within the framework of this concept, the waste re-use can be maximized due to recycling of waste and manufacturing of new products. It allows decreasing the negative influence on the nature. The concept includes costing, waste analysis, selection of recycling and fabrication technique and quality control. Costing and waste analysis assured the initiation of a project. Waste analysis evaluates the suitability of valuable materials for processing. The selection of manufacturing processes and quality control manage the performance of fabricated products for commercial applications.

The suitable selection of recycling technique increases sustainability of fabricated products. The machinery parts (cutting blades, shredding tools, *etc.*) of the recycling industry are mostly made using steel. To improve the performance and lifetime of the machinery parts can be coated with hard coating. Investigation of the mechanical and tribological properties of machinery parts and materials that is used in recycling of waste is important aspect to evaluate the quality and performance of recycling [4]. The hard coatings like diamond-like carbon, carbide (SiC, *etc.*), nitride (TiCN, TiAlN, *etc.*), ceramic oxides (Al_2O_3, ZrO_2) and boride are introduced for tool surface modification. The surface modification enhanced the life of machinery parts and the performance of manufactured products [5].

The natural and synthetic polymers waste contains several valuable materials, which can be re-used to produce composites. The pure polypropylene (PP) widely used as a matrix material in composites. The PP provides reasonable wettability and interfacial adhesion with different filler materials and resistance towards environmental impacts. The extrusion, compression and injection molding are commercial techniques to manufacture composite materials. The thermal, chemical, tensile, bending, abrasion, erosion, fatigue, creep tests, surface analysis, *etc.* of fabricated composites certified the quality and performance for customer satisfaction [6].

The aim of the thesis is manufacturing and investigation of the mechanical, tribological, surface and thermal properties of post-consumer cotton fibers (PCCF), PP-post-consumer synthetic polyester fibers (PCPESF) and PP-post-consumer polyethylene terephthalate fibers (PCPETF) reinforced PP-based composites.

The novelties of the present study are as follows:

I. Investigation of the tribological properties of different tribosystems, including PCCF waste - steel, PCCF waste - Al_2O_3, PCCF waste - WC-Co, PCCF waste -ZrO_2, PCCF waste - TiCN and PCCF waste - TiAlN.

II. The manufacturing of PP-PCCF, PP-PCPESF and PP-PCPETF composites. The PCCF and PCPESF were produced from Estonian military woven fabric T-shirts and PCPETF was produced from PET beverage bottles.

III. Comparative study of PP based natural and synthetic fibers reinforced composites. Comparison of thermal, mechanical, tribological properties, surface morphology and roughness.

Abbreviations

4IR	Fourth industrial revolution
5IR	Fifth Industrial revolution
AISI	American Iron and Steel Institute
Al_2O_3	Aluminum dioxide or alumina
AlN	Aluminum nitride
ANN	Artificial neural network
ANOVA	Analysis of variance
ASTM	American society for testing of materials
BEA	Break even analysis
BEP	Breakeven Point
CE	Circular economy
COF	Coefficient of friction
CrC	Chromium carbide
CRI	Cutting resistance index
CT	Cotton
DLC	Diamond-like carbon
DSC	Differential scanning calorimetry
EOLW	End of life waste
EW	End waste
FC	Fixed cost
FRC	Fiber-reinforcement composites
G	Weight of sand
GSM	Grams per square meter
GSM	Grams per square meter weight
HSLW	High strength low weigh
HV	Hardness of Vickers Scale
ISO	International Organization for Standardization
PC	Poly carbonate
PCCF	Post-consumer cotton fibers
PCDT	Poly-1.4 c yclohexyl-di-methylene terephtalate
PCPESF	Post-consumer polyesters fibers
PCPETF	Post-consumer polyethylene terephthalate fibers
PE	Polyethylene
PES	Polyesters
PET	Polyethylene terephthalate
PP	Polypropylene
PR-CW	Pre-consumer waste
PU	Polyurethanes
PVD	Physical vapor deposition
SEM	Scanning electron microscope

SiC	Silicon carbide
SNK	Student-Newman-Keuls
TC	Total cost
TEM	Transmission electron microscope
TGA	Thermogravimetric analysis
TiAlN	Titanium aluminum nitride
TiCN	Titanium carbo nitride
TiN	Titanium nitride
VC	Variable cost
WC	Tungsten carbide
ZrO_2	Zirconium dioxide or zirconia

Symbols

σ_p	Ploughing stress
σ_s	Shear stress
ΔH_f	Heat of fusion of composite materials
ΔH_f^o	Heat of fusion of 100% reference material
Δm	Change in mass
A	Area
L	Applied load
l	length
M	Specific weight loss
P	Ploughing force
R_a	Average surface roughness
R_p	Maximum profile peak height
rpm	Revolution per minute
R_q	Root mean square roughness
R_t	Maximum height of the profile
R_z	Average maximum height of the profile
t	Thickness
T/m	Twsit per meter
W	Abrasive wear
wt.%	Weight percentage
Xc	Amount of fiber's fraction
E	Erosive wear rate
v	Shear of sand
v	Volumetric wear loss
μ	Coefficient of friction
ρ	Density

1 Review of literature

1.1 Polymer waste and concept of circularity

The world's requirement for the reuse of textile products increases. The notion polymer textile used in the following text means the textiles made as from natural, as well as from synthetic fibers [7]. The EU Directive 2018/851 of the European Parliament and of the Council of 30 May 2018 amending Directive 2008/98/EC on waste determinates that any textile waste should be recycled beginning from the year 2024 [8]. In Estonia, there is a program of the textile recycling, for instance, from 4021 tons of textile waste collected in 2020, 840 tons were recycled [9]. Usually, 65-70% of the polymer textile materials are manufactured from synthetic fibers. The remaining 35-30% of textile products are produced from natural fibers. The utilization of these synthetic and natural textile materials creates huge waste. Internationally, 55% of polymer wastes end up in a landfill, 20% are used for recycling and 25% for energy production [10].

The pre-consumer waste (PR-CW) and post-consumer waste (PCW) are two primary types of waste [11]. The pre-consumer waste is generated due to operational problems during manufacturing of products. Post-consumer waste is produced due to the use of manufactured products in household, automotive, agricultural, electronic, nuclear and construction commercial applications. The end-of-life waste (EOLW) is distorted, damaged and hazardous in nature [12].

Table 1.1. *Industrial waste and recycling technology [Publication I].*

Polymer waste type	**Polymer waste material**	**Separation and sorting operation**	**Polymer recycling type**	**Output product**
PR-CW/PCW/EW/EOLW	PES, CT, PE, PET, PC	Manual and automatic	Primary	Relevant recycled product
EW	Defective and distorted wastes	Mostly manual	Primary and secondary	Material retrieval and recycled product
EW/PCW	CT, PES, PE, PET, silk *etc.*	Manual	Secondary and tertiary	Materials retrieval
PCW/EOLW	Hazardous wastes (all possible natural and synthetic polymers)	Manual	Tertiary and Quaternary	Low quality recycled product
EOLW	Hazardous and contaminated wastes	Manual	Quaternary and Biodegradable	Industrial Energy production and landfill

Pre- and post-consumer wastes are mostly recycled directly due to usually higher purity > 85% of valuable material (see Table 1.1). Similarly, end-waste materials contain impurity more than 15%. Mechanical separation is usually used before recycling to increase the purity. Purification techniques in presence of acids, mixings, additives and degassing agents are used for removal of impurities from end-waste materials. However, end-of life wastes are incinerated, degraded biologically and dumped in land. The closed loop manufacturing shown in Figure 1.1 is a modern approach of textile recycling.

Figure 1.1. *Closed loop manufacturing [adapted from Publication I].*

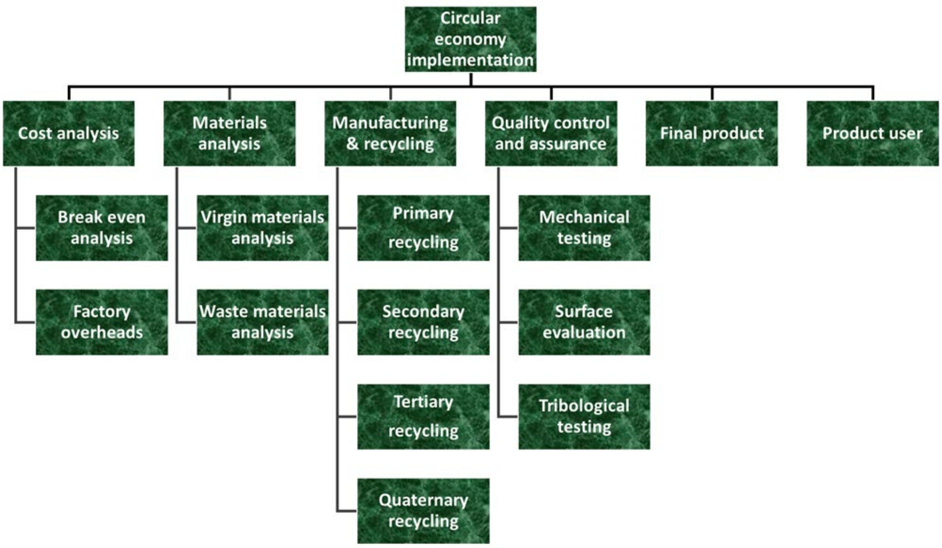


Figure 1.2. *The circularity paradigm [adapted from Publication II].*

The circularity paradigm and technical strategies are fundamental tools for sustainable and green technological development, see Figure 1.2. Cost studies, polymer waste analysis, recycling types, quality and performance testing have been introduced to reduce the use of natural resources [13].

The concept of circular economy (CE) was first proposed in the European Union in 1962. CE is a set of innovative technical management, manufacturing and testing strategies. The strategies transform open-system manufacturing into restorative, regenerative and closed-system manufacturing, see Figures 1.1 and 1.2 [14,15].

Break even analysis (BEA) in terms of total, fixed, variable cost and factory overhead is an industrial costing tool. Variable cost constitutes materials cost, industrial machinery cost, packaging cost and commercial royalties, see Figure 1.3. Similarly, fixed cost consists of relative depreciation, factory rents and labor salaries. Commercially, these costs are categorized into direct and indirect costs. The pricing of manufactured units (products) governs and regulates the survival of an organization. Figure. 1.3 shows the BEA model in terms of revenue, loss, profit and sold units. The BEA formulation can be expressed as:

$$Total\ Cost\ (TC) = Fixed\ Cost\ (FC) + Variable\ Cost\ (VC) \quad (1)$$

TC also assists in evaluating the initiation of an industrial project. Similarly, FC and VC represent the initial amount of money required for a project in industry. The point at which loss equals profit is known as breakeven point (BEP). Therefore, BEP also helps to initiate, expand, lower and narrow the business during the implementation of project [16,17]. Additionally, Z is the initial amount of revenue required to produce Y number of units of products without risk of loss.

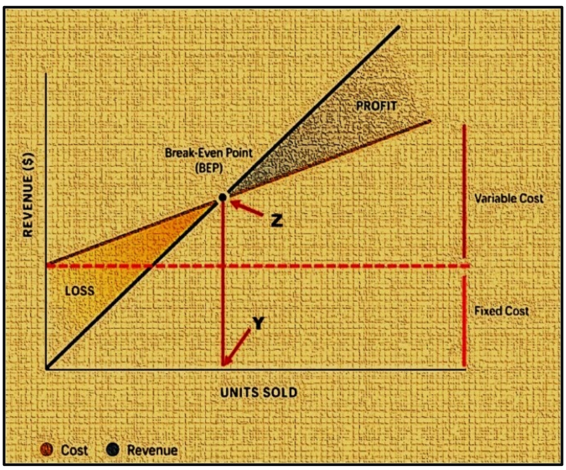


Figure 1.3. *BEA Model [Publication II].*

The remanufacturing of textile waste into sustainable polymer products is termed as recycling. However, the recycling of textile waste is very complex [18,19]. Generally, the recycling of polymer (textile) waste is categorized into primary, secondary, tertiary, quaternary and biodegradable processing. Initially, the textile wastes are separated and sorted. After separation, the grinding of waste is performed at the desired size of fibers [20,21]. The polymeric waste is shredded using different metal blades and cutters.

The cutting is used to convert waste into small pieces. Hard metallic surface (cutting tool) interacts with soft polymer surface. Shearing and ploughing mechanisms are involved in cutting of waste. The shredding mechanism is associated with rotary drums and cylinders [22]. The rotary drums and cylinders are divided into different sections for collection of desired, smaller and oversize fiber materials [23]. Mostly, mechanical shredding is used for the transformation of waste into fine fibers. The cutting speed and applied torque are controlled for optimum outcomes [24]. All types of waste materials are shredded to increase the purity and performance of recycled products. After size reduction, a reasonable recycling technique is selected for further processing of waste. Usually, selection of recycling technique relies on purity of waste and application of the fabricated product [25].

Primary [26] and secondary [27] recycling techniques are mostly utilized for processing of defective samples, scrap and low impurity waste [27]. The tertiary and quaternary methods are introduced for recycling of highly damaged, distorted, impure and hazardous polymer materials [28-30] (Figure 1.4 and Table 1.1).

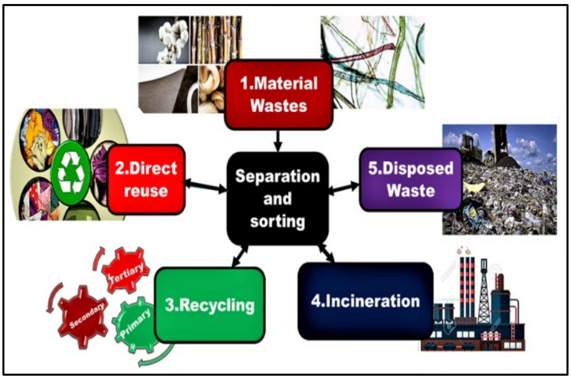


Figure 1.4. *The polymer waste management model [Publication I].*

Generally, the terms like pre-consumer waste, post-consumer waste, end waste (EW) and end of life waste are used for different waste in recycling industries. PR-CW are produced during manufacturing of polymer products. The PCW mostly derived from end-user of polymer products after service life. The EW contained highest level of impurity in distorted conditions. Generally, secondary treatments are required to enhance the purity of valuable polymeric material. However, EOLW lost its utility during service life and mostly used for energy production, incineration and landfills [31-34]. The highly contaminated and impure textile wastes are used for energy production [35] and ground landfills. The enhancement of quality and performance of recycled products are complex functions in nature. The performance attributes directly rely on testing of polymer waste, separation, selection of recycling technique, finishing and commercial use of recycling product. The polymers-machinery parts interactions and mechanical testing during recycling have a fundamental role to check the usability of products [36,37].

The polymer waste-metallic machinery parts interactions initiate wear during recycling. The damage of metallic and polymer surfaces appeared in the form of erosion, abrasion, surface fatigue, damage and corrosion. The wear of polymers, machinery parts and cutting tools can be controlled using hard coatings. Diamond-like carbon (DLC) coatings offer excellent resistance to erosion and abrasion. DLC-based materials are mostly used for cutting tools, molding dies and grinding tools. Nitride coatings like TiCN, TiAlN, TiN, *etc.* are introduced for high-speed cuttings at extreme temperatures. The carbide materials (WC, CrC, SiC, *etc.*) as a thin film coating impart low residual stresses, porosity, higher bond strength, reasonable corrosion resistance and good wear resistance. The oxide ceramics (Al_2O_3, ZrO_2, *etc.*) possess high temperature resistance and strength.

The first, second and third generations of industrial revolution are considered as a conventional mode of the manufacturing [Publication II and V]. Fourth industrial revolution (4IR) is a modern industrial approach of manufacturing. Circular economy and fourth generation of industrial revolution provide tools to optimize various recycling techniques. The combination of CE and 4IR can enhance recycled product quality. The polymer waste can be directly reused, recycled, incinerated or disposed after automatic separation and sorting steps. Recently, EU has introduced the utilization of artificial intelligence, robot and smart machine working to achieve the goals of sustainability, green manufacturing and circular production. This is known as fifth industrial revolution or industry 5.0 (5IR). The combination of robot-human centric collaboration enhanced the implementation of concept of circularity in manufacturing industries.

1.2 Natural and synthetic polymers

Polymers are categorized into natural and synthetic materials. Natural polymers are mainly made up of cellulose (core), hemicellulose and lignin [38,39]. Fundamentally, cellulose's contents in a provided fiber govern the stiffness and strength [40].

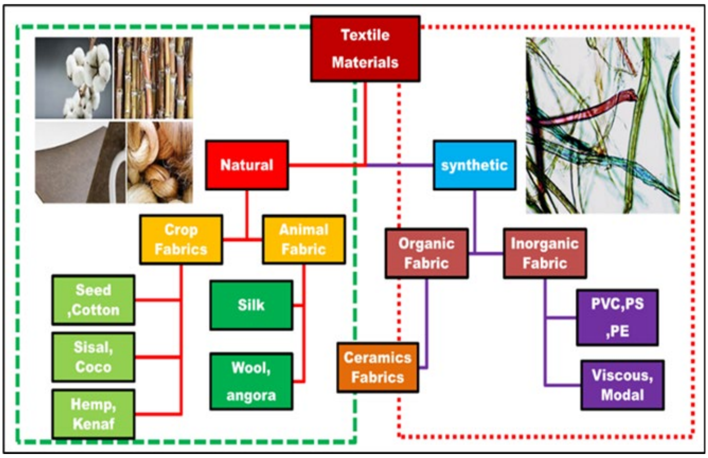


Figure 1.5. *Types of polymers [Publication I].*

Cotton, hemp, kenaf, sisal, jute, wool, kapok, *etc.* are widely used natural polymers. Similarly, PES, PET, PP, PC, rubber and PU *etc.* are synthetic polymers utilized in textile, packaging, automotive and construction industries. The types of polymers are shown in Figure 1.5 [Publication I].

Fiber cultivation, adherence to constituents, surface conditions and fibber's age are main characteristics that affect its functionality [41]. The hemicellulose's amount controls the moisture absorption, thermal and degradation properties [42]. Moreover, lignin protects fiber from thermal and optical shocks [43]. Among the constituents (core, hemicellulose and lignin) of cotton's fabric structure, the fabric core exists as a fundamental component for determination of mechanical properties [44]. The nature of core materials, angle of microfibrils and the content of core material are also considered important factors for mechanical properties. The core is hemicellulose embedded with lignin [45]. This type of cotton fabric structure produces porosity. Naturally, higher amounts of cellulose material and smaller microfibril angles are considered suitable for excellent mechanical properties. However, porosity absorbed impurities and caused various surface defects [46].

Synthetic polymers are artificial materials derived from petroleum products. Synthetic fibers have better chemical resistance, mechanical (tensile, flexural, bending, *etc.*) and tribological (erosion, abrasion, fatigue, *etc.*) properties as compared to natural fabric materials [47,48]. Synthetic polyester, PP and PET are unsaturated hydrocarbons. Flexibility in nature, recyclability, resistance to environmental impacts and toughness make these polymers commercially versatile materials for various industrial applications [49]. Mostly, PP is used as a matrix material in composite fabrication due to properties like unbreaking ability, high strength, low weight, better wettability with fibers, good hardness and resistance to moisture. Higher abundance, ease of recycling, lowest use of additives and nontoxicity made polyesters (especially synthetic and PET) a potential candidate for synthesis of fiber-reinforcement composites (FRC) [50], see Figure 1.6 [Publication I].

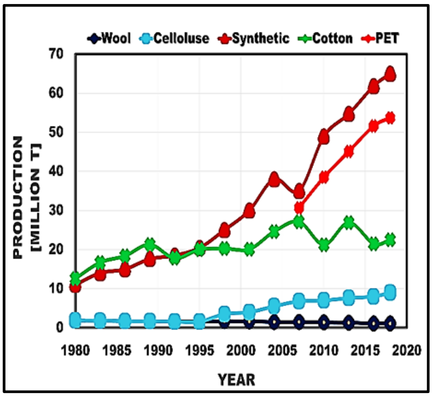


Figure 1.6. *Commercial annual production of polymeric materials [Publication I].*

1.3 Types of composites

The polymer waste can be used as reinforcement to manufacture composites. The reinforcement can increase the performance of composites. The fiber reinforced composites are formed with two main phases, i.e., continuous and dispersed. The continuous phase is known as matrix phase. The matrix phase may be polymer, metal or ceramic. It binds reinforcement (or dispersed phase) together, transfer loads, provide prominent shape to bulk and resistance toward various impacts. Polymer matrix are used for high performance automotive, construction and aerospace applications. The styrene, polyesters, epoxies, phenolics, polyimides, epoxy resins, polybutadiene are common examples of advanced thermosets matrices. The common examples of thermoplastic matrices are PP, PC, PET, polyetheretherketone and liquid crystal polymers. The reinforcement phase increases the stiffness, strength, hardness, impact behavior, environmental resistance, fatigue strength and electrical properties. The reinforcement phase is continuous and discontinuous in nature. The continuous reinforcement involves textiles and unidirectional filaments. However, the discontinuous fiber loadings include short fibers and particles. The addition of short fibers improves the performance of composites for lightweight applications. The short-fiber reinforced composites are cheaper and easier to produce. The mechanical properties of these composites rely on fiber length and distribution. The elastic modulus of composites improved with enhancement of fiber fractions and orientation. The ease of processing and ability to recycle lower the manufacturing cost of short fiber reinforced composites [51].

1.4 Recycling process steps

The process of recycling includes different steps. The first step is a collection of waste. In the EU, polymer waste is collected from sources like packaging (59%), electronics (8%), construction (5%), automotive (5%), agriculture (5%), household (5%) and others (14%) [52]. Various types of waste are stored and transported to warehouses of recycling industries [53]. After collection, identification of polymer wastes is performed using manual (wastes in large amounts), near infrared (for dark colored plastics), electrostatic (smart and conductive polymers), float sink (separation based upon densities) and selective dissolution sorting techniques [54,55]. Identification technologies reduce contamination, maintain waste material integrity, remove problematic polymers and control the properties of recycled products [56]. After separation, the polymeric waste is milled into fine fibers. The fine fibers are separated using sieve classification. The sieve classification screening distributes fibers into various sizes. Normally, these fibers are utilized as reinforcement in the fabrication of composites [57]

The fiber reinforced composites are mostly prepared using extrusion, injection and compression molding techniques. The extrusion technique is applied for preparation of composites in the case of short fibers. In polymer extrusion, the mixture of matrix and reinforced materials passes through a heated cylinder. The obtained polymeric materials are pelletized. Additives, mixtures and binder materials are also used to increase wettability, mechanical properties, crystallinity and interfacial adhesion of composite materials [58-60].

The injection molding is a manufacturing process for fabrication of different parts by injecting molten material into a mold [61]. The die of mold is made of a desired shape. The thermosetting and thermoplastic polymers can be used as raw materials [62]. Thermosetting polymers are irreversible curing materials. These materials become rigid on heating and cannot regain its original shape again. The vulcanized rubber, fiber glass, polyurethane, Bakelite, epoxy resins and silicon resins are examples of thermosetting polymers. The thermoplastics polymers can remelt and recure its shape after heating or cooling. These materials become soften on heating. The PP, PE, Polystyrene, PET and polycarbonate are examples of thermoplastics polymers. The thermoplastic and thermosetting materials are transformed into designed products in cold and hot molds, respectively. Injection molding technique is suitable for high volume production, low-cost components and short fiber reinforcements [63]. The PP-Abaca, PP-jute, PP-flax [64], PP-wood fibers [65], PP-hemp [66] and PP- glass [66] are examples of injection molded composites.

The process of forcing polymeric materials (between the two open die mold pressers) into a desired shape is known as compression molding. Compression molding is a very old processing technique [67]. The application of higher pressure removes the possible cross-sectional and surface defects in composite materials. Compression molding provides better control of physical, mechanical, surface and tribological properties [68]. Moreover, complex shapes with higher and lower variations in dimensions can be manufactured [69]. The PP-silk [70], PP-Ca-alginate fibers [71], PP-jute [72] and PP-glass [73] fiber are examples of compression molded composite materials. The high tool cost, low volume production and creation of micro cracks are major drawbacks of this technique [74].

1.5 Testing of polymer waste and composites

The quality and performance of recycled products relies on inspection and testing of polymer wastes and composites [75]. The mechanical properties of fabricated reinforced composites rely strongly on the properties of reinforced fibers [76]. The testing enables to select the suitable recycling technique as well [77]. The testing techniques are mentioned in Table 1.2.

Table 1.2. *Testing standards for quality control of polymeric waste and composites [adapted from Publication III].*

Testing technique	Polymer materials	Outcomes
Thermogravimetric analysis (TGA), Differential scanning calorimetry (DSC)	Engineering polymers, reinforced composite, particulate composites	Determination of thermal stability. Measurements of melting, degradation and crystallization temperatures. Degree of crystallinity
Student-Newman-Keuls (SNK) test. ISO 4287-1997 surface roughness contact and non-contact methods.	Polymer virgin/wastes materials, fiber-reinforced composites, hybrid composites.	Estimation of surface roughness.
ASTM D5034-95-08 grab and the ASTM D5034-95-06 strip tests.	Natural and synthetic	Determination of tensile strength, breaking force, fracture strength, strain and elongation.
ASTM D3039 tensile test, ASTM D5467 bend test, ASTM A370 Charpy test.	Manufactured/recycled Polymer composites for instance PP-cotton, PP-PES, PP-PET, *etc.*	Evaluations of mechanical and flexural properties.
Abrasion test, erosion test, fatigue test, creep test	Natural and synthetic polymers and composite materials	COF, wear, plastic deformation.
Scanning electron microscopy (SEM) characterization, optical and mechanical surface roughness measurements	Polymer and composite materials	Surface and cross-sectional micro defects evaluations, roughness parameter evaluations.

The tensile, bending, impact and hardness testing are most important for estimation of the properties of recycled composite materials [78]. The tensile strength and modulus of elasticity assured stability and stiffness during service life. Similarly, the percentage of elongation or strain evaluates the flexibility and ductility of a composite. Composites with reasonable ductility are preferred for structural applications. However, the composites either with the low ductility or with high brittleness are mostly used for static applications [79,80].

In bending conditions composites bear load in compressive and tensile conditions. The flexural strength and constant assured the withstand ability and stiffness in compressive conditions [81,82]. Similarly, flexural strain analyzes the performance and limits of load bearing capacity. The higher flexural values are a good indication of recycled composites for commercial applications. Higher flexural strength is mostly desirable for designing of composite materials. To satisfy desirable properties of composites, a fiber's diameter, length, composite surface properties, processing parameters like temperature, pressure, fabrication environment and cooling rates must be optimized carefully [83-86].

The recycled composites also face impact loads during service life. The impact loads are measured in terms of impact energy (toughness). The polymeric fibers help in transferring of load during impact load bearing applications.

Thermal analysis of polymers helps to understand the characteristics like melting, crystallization and degradation temperatures and degree of crystallinity. The thermogravimetric, differential scanning calorimetry and thermomechanical techniques are most common examples. The evaluation of chemical stability in different environments can be carried out in the case of specific applications.

The tribology deals with the study of friction, wear and lubrication of interacting surfaces. The processing and tribological interactions produced different imperfections and flaws. The abrasion, erosion, fatigue and creep testing is introduced to evaluate the surface performance of composite materials. The selection of the type of tribological test depends on type of composite material and possible commercial application.

The bulk and surface evaluations of composites are performed using optical, scanning and transmission electron microscopes and surface profilometers. These engineering instruments observe the presence of defects, surface roughness parameters and morphology of composites. This characterization plays important role in optimization of manufacturing of polymer products.

The single and double factor ANOVA are used to predict and optimized the mechanical and surface properties of composites [87]. ANN regressions also used to predict the behavior of composites [88].

1.6 Applications of composites

Due to combination of matrix and filler in composite, the composites are materials possessing the properties of both constituents. The main properties important for applications include mechanical properties (tensile and flexural stiffness, fracture toughness, *etc.*), thermal and chemical resistance, and wear resistance (erosive, abrasive, fatigue, *etc.*) [Publication VI]. Commercial applications include automotive (tires, different belts, hoses, bumpers, internal body panels, windscreens), marine (boats, mats, pressure vessels, barges, *etc.*), medical (MRI scanners, X-ray couches, surgical tools, wheels chairs, *etc*.), construction (pipes, insulations, sheets, glass-windows, doors, *etc*.) and other applications [89].

1.7 Aims and objective of the doctoral thesis

The main aim of thesis is the synthesis and investigation of mechanical (tensile, bending and impact) and tribological (COF, wear) properties, surface roughness and morphology and thermal (melting, crystallization, degradation temperature and percentage of crystallinity) properties of PP based composites reinforced with different type of post-consumer polymer waste (PCCF, PCPESF and PCPETF). Another important aim of the study was to link research with the concept of circularity. This concept assumes implementation of some steps to achieve the goals of sustainable industries.

I. Designation of the circularity paradigm, as close-loop recycling procedures of polymer waste materials. Fulfilment of the requirements for each step of circularity concept in the course of the study.

II. Investigation of the properties of PCCF waste to estimate the quality of the waste for the following recycling methods. Investigation of the tribological properties of different materials (steel, WC-Co, Al_2O_3, ZrO_2, steel ball coated with TiCN and TiAlN hard coatings) to estimate the applicability of these materials for the cutting of the PCCF waste in recycling process.

III. Selection of the suitable technique for the preparation of the PP-PCCF, PP-PCPESF and PP-PCPETF composites.

IV. Fabrication of the PP-PCCF, PP-PCPESF and PP-PCPETF composites, and investigation of the thermal, mechanical and tribological properties of PP-PCCF, PP-PCPESF and PP-PCPETF composites. Comparison of their properties and estimation of possible applications.

2 Materials and methods

The following steps were proposed regarding experimental section of the thesis. The steps include collection, separation, sorting, grinding, mixing, extrusion, injection, compression molding, inspection and testing of composite materials, see Figure 2.1.

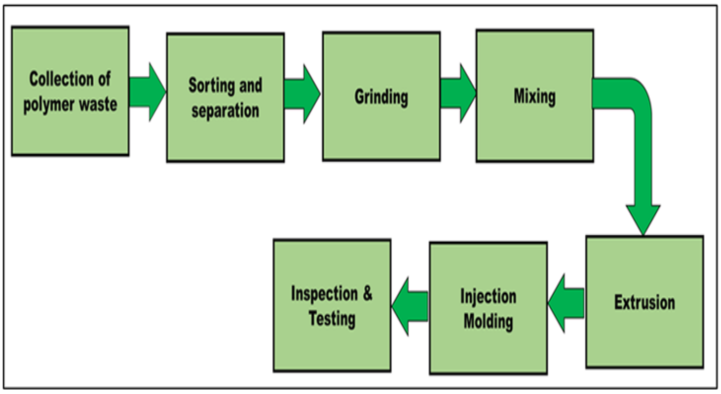


Figure 2.1. *Recycling process steps [Publication IV].*

2.1 PC waste materials

The PP powder was purchased from Egyeuroptene company. The post-consumer cotton fibers and post-consumer polyester fibers were derived from Estonian military T-shirts. Post-consumer polyethylene terephthalate fibers were derived from PET beverage bottles, see Figure 2.2a-d. Therefore, both the natural and synthetic polymer waste were used in study. The sorting and separation of all waste were done manually.

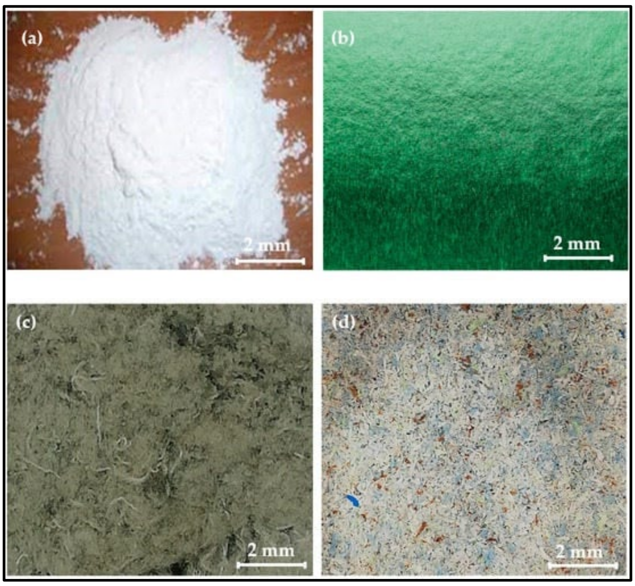


Figure 2.2. *Optical micrographs of selected PCF for fabrication of composites: (a) pure PP, (b) PCPESF, (c) PCPESF and (d) PCPETF [Publication IV].*

The following steps were proposed regarding experimental section of the thesis. The steps include collection, separation, sorting, grinding, mixing, extrusion, injection, compression molding, inspection and testing of composite materials, see Figure 2.2.

The virgin PP powder was selected as a matrix material. The pure PP has melt-flow index of 14 g/10 min at 2.16 kg of mass (ASTM D1238 standard). The melting temperature of PP was 210 °C. The PP powder was purchased from Egyeuroptene. The PP as a matrix phase provide excellent adhesion, interfacial bonding, fiber wettability, thermal, moisture and chemical resistance. Therefore, PP was also standardized as a reference material for development of composite materials.

The pure PCCF was derived from plain woven fabric T-shirts of Estonian army. PCCF has 1.55 g/cm^3, 5-25% ± 1.5%, 0.06 MPa ± 0.001, 0.10 ± 0.004 MPa, 0.04 ± 0.002 MPa and 237 g/m^2 values of density, elongation, tensile strength, design strength, breaking strength and weight, respectively. The PCCF was dried at 60 °C for 70 minute to remove moisture.

The pure PCPESF poly-1.4 cyclohexyl-di-methylene terephthalate (PCDT) was also derived from plain woven fabric T-shirts of Estonian army. The values of density, percentage of elongation, tensile strength, design strength, breaking strength and weight of PCPESF were 1.45 g/cm^3, 8-30% ± 1.7, 0.08 ± 0.004 MPa, 0.13 ± 0.004 MPa, 0.06 ± 0.002 MPa and 230 g/m^2, respectively.

The pure PCPETF poly (ethylene terephthalate (PET)) was derived from beverage bottles. The values of density, percentage of elongation and tensile strength of PCPETF were 1.45 g/cm^3, 6-12 ± 2% and 90 ± 10 MPa, respectively.

2.2 Grinding of waste

Manually sorted post-consumer cotton, post-consumer synthetic polyester, and post-consumer polyethylene terephthalate waste were ground using direct disintegration milling machine. Grinding machine grounds post-consumer waste into fine fibers (PCCF, PCPESF and PCPETF). The fibers length, diameter and area were measured using sieve analysis and SEM. The average length, diameter and area of PCCF and PCPESF were in the range of 2-5 mm, 11-21 µm and 200-250 µm^2, respectively. The average length, diameter and area of PCPETF were 0.31 mm, 0.01 mm and 0.20 µm^2, respectively, see Table 2.1.

Table 2.1. *Size distribution of fibers of post-consumer wastes [Publication IV].*

Fiber Nature	Average length (mm)	Average diameter (µm)	Average area (µm^2)
PCCF	3	17.87	250
PCPESF	3.5	17.50	245.21
PCPETF	0.31	0.01	0.19

2.3 Selection and fabrication of composites

The PP were mixed with PCCF, PCPESF and PCPETF using mixture machine. The 0, 10, 30 and 40% wt. fiber loading were used. The mixing time and speed were 15 min and 80 rpm, respectively.

The twin Brabender extrusion compounder machine (PLE 651-plasic corder) was utilized to produce extruded mate in the form of long cylindrical wires of diameter 1-3 mm. During extrusion, torque, time and speed were 60 Nm, 7 min and 40 rpm, respectively. The compounder consists of five different heating zones. The heating zones temperatures were 120 °C, 150 °C, 180 °C and 190 °C. The wires were ground into pellets of size 2mm. The pellets were dried for three hours at temperature of 60 °C.

The dried pellets were used as a raw material for injection and compression molding. The Battenfeld (BA 230A) injection molding machine was utilized to fabricate PP-PCCF, PP-PCPESF and PP-PCPETF composites. The machine operates at 120, 150, 180 and 190 °C for four different temperature zones. The mate in the form of pellets was passed through these melting zones and injected into the mold cavity. The injection, cooling and mold opening times were 8, 25 and 30 s, respectively. The composites were designed into 150 mm × 25 mm × 4 mm ASTM standard size for surface, mechanical and tribological investigations.

In the case of compression molding, a hydraulic press was used for compounding mate into flat sheets of size 1, 3 and 6 mm. During compression molding, time, pressure and compression temperature were 7 min, 80 kg/cm^2 and 190 °C, respectively. Finally, the compounding mate was forced into mold cavity and shaped into desired shape. The sheets were quenched for 1 min and annealed for 30 min. The formulation of composites is shown in Table 2.2.

Table 2.2. *Formation scheme of PP-PCCF, PP-PCPESF and PP-PCPETF composites [Publication IV].*

Cotton fiber's content (wt.%)	Matrix's content (wt.%)	Net weight of fibers (g)	Net weight of matrix (g)
0.0	100	0.0	300
30	90	30	270
90	70	90	210
120	60	120	180

2.4 Characterization of composites

The inspection and testing of fabricated composite materials were controlled using different techniques.

The differential scanning calorimetry (DSC) tests were performed using simultaneous thermal analyzer (Model STA 449 F3 Jupiter, NETZSCH Co) to evaluate melting and crystallization temperatures and degree of crystallinity. Each sample of 10 mg was heated and cooled from 0 to 250 °C and 250 to 0 °C at a heating rate of 15 °C/min, respectively. Similarly, thermogravimetric analyzer (TGA 1000 system, Anderson Materials Evaluation, Inc.) was used to carry out thermogravimetric (TGA) analysis for thermal degradation analysis to evaluate degradation temperature of composites. The composite samples of 10 mg were heated from 0 to 600 °C in alumina ceramic crucible at a heating rate of 10 °C/min.

Instron 5820 mechanical testing machine was utilized to evaluate tensile and bend properties. American society standards for testing of materials (ASTM D5034-95-05 grab test, ASTM D5034-06 strip test, ASTM D3039 tensile test and ASTM D5467 compression test) were used to evaluate mechanical properties. ASTM A370 impact test was utilized to measure the impact toughness of developed composites. The total, gauge and clamp lengths were 150 mm, 100 mm and 25 mm. The Instron computerized data acquisition software was used to analyzed and predict mechanical properties.

The ASTM D5034-95-05 grab and ASTM D5034-06 strip tests were used to measure tensile and breaking force of PCCF. The gauge length and width of PCCF samples were 100 mm × 25.4 mm. The crosshead speed of moving jaw of mechanical testing machine was 50 mm/min. Additionally, fracture surface of PCCF after grab and strip tests were characterized using SEM.

Abrasive sliding tests were performed to study the abrasive wear and COF. ASTM G132-96 standard was introduced for abrasion testing. Silicon carbide sandpaper was utilized as counter body. The pins of 5 mm × 5 mm × 15 mm in size have been used as a counter body against SiC sandpaper. The tests were performed on CETR UMT-2 tribometer. The applied load, speed and sliding distance were 1 N, 0.1 m/s and 70 mm, respectively. The tests were performed at room temperature for 18 m of distance and 3 minutes of time. Each test was repeated three times. The humidity level was 60%. After plastic deformation and local materials removal, the abrasive wear (mm^3/Nm) was calculated using following formulation:

$$W = \frac{v}{L \times S} \tag{2}$$

where W, *v,* L and S are abrasive wear, volumetric wear loss (mm^3), applied normal load (N) and complete sliding distance (m), respectively.

Table 2.3. *Composites, fabrication techniques and testing methods specifications [Publication III].*

PC waste	Fabrication technique	Composite material	Testing methods
PCCF	Compression and injection molding	PP-PCCF	Tensile test (ASTM D3039, compression (ASTM D5467), impact test (ASTM A370), ASTM D5034-95 grab test, ASTM D5035-95 SEM characterization
PCPESF	Injection molding	PP-PCFESF	SEM characterization
PCPETF	Injection molding	PP-PCPETF	SEM characterization

The four-channel accelerator erosive machine was used to estimate the erosive wear rate of composites. Silica sand of 6 kg in weight was used as an erosive medium. The particles size of silica sand were in the range of 0.10 to 0.60 mm. The test time, velocity of sand medium and impact angle during erosive test were 30 min, 30 m/s and

30°, respectively. The test was repeated for three times at room temperature. The weight loss was calculated using Mettler Toledo ME204 weight balance. Finally, specific weight loss M (mg/kg) was calculated using following formula:

$$M = \frac{\Delta m}{G \times v} \qquad (3)$$

where Δm, G and v are weight loss of each sample, weight of sand and share of sand per sample.

Similarly, erosive wear rate in terms of volumetric loss E was calculated using following formulation:

$$E = \frac{m}{\rho} \qquad (4)$$

Where m and ρ were weight loss and density (mg/mm^3) of composite material.

Scanning electron microscope (SEM) was used to analyze surface and cross-sectional areas of all composites. Besides this, about 20 fibers of each types (PCCF, PCPESF and PCPETF) were also selected for SEM analysis. The 2 nm thin layer of gold was deposited on fiber surface using physical vapor deposition (PVD) for better evaluation. The length, diameter and area of each type of fiber were evaluated. Additionally, mechanical profilometer (Mahr Perthometer PGK120) and an Optical profilometer (Contour GT-K0+ 3D) were utilized in verticle shift interference mode to determine average surface roughness R_a (µm), root mean square roughness R_q (µm), maximum profile peak height R_p (µm), average maximum height of the profile R_z (µm) and maximum height of the profile R_t (µm). The mechanical properties, hardness and surface roughness parameters of metallic balls and thin film coatings are shown in Tables 2.4-2.6.

Table 2.4. *Surface roughness parameters of balls.*

Materials	Surface roughness parameters (µm)		
	R_a	R_z	R_p
AISI 52100 steel balls	0.34	0.67	0.56
C10 alumina ceramic ball	0.24	0.34	0.32
ISO 3290 G10 WC grade balls	0.10	0.17	0.21
ISO 3290 G3 zirconia balls	0.05	0.13	0.10

Table 2.5. *Surface roughness parameters and hardness of coatings.*

Thin film coatings	Surface roughness parameters (µm)			Vickers Hardness (HV10)
	R_a	R_z	R_p	
TiCN	0.43	0.33	0.25	1500
TiAlN	0.40	0.20	0.17	1400

Table 2.6. *Properties of steel, alumina, zirconia and WC balls.*

Materials	Modulus of Elasticity (kN/mm^2)	Vickers Hardness (HV10)	Ultimate tensile strength (kN/mm^2)	Rupture strength (kN/mm^2)	Ultimate compressive strength (kN/mm^2)
Steel	174	-	1.96	-	-
Alumina	350	1450	0.03	0.21	2.1
WC	350	1800	1.70	1.90	5.70
ZrO_2	195	2040	0.05	0.53	2

2.5 Characterization of tribosystems

The COF of cotton waste against different counterbody materials was measured in warp and weft directions in abrasion sliding tests. The 10 PCCF samples were prepared for each type of counterbody (AISI 52100 steel, alumina, WC-Co, zirconia balls, TiAlN, and TiCN coatings). The speed, time, force and sliding distance were varied in the range of 0.5-10 mm/s, 4-320 s, 0.50-9 N and 0-80 m respectively. These parameters were changed to study the variations in the COF value. The reference PCCF-AISI 52100 steel tribological system was used for testing tribological properties of AISI 52100 steel as an usual industrial machinery part used for the cutting of PCCF waste. Similarly, the PCCF-TiCN and PCCF-TiAlN tribosystems were developed with the aim to check for potential applications of coatings in recycling. Additionally, PCCF-Al_2O_3, PCCF-WC and PCCF-ZrO_2 tribological systems were used. The properties of tribosystems were also characterized using SEM and mechanical and optical profilometers to determine the surface morphology and roughness before and after abrasion testing.

3 Results and discussion

3.1 Circulatory management of polymer wastes

This part of thesis is a review of concept of circularity. Figures 1.1 and 1.2 represent the circular recycling model of polymeric material and closed-loop manufacturing. Visual (subjective) inspection, composition analysis, mechanical testing, tribological investigation and surface evaluations are introduced to select suitable recycling technique, i.e., choose between primary to ternary [90]. In addition, selection is also relied on availability of waste, nature of material, recycling cost and potential application.

The recycled products as a high strength low weigh (HSLW) material, i.e., materials with appreciated properties can be used as a potential candidate for smart textiles, electronics, nuclear, automotive, packaging, electrical and construction industries.

To summarize, commercial polymers are classified into natural and synthetic polymers. Pre- consumer, post-consumer, end-waste and end-of-life polymer waste can be recycled using primary, secondary, tertiary and incineration recycling techniques. In the present study, the following stages like Waste-Recycling-Raw Materials-Materials Synthesis were demonstrated (Figure 1.1). It should be stressed that Cost analysis, Final Product and Product user (Figures 1.1 and 1.2) as steps of the closed-system manufacturing were not carried out, as these tasks are out of the scope of the present study (Publications I and II).

3.2 Analysis of PCCF waste

The post-consumer cotton samples were prepared from T-shirt used in Estonian Army. The samples and the warp, weft directions in the samples are shown in Figure 3.1. The analysis of waste starts from subjective assessment of PCCF as shown in Table 3.1. The surface roughness parameters like R_a, R_q, R_p and R_z of the samples are shown in Table 3.2. The subjective assessment includes the measurement of the weight, length, density, direction of fiber alignment and other textile properties. In the case of the present cotton samples, no heavy worn and dirty parts were observed. Based on these data the preliminary estimation of the quality of the waste can be done.

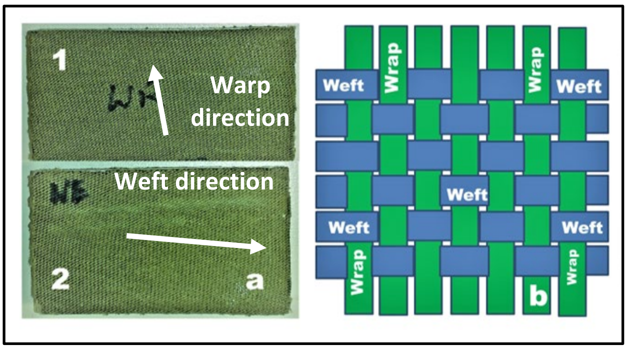


Figure 3.1. *PCCF samples: (a)1 - specify warp, 2 - weft directions, respectively and (b) weft (0°) and warp (90°) directions demonstrated on the model of the piece of the textile.*

Table 3.1. *Subjective evaluation of PCCF waste.*

Physical Property	Unit	Value	Physical Property	Unit	Value
Woven-Weft	-	Plain	Thread diameter in weft direction	mm	0.345
Woven-Warp	-	Plain	Thread diameter in warp direction	mm	0.345
Weight	g-m^{-2}	237	Twist value	T/m	800
Warp linear density	cm^{-1}	29	Thickness	mm	0.45
Weft linear density	cm^{-1}	29	Weft-Warp thread setting	cm^{-1}	18-36 Thread setting

Table 3.2. *Surface roughness parameters of PCCF samples measured by optical and mechanical profilometers.*

Device	Values of surface roughness parameters in weft direction (µm)				Values of surface roughness parameters in weft direction (µm)			
	R_a	R_q	R_p	R_z	R_a	R_q	R_p	R_z
Optical	40.33	54.78	260.57	340.15	25.31	56.47	220.55	321.37
Mechanical	35.19	47.49	227.27	-	26.31	51.87	204.88	-

The results of mechanical evaluations (grab and strip tests) and SEM observations of cotton fabric waste are show in Figure 3.2a, b and Table 3.3. Five samples for each type of the test were used for investigation. The values of tensile and breaking force were higher in warp direction. Higher values indicate expected good quality and performance of cotton waste. Furthermore, the tensile properties of cotton waste are considered good for primary recycling and therefore a new product made from this material is expected possesses good quality. For comparison, the tensile force for single jersey knitted virgin 100% cotton fabric is about 220.8 N [91].

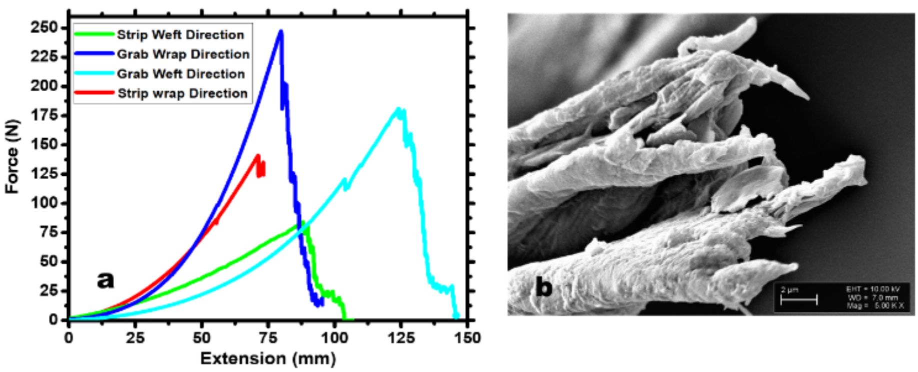


Figure 3.2. *(a) Results of tensile grab and strip tests and (b) SEM image of fractured cotton fibers.*

Table 3.3. *Results of tensile grab and strip tests.*

Tensile test	Tensile values in warp direction (N)		Tensile values in weft direction (N)	
	Tensile force	Breaking force	Tensile force	Breaking force
Grab test	251	212	180	150
Strip test	140	-	80	68

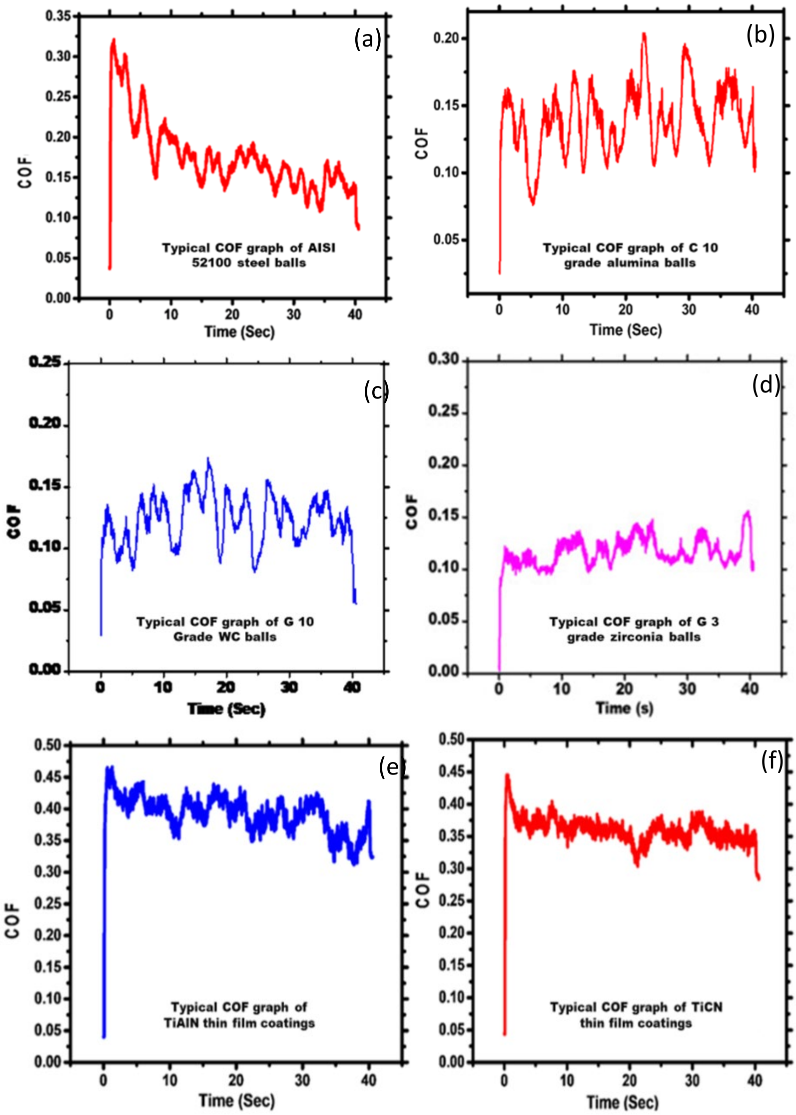


Figure 3.3. *Typical COF versus time graphs measured in sliding tests of different counter bodies against PCCF waste: (a) AISI 52100 steel, (b) alumina, (c) WC-Co, (d) zirconia, (e) TiAlN and (f) TiCN coatings.*

Basit et al. have found the tensile force of pure cotton fabric 245 N [92]. The similar results were also found in other investigations conduct by Penava et al., the tensile and breaking force of pure cotton were 280 N and 220 N [93].

One of the important aspects of the recycling is a correct choice of the recycling technology and tools using in recycling. It is desirable to reduce the damage of the fibers during the cutting, shredding, *etc*. of the waste. The tribological investigation of the materials using for production of tools is important for industrial applications.

The tribological behavior of PCCF waste was investigated against different counter body materials. The surface roughness of various materials is show in Tables 2.4 and 2.5. Examples of tribological behavior of PCCF waste against different materials (steel, alumina, WC-Co, zirconia, TiAlN, TiCN) are shown in Figure 3.3. The COF value of PCCF against AISI 52100 steel balls were found 0.12-0.21 and 0.10-0.17 in warp and weft directions, respectively. The COF value varied for 8-25 m range of sliding distance and no variations in COF value were found for 25-80 m range of sliding distance. Additionally, the minimal plastic deformation was also detected on surface of PCCF waste.

Sliding tests were performed on the PCCF-alumina tribosystem. The R_a, R_z and R_p surface roughness parameters were 0.24, 0.35 and 0.37 µm, respectively. The variations in speed, time, force and sliding distance have negligible effect on values of COF and surface conditions of PCCF. The average COF values were in the range of 0.11-0.15 in warp and 0.12-0.17 in weft directions. The tribological interactions deformed the PCCF waste. However, no fracture and abrasive wear of PCCF waste were detected during SEM characterization.

The COF value of PCCF-WC-Co tribological system varied in the range 0.06-0.15 in warp and 0.10-0.18 in weft directions for variations of speed, force and sliding distance during abrasion testing. The sliding of WC balls deformed PCCF waste.

The COF value of PCCF-zirconia tribological system was found in the range of 0.04-0.20 in warp and 0.05-0.18 in weft directions. The variation in sliding distance produced minimal plastic deformation on surface of PCCF waste.

The TiAlN coating was deposited on AISI 52100 steel balls using physical vapor deposition (PVD). The scratches, pits and impurities were detected on surface of TiAlN coated steel balls during SEM analysis. The TiAlN coated steel balls show high surface roughness parameters due to presence of surface defects, see Tables 2.5. During tribological testing, the average COF value was found in the range of 0.30-0.47 in the case of PCCF-TiAlN tribosystem. The plastic deformation on the surface of PCCF was observed. After 40 m of sliding distance, the COF value becomes constant. The increase in sliding distance leads to fracture of PCCF waste.

For PCCF-TiCN tribological system, the COF value varied in the range of 0.30 to 0.37 in warp and 0.23-0.37 in weft directions. Furthermore, the COF value becomes constant after 40 m of sliding distance. The fracture of PCCF waste was observed.

The SEM evaluations of post-consumer cotton waste show formation of different surface defects like bobs, jerks, damage, distortion and production of microfibrils. These defects cause the increase in surface roughness of PCCF with following unwanted slipping, buckling and tangling interaction effects between the waste and tool during recycling. Due to defects the grip between the worn fabric and cutting tools could decrease leading to worse cutting quality. Therefore, increasing of COF for better recycling of waste can be expected. The combination of suitable mechanical properties and surface roughness can enhance the abrasive, erosion, fatigue, corrosion resistance of machinery parts during processing. The higher hardness are usually required during recycling to reduce the wear of the cutting tools.

It is clear also from Figure 3.3 that smoother COF curves (peak-valley COF values) were recorded after the tests with hard coatings (TiAlN and TiCN) in comparison with steel, alumina and zirconia balls indicating smoother sliding for the ball coated with coatings, indicating stronger adhesion or grip. It can be suggested that the higher COF value > 0.25 is required during recycling of PCCF waste [94-96]. However, the COF < 0.20 is recommended for virgin cotton cutting by blades.

To summarize, the PCCF waste was used as a material for recycling. The subjective investigation of the samples suggests that PC cotton waste were only slightly damaged and worn. The good mechanical properties in terms of tensile (251 N in warp direction) and tensile breaking forces (212 N) assured the suitablity of cotton waste for recycling. It is expected that an acceptable qualtiy of a new product can be produced from the cotton waste. From the point of view of the tribological tests, the cutting tools made from steel, zirconia, WC-Co and alumina are probably suitable materials for cutting of virgin cotton fabrics and tools coated with TiCN and TiAlN coatings are suitable for the cutting of cotton waste.

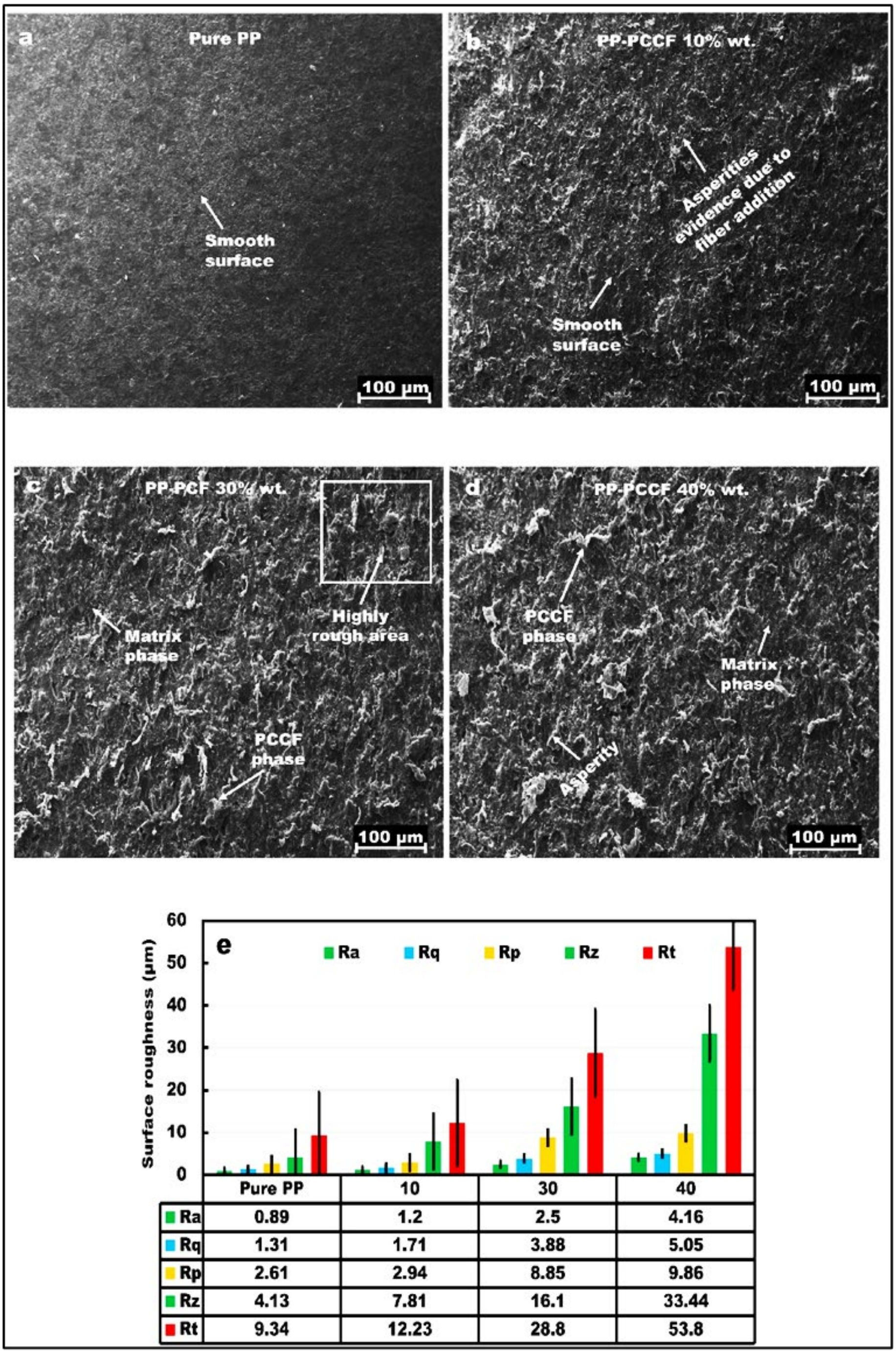
This part of study demonstrates the first stages of close-loop manufacturing, namely estimation of the quality of the waste and selection of suitable materials for recycling of the initial waste to produce high-quality product from waste for the next stages (Publication II).

3.3 Fabrication of PP-PCCF composites

The PCCF composites were manufactured using compression and injection molding techniques. The SEM micrographs and surface roughness parameters of compression molded reference material (PP) and recycled composites (PP-PCCF) sheets are presented in Figure 3.4a-e. The surface of pristine PP is smooth, however on the surface of composite with 10 wt.% fiber loading surface's asperities can be observed. Number and size of surface asperities increase with increase in fiber loading. The surface roughness parameters of composites were measured. The values of parameters proved the surface smoothness especially pristine PP and composite with 10% wt. fiber fraction. However, increase in fiber contents causes poor bonding and the enhancement of surface roughness parameters.

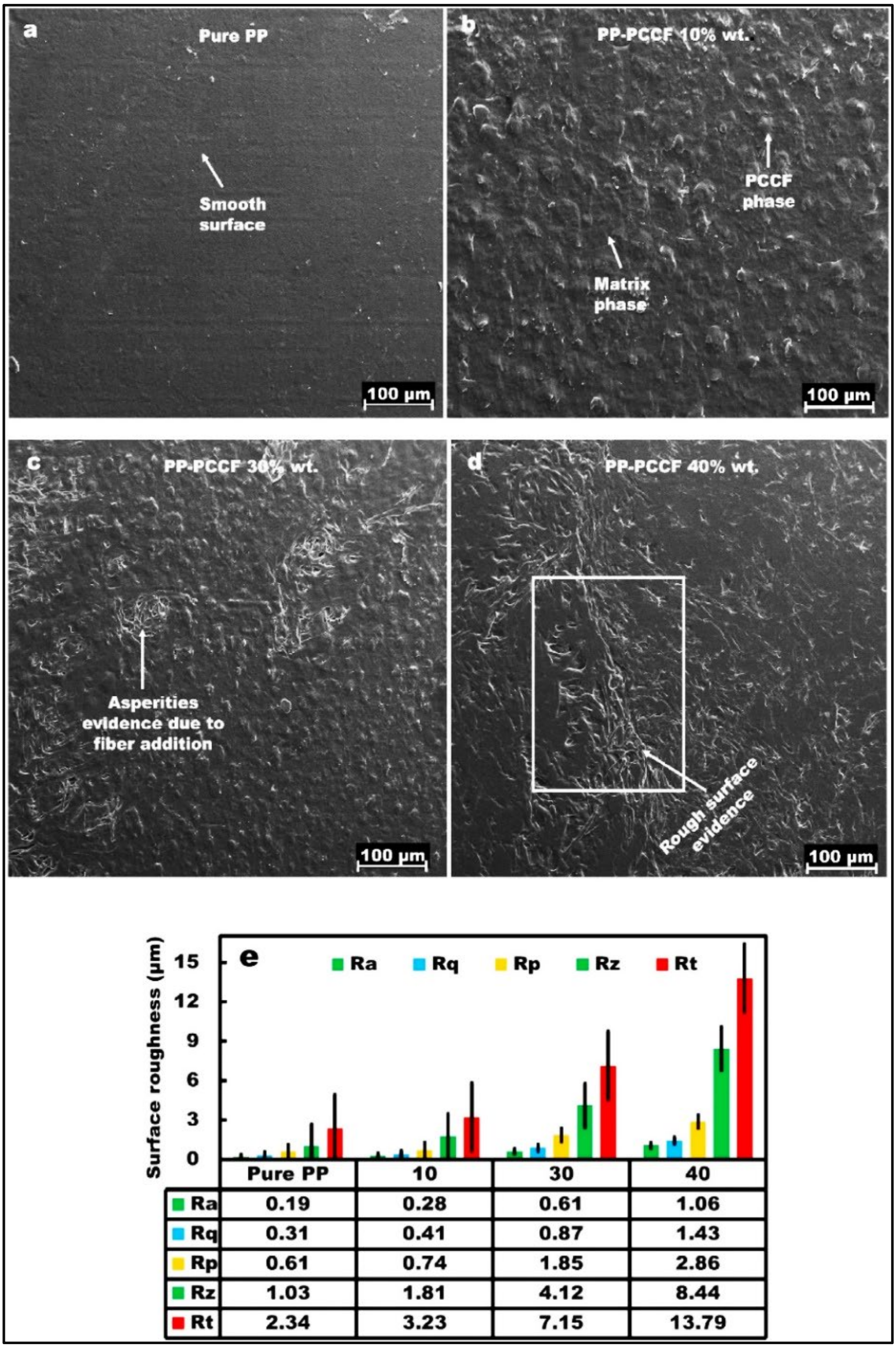
The injection molding processing provides smoother surface with less defects and lower surface roughness as for pristine PP, as well as for PP-PCCF composites in comparison with compression molding (Figures 3.4 and 3.5). The surface roughness was evaluated on five different places on the surface. The uniformity of surface decreases with increase in fiber addition due to poor bonding and defects formation.

Figure 3.6a-d shows the cross-sectional SEM images of the pure PP and recycled composites prepared by compression molding. Voids of different sizes can be seen. These micro defects probably formed due to poor manual temperature and pressure control during processing. Insufficient control of processing parameters caused the poor compaction and lower wettability.



	Pure PP	10	30	40
Ra	0.89	1.2	2.5	4.16
Rq	1.31	1.71	3.88	5.05
Rp	2.61	2.94	8.85	9.86
Rz	4.13	7.81	16.1	33.44
Rt	9.34	12.23	28.8	53.8

Figure 3.4. *SEM images of the composites fabricated by compression molding: (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt., (d) PP-PCCF 40% wt. and (e) surface roughness parameters.*



	Pure PP	10	30	40
Ra	0.19	0.28	0.61	1.06
Rq	0.31	0.41	0.87	1.43
Rp	0.61	0.74	1.85	2.86
Rz	1.03	1.81	4.12	8.44
Rt	2.34	3.23	7.15	13.79

Figure 3.5. *SEM images of the composites fabricated by injection molding: (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt., (d) PP-PCCF 40% wt. and (e) surface roughness parameters of composites.*

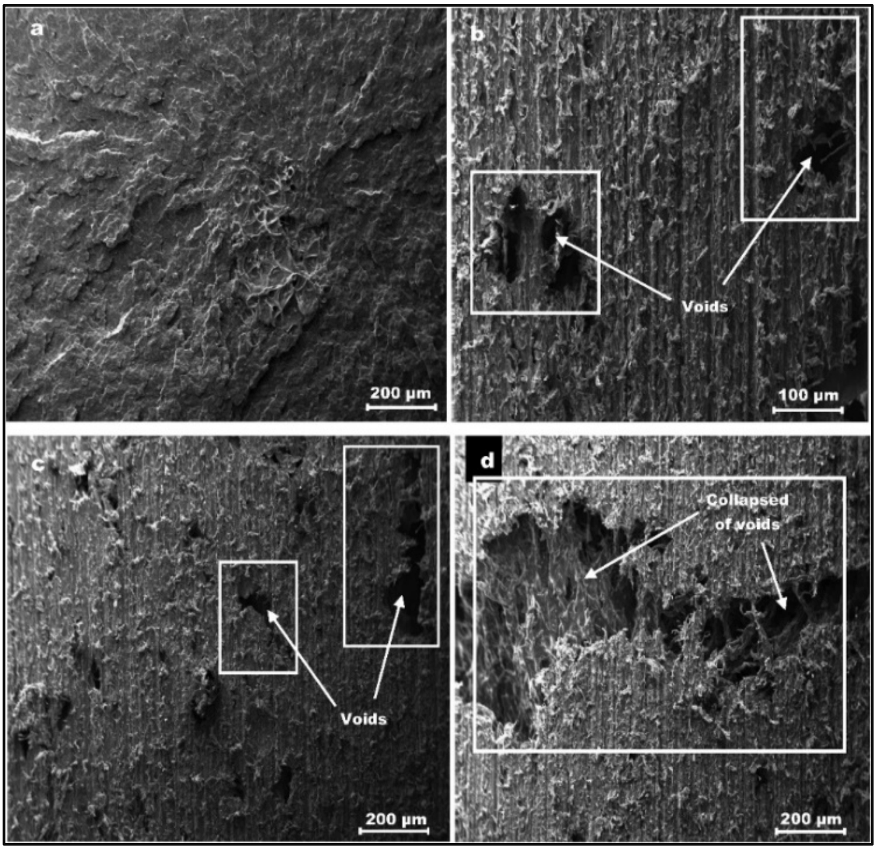


Figure 3.6. *SEM images of the cross-sectional area of the composites fabricated by compression molding: (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt. and (d) PP-PCCF 40% wt.*

Figure 3.7a-d shows the almost defects free SEM images of pure PP and recycled composites prepared by injection molding. Anevidence of voids was found only in PP-PCCF-40% wt. composite.

To summarize, the surface morphology, cross-sectional SEM analysis and roughness parameters measurements show lower number and size of defects in structure of composites prepared by injection molding in comparison with ones prepared by compression molding. Therefore, injection molding tehnique was used in following studies as for preparation of PP-PCCF composites, as well as for other types of composites.

This part of the study demonstraites next step within the framework of close-loop circularity concept (Figures 1.1. and 1.2), namely the selection of suitable manufacturing technique for the preparation of product, i.e. in our case composites (Publication III).

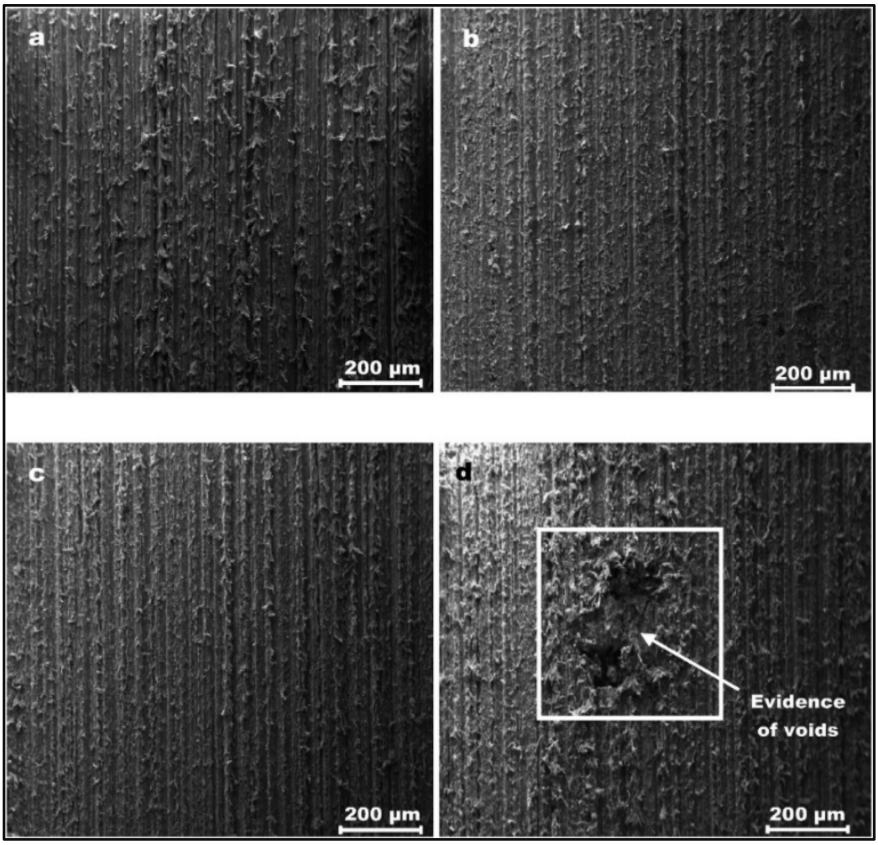


Figure 3.7. *SEM images of the cross-sectional area of the composites fabricated by injection molding: (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt. and (d) PP-PCCF 40% wt.*

3.4 Comparative analysis of PCCF, PCPESF and PCPETF reinforced PP based composites

The all three types of composites were fabricated using injection molding, as most suitable technique, see previous section. The composites with 0, 10, 30 and 40% of PCCF, PCPESF and PCPETF fiber loadings in PP matrix were prepared. The results of thermal analysis are shown in Figure 3.8. The addition of PCCF, PCPESF and PCPETF affect the thermal properties of composites. Melting temperature (Figure 3.8a) indicates the temperature of melting of composite matrix (PP). Perfectly isotactic PP has a melting point of 171 °C. The slight decrease in melting temperature with the higher fiber loads was found due to removal of moisture and volatile compounds from composites. The PP crystalline phase plays vital role for mechanical properties of composites. The better mechanical properties can be expected for compounds with the higher degree of crystallinity. In fact, amorphous phase exists in composite as well. Crystallization temperature was lowest for 10 and 30% of fiber loading in the case of PP-PCCF composites (Figure 3.8b). However, degree of crystallinity 44% was highest for 10% PP-PCCF and PP-PCPESF-10% composites (Figure 3.8c). An increase in fiber fraction produced amorphous phases and micro-defects leading to decrease in degree of crystallization. The orientation of fibers, nature of reinforced materials and fibers length also affect the degree of crystallinity. Thermal degradability relates to the collapse of molecules of the PP matrix phase. According to Figure 3.8d, the thermal degradability of composite materials occurs in the range of 445 °C to 470 °C, in comparison with 475 °C for pure PP.

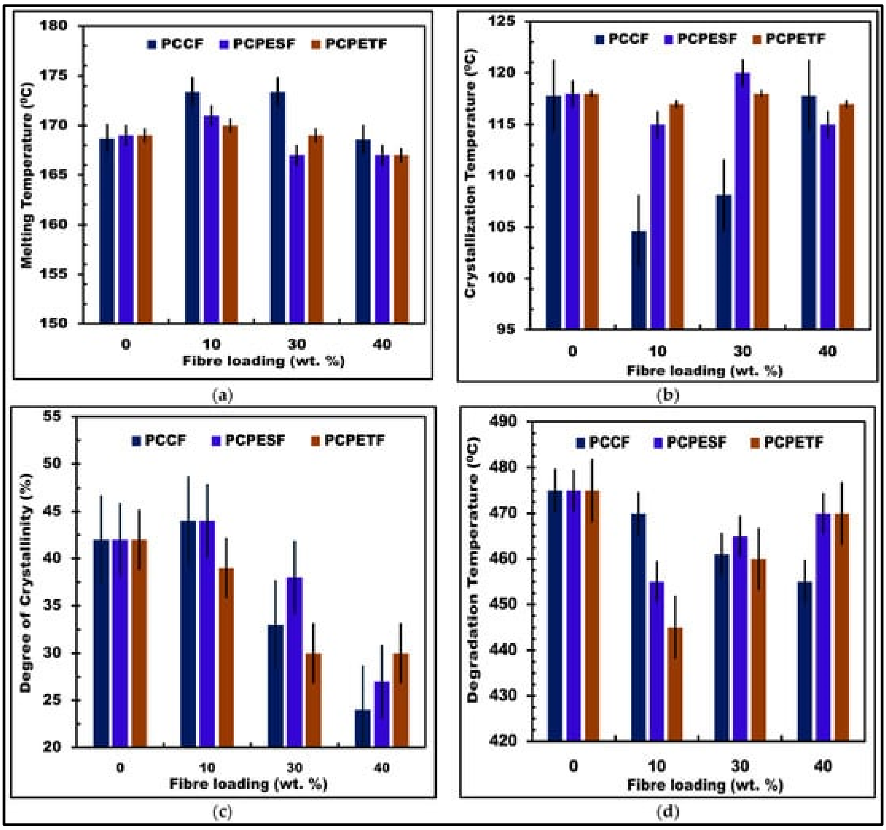


Figure 3.8. *Results of thermal analysis of pure PP, PP-PCCF, PP-PCPESF and PP-PCPETF composites: (a) melting and (b) crystallization temperatures, (c) degree of crystallinity, (d) degradation temperature.*

The surface morphology of PP-PCCF, PP-PCPESF and PP-PCPETF composites was characterized using SEM. The PP-PCCF, PP-PCPESF and PP-PCPETF composites melt, degrade, crystallize at different temperatures, due to different nature of fibres. Therefore, it is expected that the surface morphology of composites is different as well. Porosity, microfibrils and distortion of PCCF was found on the surface of PP-PCCF composites (Figure 3.9a). Smooth surface and asperities were detected on the pure PP and PP-PCCF-10% wt. composites, see Figure 3.9b and c. In Figure 3.9d and e, micro cracks and high rough areas were appeared on surface of PP-PCCF-30% wt. and PP-PCCF-40% wt. composites, indicating the lowering in PP-PCCF inter-bonding adhesion. The deformed and rough areas were detected on surface of PCPESF composite, see Figure 3.10a. Evidence of micro-pits (Figure 3.10b), grooves (Figure 3.10c) and highly rough surface regions (Figure 3.10d) on PP-PCPESF-10% wt., PP-PCPESF-30% wt. and PP-PCPESF-40% wt. composites was found. Figure 3.11a shows the rough regions of surface of PCPETF flakes. The surface cracks, uniform regions and asperities were observed on surface of PP-PCPETF composites, see Figure 3.11.

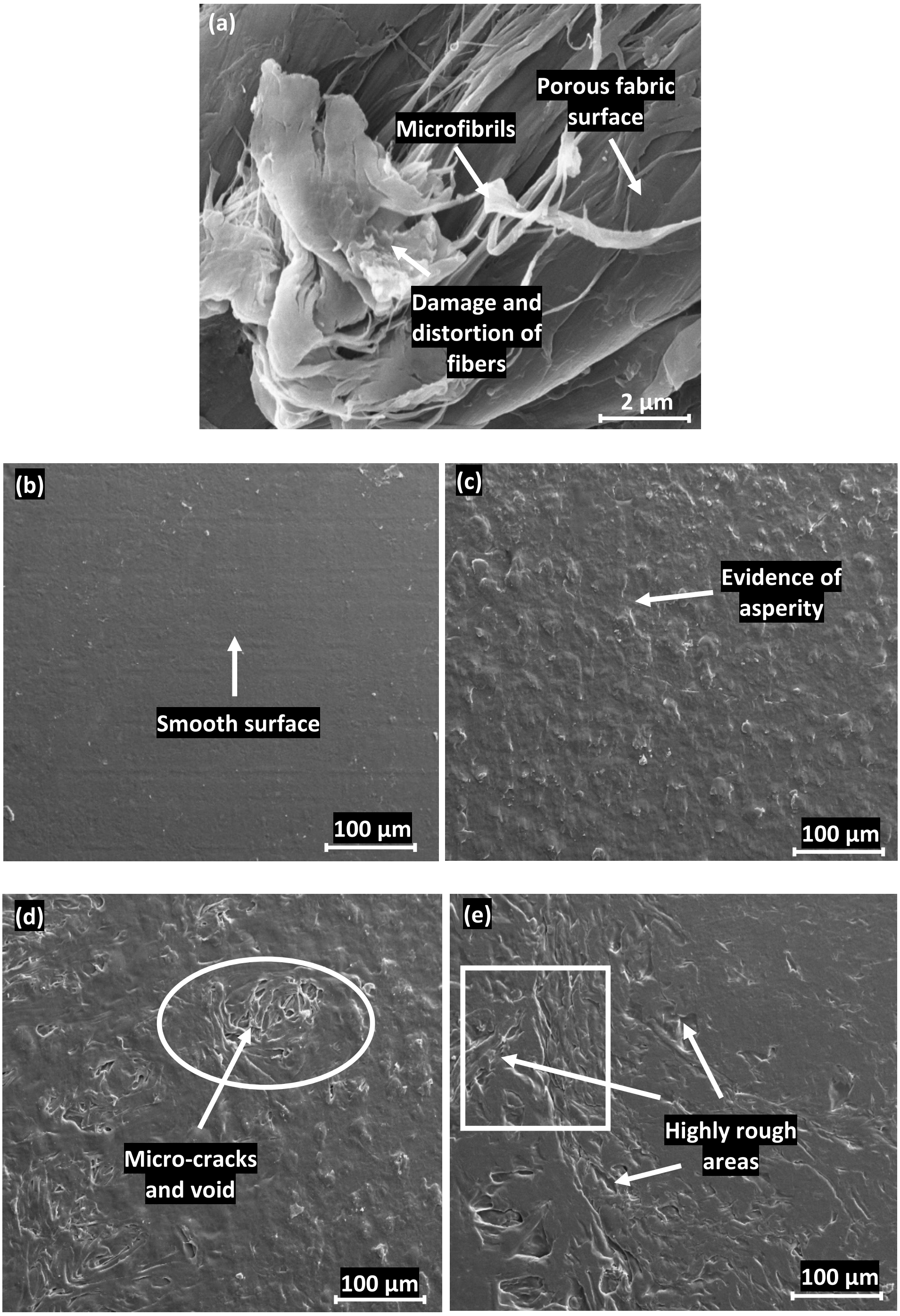


Figure 3.9. *SEM images of PCCF waste and PP-PCCF composites: (a) PCCF, (b) pure PP, (c) PP-PCCF-10% wt., (d) PP-PCCF-30% wt. and (e) PP-PCCF-40% wt.*

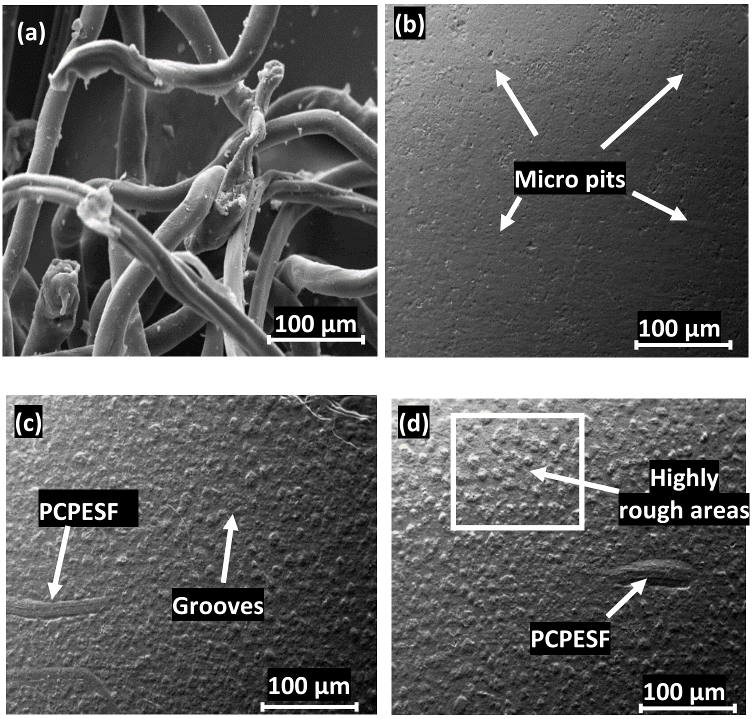


Figure 3.10. *SEM images of PCPESF waste and PP-PCPESF composites: (a) PCPESF, (b) PP-PCPESF-10% wt., (c) PP-PCPESF-30% wt. and (d) PP-PCPESF-40% wt.*

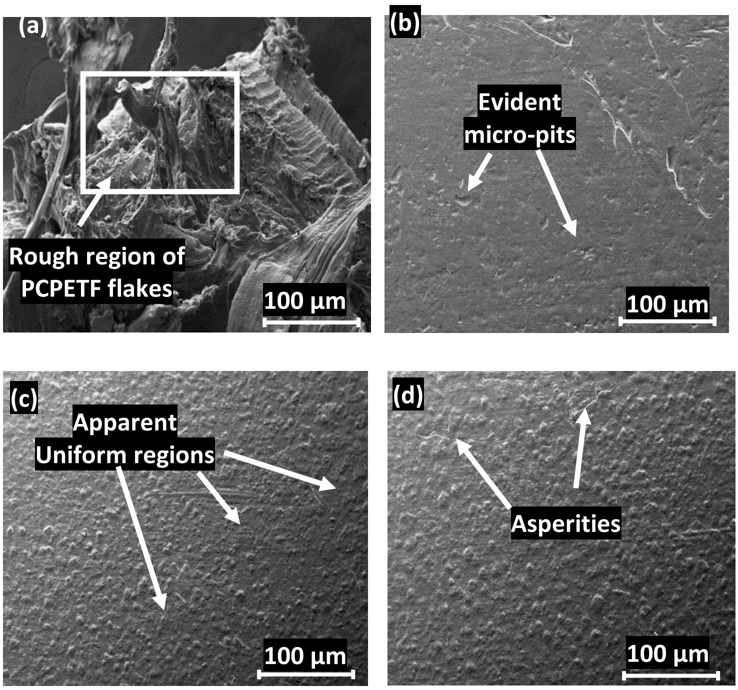
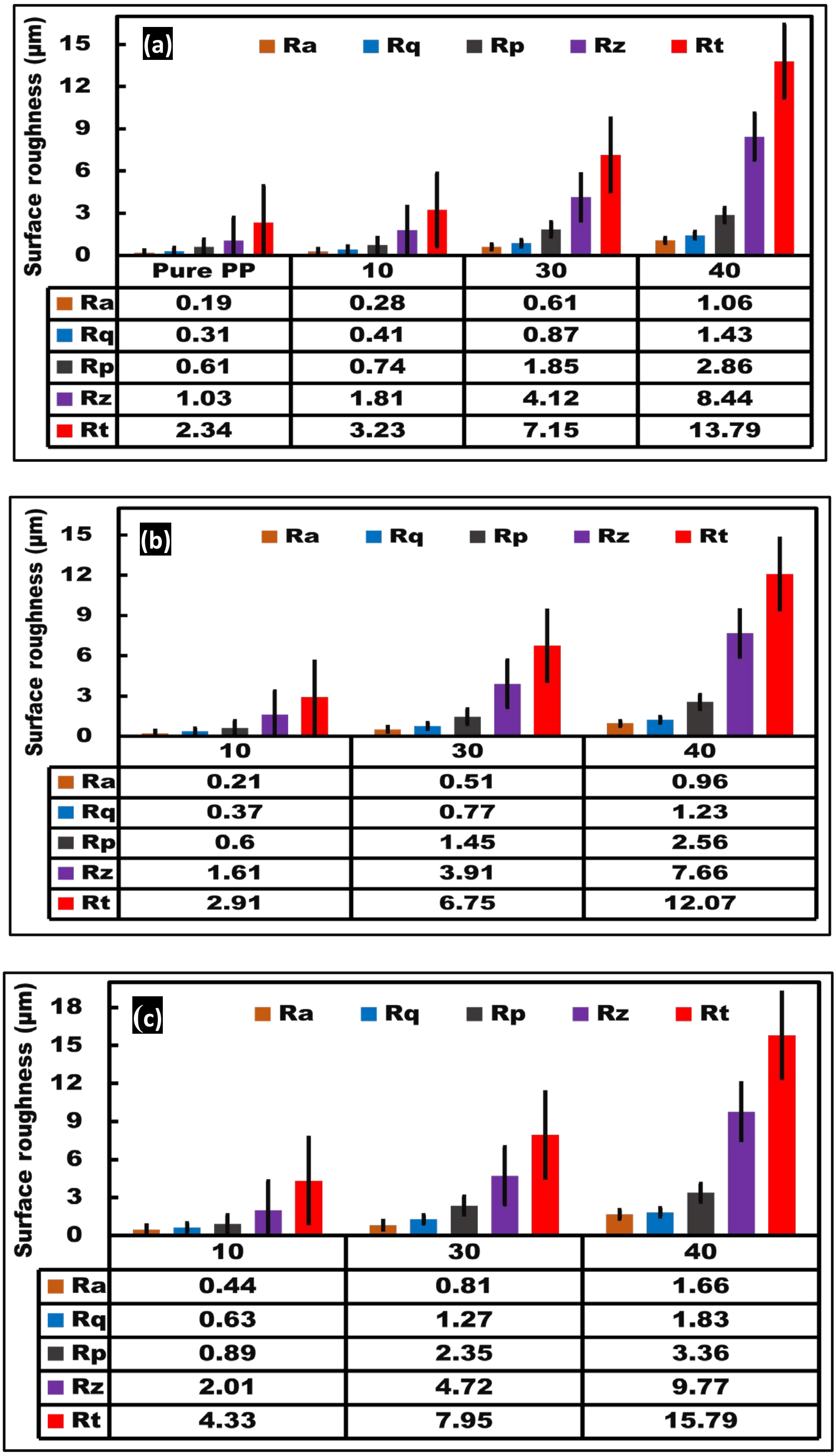


Figure 3.11. *SEM images of PCPETF waste and PP-PCPETF composites: (a) PCPETF, (b) PP-PCPETF-10% wt., (c) PP-PCPETF-30% wt. and (d) PP-PCPETF-40% wt.*



	Pure PP	10	30	40
Ra	0.19	0.28	0.61	1.06
Rq	0.31	0.41	0.87	1.43
Rp	0.61	0.74	1.85	2.86
Rz	1.03	1.81	4.12	8.44
Rt	2.34	3.23	7.15	13.79

	10	30	40
Ra	0.21	0.51	0.96
Rq	0.37	0.77	1.23
Rp	0.6	1.45	2.56
Rz	1.61	3.91	7.66
Rt	2.91	6.75	12.07

	10	30	40
Ra	0.44	0.81	1.66
Rq	0.63	1.27	1.83
Rp	0.89	2.35	3.36
Rz	2.01	4.72	9.77
Rt	4.33	7.95	15.79

Figure 3.12. *Surface roughness parameters of composites: (a) PP-PCCF, (b) PP-PCPESF and (c) PP-PCPETF.*

The evaluation of PP and composites properties in terms of surface roughness parameters is shown in Figure 3.12. The mechanical profilometry was used as experimental technique. The fiber addition causes the creation of surface defects and increasing of the surface roughness. The Ra, Rq, Rp, Rz and Rt values increase with increasing of the load of each reinforced material. The surface roughness of PP-PCPETF composites were highest in comparison with PP-PCPESF and PP-PCCF composites.

Figure 3.13a and b show the comparison of tensile and flexural (bending) strains. The applied tensile force produced extension (deformation) in PP and composites. The tensile and flexural strains of pure PP are 10 ± 1.75% and 14 ± 2%. The sharp drop of tensile strain was observed for the composites with 30 and 40% wt., i.e., showing brittleness of composites. In the case flexural or bending strain, the degrease of strain was observed as well, however for higher fiber loading it was not sharp like in case of tensile strain.

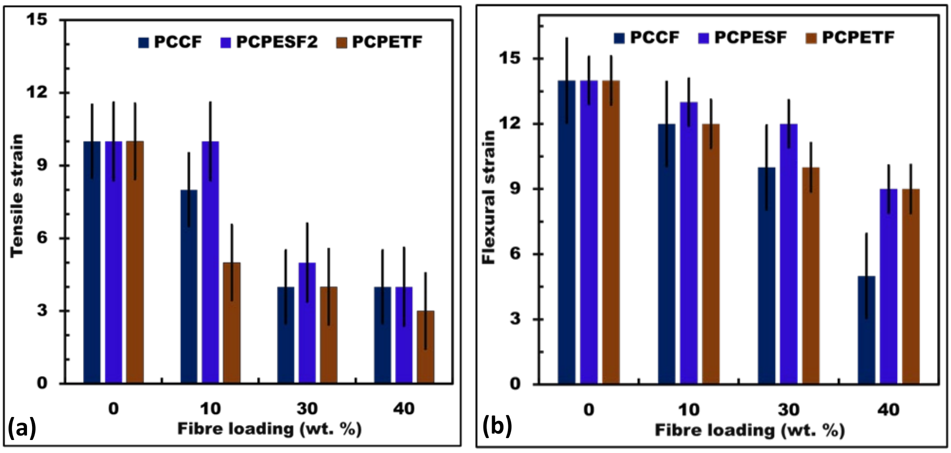


Figure 3.13. *Comparison of tensile and flexural strains between composites: (a) tensile and (b) flexural strains.*

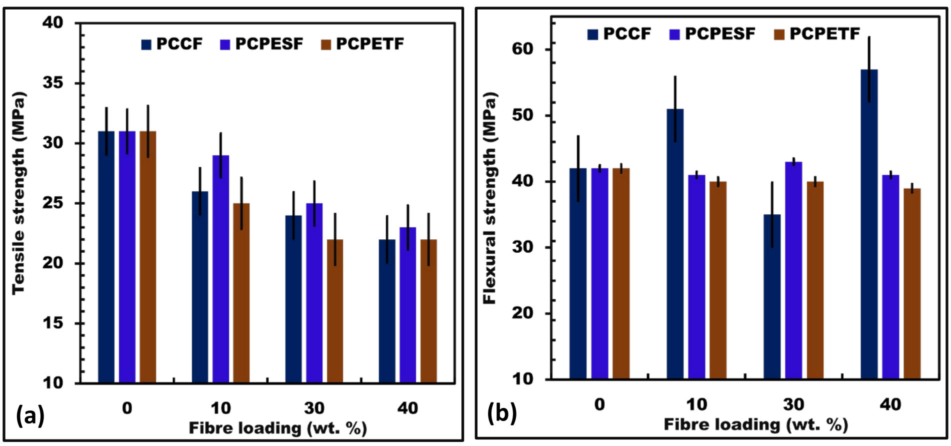


Figure 3.14. *Comparison of tensile and flexural strengths between composites: (a) tensile and (b) flexural strengths.*

Similarly with the results shown in Figure 3.13a, the tensile strength decreases with increase of fiber loading (Figure 3.14a). However, the flexural strength (Figure 3.14b) does not depend strongly on the type and load of fibers, with exception for PP-PCCF composite. The highest values of flexural strength of 51 and 57 MPa were found for PP-PCCF-10% and PP-PCCF-40% wt. composites, respectively. It can be concluded that composite behavior strongly depends on the direction of applied force, i.e., tensile or flexural stress.

The modulus of elasticity and flexural modulus of elasticity are shown in Figure 3.15a and b. The modulus of elasticity increases for PP-PCCF and PP-PCPESF composites with the increasing in fiber loading and decreases for PP-PPETF. In opposite, in the case of flexural modulus of elasticity the increase in value of modulus was observed for all composites (Figure 3.15b). The highest values of modulus of elasticity and flexural modulus were observed for PP-PCCF-40% wt. composite, i.e., 2751 MPa and 3780 MPa, respectively. In conclusion, stiffness or resistance to elastic deformation increases for PP-PCCF and PP-PCPESF composites.

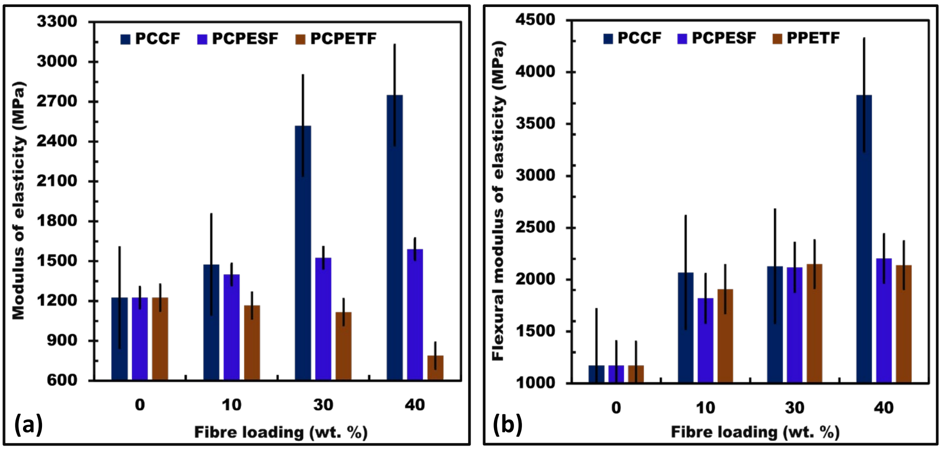


Figure 3.15. *Comparison of mechanical moduli between composites: (a) tensile and (b) flexural moduli.*

The fracture surfaces observed after tensile and flexural testing are shown in Figure 3.16a and b. The fiber transfers the load applied to composites in tests. Under higher applied load the fibers pull out takes place indicating failure in bonding. This type of failure was observed in both tensile and flexural tests. Beside fiber pull out, evidence of plastic deformation and fiber fracture were found on fracture surfaces of PP-PCPESF-40% wt. composites, see Figure 3.16b.

Figure 3.17a shows the results of impact tests. In the case of PP-PCPESF and PP-PCPETF composites, after sharp drop for 10% wt., the values of impact energy increase with increase of fiber loadings. In contrast, PP-PCCF composite shows good impact properties as compare with PP. The highest value of impact energy (5.5 kJ/m^2) was found for PP-PCCF-40% wt. composite. The impact tests result in deformation and fiber pull out observed on fracture surface, similar to tensile and bend testing, see Figure 3.17b.

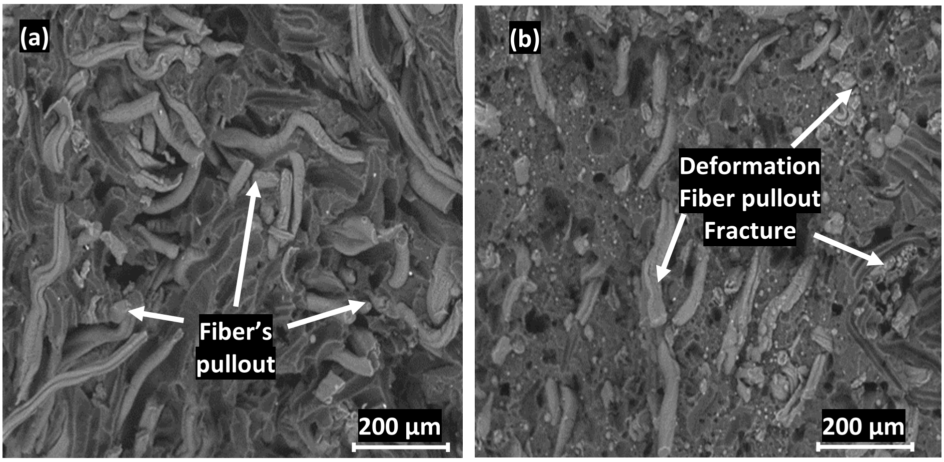


Figure 3.16. *SEM images of fracture surfaces of PP-PCPESF-40% wt. composite after mechanical tests: (a) tensile and (b) flexural tests.*

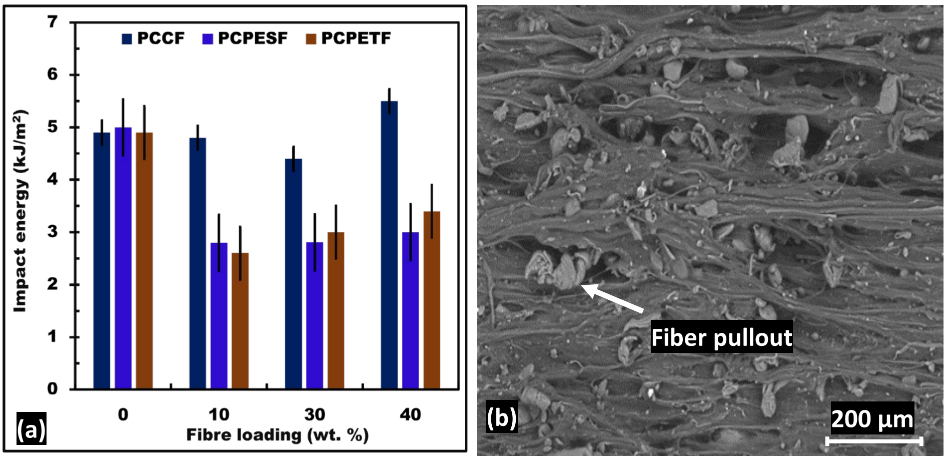


Figure 3.17. *Impact energy and SEM image of the surface of composite after impact tests: (a) impact energy (b) SEM image of PP-PCPESF-40% wt. composite surface after impact test.*

The results of the abrasive sliding wear and COF value are shown in Figure 3.18a and b. The pure PP shows lowest value of abrasive wear (3×10^{-6} mm^3/Nm) and COF value (0.70). However, addition of fibers (PCCF, PCPESF and PCPETF) produces defects on the surface leading to higher roughness of the surface and weaker adhesion between the matrix (PP) and fibers. Therefore, abrasive wear increased with increment in fiber loadings.

The COF value behavior was similar to one for abrasive wear rate. According to Figure 3.18b, the COF strongly increased with fiber loading. The increase in COF value is probably caused the same as in the case of wear rate, i.e., increase in number of defects on the surface and formation of rougher surface. The abrasive sliding between SiC P150 grade and composites caused the creation of plastic deformation and wear, see Figure 3. 18c.

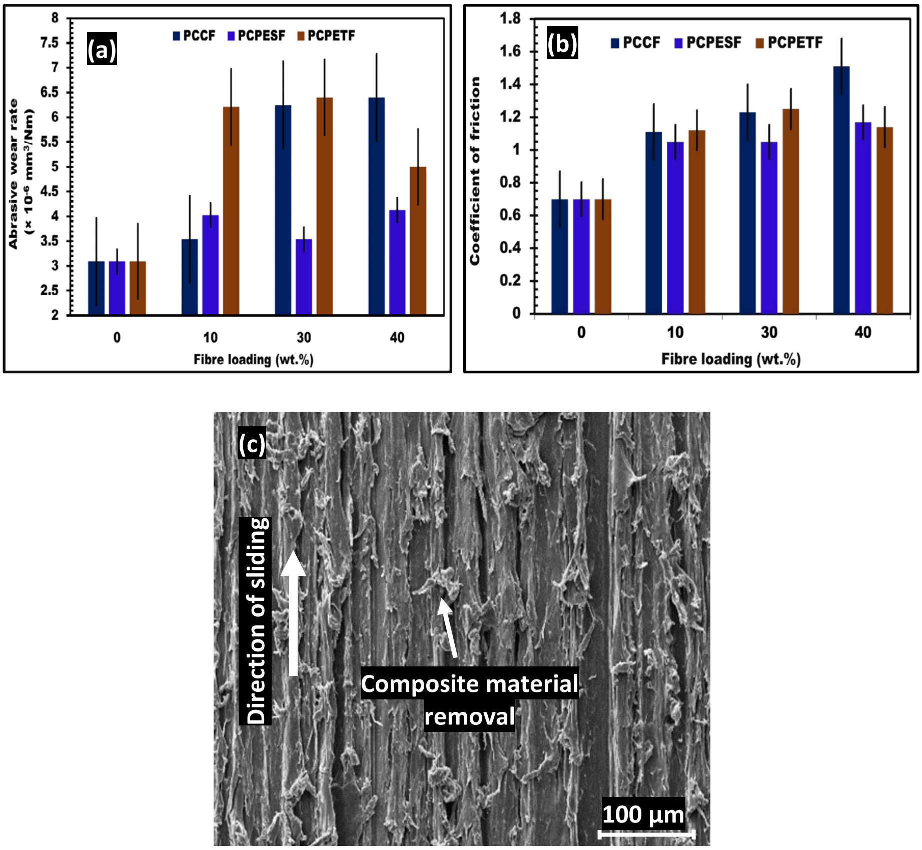


Figure 3.18. *Abrasive wear rate and COF of composites after abrasion sliding tests: (a) abrasive wear rates, (b) COF and (c) SEM image of the surface of pure PP after abrasion test.*

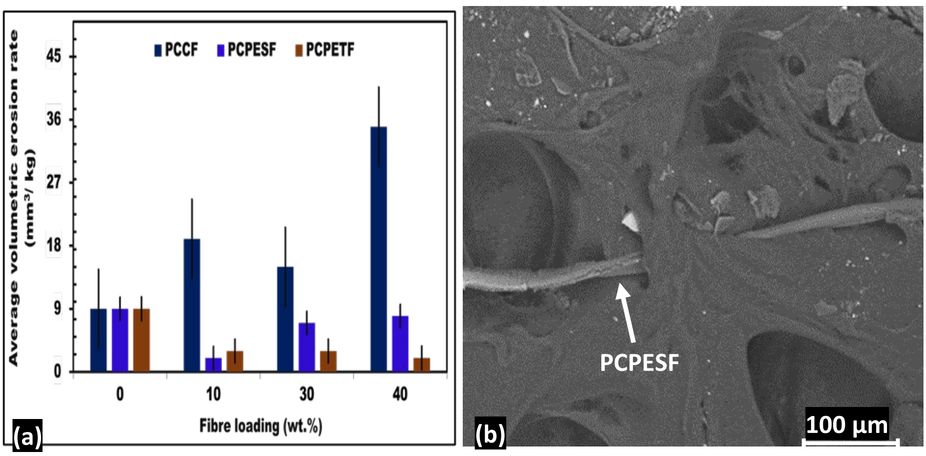


Figure 3.19. *Erosion wear rate and SEM image of the surface of PP-PCPESF composite after erosion test: (a) erosion wear rate and (b) SEM image of PP-PCPESF-40% wt. composite surface after erosive test.*

The results of erosive wear tests are shown in Figure 3.19a. The minimum, intermediate and maximum erosive wear were found correspondingly for PP-PCPETF, PP-PCPESF and PP-PCCF composites. The best erosive resistance (2-3 mm^3/kg) was shown on the PP-PCPETF composite, indicating ability of this type of composite to absorb impact energy of silica particles. Furthermore, fibers pull out and evidence of erosive wear were found on the surface of composites after erosive tests (Figure 3.19b).

To summarize, the PP-PCCF, PP-PCPESF and PP-PCPETF composites were manufactured using injection molding technique with 0, 10, 30 and 40% wt. of fiber loadings.

The PP-PCCF-10% wt. and PP-PCPESF-10% wt. composites exhibit highest value of crystallinity (44%) in comparison with pure PP (42%). However, in general, increase in fiber loading decreases the value of crystallinity. Highest degree of crystallinity for PP-PCCF 10% wt. and PP-PCPESF-10% wt. suggests better mechanical properties. Crystallization temperature of all materials was in the range of 115-120 °C, except PP-PCCF-10% wt. (105 °C). The melting and degradation temperature of all composites were in the range of 167-170 °C and 445-470 °C, respectively. In comparison, crystallization, melting and degradation temperatures for pure PP were 118 °C, 169 °C and 475 °C, respectively.

Highest surface roughness was found for PP-PCPETF composites.

Among the manufactured composites, the PP-PCPESF 10% wt. composite exhibits highest values of tensile strength (29 MPa) and strain (10%). The addition of fibers in PP matrix decreases tensile and flexural strain, as well as tensile strength decrease was observed. In opposite, flexural strength value was not affected by fiber loading and even increase in flexural strength value for the PP-PCCF 10% wt. and PP-PCCF 40% wt. composites was found. Increase in stiffness (modulus of elasticity), or resistance to elastic deformation was found for PP-PCCF and PP-PCPESF composites with increasing of fiber loading. Behavior of flexural modulus of elasticity was relatively similar between all composites, i.e. it increased with increase in fiber loading and modulus values were much higher than for pure PP.

The PP-PCCF 10% wt. composite shows the best mechanical properties (except tensile strain and strength and flexural strain) among the fabricated composites. The higher degree of crystallinity (PP matrix phase) and good mechanical properties of cellulose as a main structural element of cotton are responsible for good mechanical properties of the PP-PCCF composites. This composite shows higher modulus of elasticity (1476 MPa) and flexural modulus of elasticity (2069 MPa) in comparison with pure PP 1226 MPa and 1172 MPa, respectively. Additionally, the value of impact energy of the pure PP, PP-PCCF 10% wt. and PP-PCCF 40% wt. composites were 4.9 kJ/m^2, 4.8 kJ/m^2 and 5.5 kJ/m^2, respectively. It shows higher toughness of PP-PCCF composites.

The higher abrasive sliding wear rates against silicon carbide sandpaper were found for all composites in comparison with pure PP (3.09×10^{-6} mm/Nm). Among the manufactured composite the smallest wear rate was found for PP-PCCF 10% wt. composite (3.54×10^{-6} mm/Nm). As well as the COF value for all composites was higher in comparison with pure PP.

In the case of erosive wear, the highest value was found for the PP-PCCF composites and the smallest for PP-PCPETF (2 mm^3/kg) in comparison with PP (9 mm^3/kg).

This part of the study can be considered as preparatory study for the next stage of the close-loop manufacturing and circularity (Figures 1.1 and 1.2), namely final product preparation and utilization of product by user (Publications IV-VI).

4 Conclusions and further research

The present study deals with the investigation of the properties of the composites with polypropylene matrix reinforced by post-consumer cotton, synthetic polyester and polyethylene terephthalate fibers. Circularity concept was introduced as a framework for selection of reasonable recycling and manufacturing techniques during the study. The post-consumer cotton fiber waste was used as the primarily recycling material for investigation. Before recycling, the properties of the waste like mechanical and tribological, surface roughness, *etc.* need to be evaluated to select reasonable following processing of waste.

Based on the outcomes of this research work the following conclusions can be drawn:

1. The study follows the concept of circularity, as the close-loop recycling procedures of polymer waste materials. The processing of polymeric waste consists of different steps like collection, sorting, separation, grinding, mixing, manufacturing of composites and testing their properties.

2. The subjective investigation of the samples suggests that PCCF waste were only slightly damaged and worn. The good mechanical properties in terms of tensile (251 N in warp direction) and tensile breaking forces (212 N) assured the suitablity of cotton waste for recycling.

The smoother COF versus time curves were recorded after the tests with steel balls coated with hard coatings (TiAlN and TiCN) as a counter body, with a higher COF value (0.35-0.45) in comparison with steel, alumina and zirconia balls (COF values 0.1-0.2) against PCCF. It indicates a smoother sliding for the ball coated with coatings and stronger adhesion or grip between the PCCF and coated balls. Therefore, a cutting tool coated with the mentioned coatings are suitable for the cutting of PCCF waste.

This part of study demonstrates the first stage of close-loop manufacturing, namely estimation of the quality of the waste and selection of suitable materials for recycling of the initial waste to produce high-quality product from waste for the next stages.

3. The surface morphology, cross-sectional SEM analysis and roughness parameters measurements showed a smaller number and size of defects in structure of PP-PCCF composites prepared by injection molding in comparison with ones prepared by compression molding. Therefore, injection molding tehnique was used for the preparation of all types of composites. The PP-PCCF, PP-PCPESF and PP-PCPETF composites were manufactured with 0, 10, 30 and 40% wt. of fiber loadings.

This part of the study demonstraites next step within the framework of close-loop circularity concept, namely the selection of suitable manufacturing technique for the preparation of product, i.e. in our case composites.

4. The PP-PCCF-10% wt. and PP-PCPESF-10% wt. exhibited highest value of crystallinity (44%) in comparison with pure PP (42%). However, in general, increase in fiber loading decreases the value of crystallinity.

5. Among the manufactured composites, the PP-PCPESF 10% wt. composite exhibited highest values of tensile strength (29 MPa) and strain (10%). The addition of fibers in PP matrix decreases tensile and flexural strain, as well as the tensile strength decrease was observed. In opposite, flexural strength value was not affected by fiber loading and even increase in flexural strength value for the PP-PCCF 10% wt. and PP-PCCF 40% wt. composites was found. Increase modulus of elasticity was found for PP-PCCF and PP-PCPESF composites with increasing of fiber loading. Flexural modulus of elasticity increased with fiber loading increase for all composites.

The PP-PCCF 10% wt. composite shows the best mechanical properties (except tensile strain and strength and flexural strain) among the fabricated composites. The higher degree of crystallinity (PP matrix phase) and good mechanical properties of cellulose as a main structural element of cotton are responsible for good mechanical properties of the PP-PCCF composites. This composite shows higher modulus of elasticity (1476 MPa) and flexural modulus of elasticity (2069 MPa) than for pure PP 1226 MPa and 1172 MPa, respectively. Additionally, the value of impact energy of the pure PP, PP-PCCF 10% wt. and PP-PCCF 40% wt. composites were 4.9 kJ/m^2, 4.8 kJ/m^2 and 5.5 kJ/m^2, respectively. In other words, PP-PCCF composites show good toughness.

6. The higher abrasive sliding wear rates against silicon carbide sandpaper were found for all composites in comparison with pure PP (3.09×10^{-6} mm/Nm). Among the manufactured composite the smallest wear rate was found for PP-PCCF 10% wt. composite (3.54×10^{-6} mm/Nm). As well as the COF value for all composites was higher in comparison with pure PP.

In the case of erosive wear (silica particles), the highest wear rate was found for the PP-PCCF composites and the smallest for PP-PCPETF (2 mm^3/kg) in comparison with PP (9 mm^3/kg).

7. It can be suggested that PP-PCPESF composites can be used in the area where ductile properties play important role.

PP-PCCF composites can be used for the dynamical applications in the area where reasonable load is applied and higher stiffness and toughness are required. The suitable examples are applications in construction, automotive and medical industries.

PP-PCPESF and PP-PCPETF composites are suitable for application in environment with erosion, for instance some parts for the cars.

The composite with higher load of fibers can be used for the static applications. Tableware, marine boats, electrical fittings and domestic appliances are possible examples.

The parts (4-7) of conclusions can be considered as a preparation for the next stage of the close-loop manufacturing and circularity concept, namely final product preparation and utilization of product by user.

For further research, it could be reasonable to use binders. The binder can increase matrix-reinforced material adhesion, crystallinity and fracture toughness. Fatigue and creep tests can also be proposed for analysis of the composites. Modeling can be utilized to predict the properties of the composites.

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Acknowledgements

PHD is a journey. I would like to express my gratitude to my supervisor Vitali Podgursky and my co-supervisor Dmitri Goljandin. This thesis and related articles were not possible without their support. I am thankful to Vitali Podgursky for enabling me to join Taltech, proffering me full independence in research, refining technical ideas, writing scientific articles and enormous support during my PHD. I am also grateful to Dimitri Goljandin to provide me the same guidance and technical support.

I am extremely delightful to Maksim Antonov for his marvelous technical support and blessed hardworking nature. He enabled, inspired and motivated me to create this innovative PHD thesis and articles.

I express my congenial acclamation to Illia Krasnou for competitive collaboration, experimentation, testing, data processing.

I desiderate to manifest my applause to Mart Viljus for SEM characterization and technical discussions.

I am thankful to the management staff of department and Taltech University i.e., Jakob Kubarsepp (Professor), Priit Kulu (Professor) Kristo Karju (director), Fyodor Sergeev (dean), Irina Hussainova (Professor), Juri Majak (Professor), Ilona Oja Acik (Professor), Andres Krumme (Professor), Rocio Estefania Rojas Hernandez, Andres Trikkel, Can Rüstü Yörük for their direct or indirect support.

My professors outside Taltech also help me directly or indirectly. Special thanks go to Dr. Tahir Ahmad (Professor, PU Lahore), Dr. Muhammad Abdul Basit (Associate Professor, IST Islamabad), Prof. Dr. Salim Iqbal Alvi (Dean, LLU, Lahore), Dr. Muhammad Mujtaba Abbas (Assistant Professor, UET LHR), Dr. Muhammad Rizwan Awan (Assistant Professor, SU LHR) and Muhammad Mubashar Farid (Lecturer, SU LHR).

Estonia and Tallinn University of Technology are my second home. I am greatly thankful for providing funding for milestone of my PHD, research exposure, conferences and innovative Environment.

The social circle of Taltech is also deserved my special thanks. A big thanks to my teachers, friends and colleagues Asad, Nikhil, Rahul, Naveed, Javad and Ramin for technical discussions.

A special thanks to scientific, research, engineering, technical and innovative institutes around the globe: UT Estonia, PU Lahore, Aalto university Finland, LLU Lahore, Superior University Lahore, UET Lahore, BZU Multan, Dongguk University South Korea, KAIST South Korea, BUCT China, Donghua University China, UM Malaysia, UPC Spain, Qatar University Qatar, WSU USA, NTNU Norway, UC New Zealand and University of Wrocław Poland.

My PHD thesis is dedicated to my late MOTHER and elder sister. I am indebted to my elder brother and sisters for their stunning support. I express my gratitude to my younger brother. I am thankful to my father and grandfather to molding me in this profession for mankind.

I acknowledged the support of project KIK 19019, GSFMT, European Union, Tallinn University of Technology, Estonian Academy of Sciences as well as Estonian Ministry of Science. I also acknowledged the financial support from the Estonian Research Council grants (PRG643), EIT Raw Materials (“RENEW” VHE22005), and the M-ERA.Net project (“DuplexCER” MNHA22040).

Abstract

Development of Sustainable Polypropylene Based Composites

The increase in global demands and utilization of polymeric engineering materials produces a variety of waste. These waste materials exist in various forms, like pre-consumer, post-consumer, end-waste, and end-of-waste. Normally, wool, cotton, polyesters, polypropylene, *etc.* are commercially recycled using different processing techniques and models. The circularity concept was proposed for transformation of engineering polymeric waste into sustainable products of high quality. The processing of polymeric waste consists of different steps like collection, sorting, separation, grinding, mixing, thermoforming (compression and injection molding), testing of properties.

The present study novelty includes manufacturing and investigation of different types of composites produced from waste. The natural fibers like cotton were produced from T-shirt. Synthetic fibers like synthetic polyester (PES) medium type woven fabrics were produced from T-shirts as well, and polyethylene terephthalate (PET) from beverage bottles. As a composite matrix, the polypropylene (PP) was used as a material with the good mechanical and thermal properties. The composites were thoroughly investigated using different methods, including DSC, TGA, surface roughness, tensile, bending, impact, abrasion, and erosion tests.

The main results suggest that composite with natural fibers, i.e., PP-PCCW shows most advanced mechanical properties, i.e. stiffness and toughness. Superior properties of cellulose as a main component of cotton and high degree of crystallinity of the PP phase can be an explanation of achieved results. On the other hand, the best ductility was observed on PP-PCPESF and the highest erosion resistance was found for PP-PCPETW composites. In addition, it was found that cutting tools coated with the TiAlN and TiCN coatings are suitable for the cutting of PCCF waste.

In the present study, the investigation was planned and realized following the circularity close-loop approach including next stages: collection, separation, grinding, extrusion, injection molding and quality testing. The following Figure briefly demonstrates the stages of close-loop recycling.

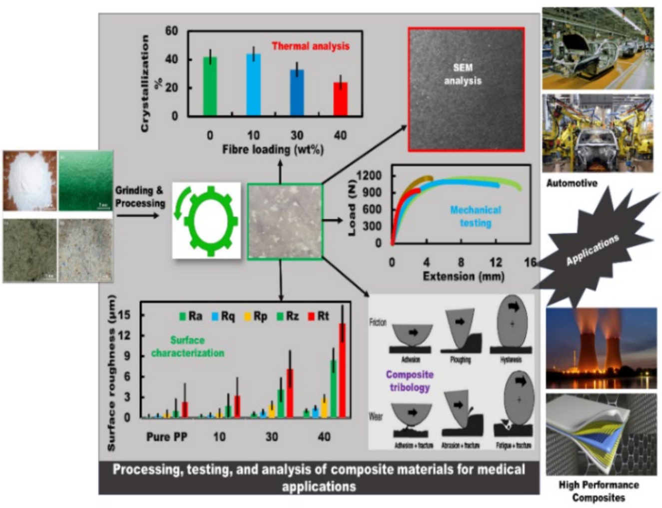


Figure. Graphical abstract of the present study.

Lühikokkuvõte

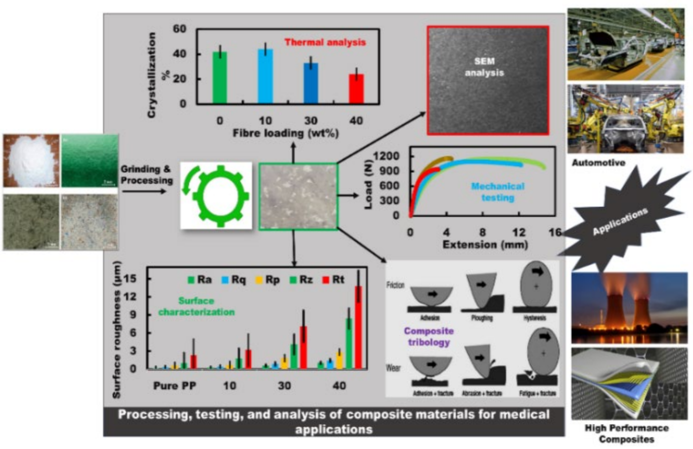
Polüpropeeni baasil jätkusuutlike komposiitide arendus

Kogu maailmas kasutatakse üha enam polümeerseid tehnomaterjale, mis omakorda tekitab mitmesuguseid jäätmeid. Need jäätmematerjalid esinevad erinevates vormides, nagu eeltarbimis-, järeltarbimis-, lõpp- ja lõppjäätmejärgsed jäätmed. Tavaliselt töödeldakse vill, puuvill, polüestrid, polüpropüleen jne kaubanduslikult ümber, kasutades erinevaid töötlemistehnikaid ja -mudeleid. Polümeersete tehniliste jäätmete muutmiseks kõrge kvaliteediga jätkusuutlikeks toodeteks pakuti välja ringmajanduse kontseptsioon. Polümeersete jäätmete töötlemine koosneb erinevatest etappidest nagu kogumine, sorteerimine, separatsioon, jahvatamine, segamine, termovormimine (survevormimine ja survevalu) ning omaduste testimine.

Käesoleva uuringu puhul on uudne jäätmetest toodetud erinevat tüüpi komposiitide tootmine ja uurimine. Looduslikud kiud nagu puuvillakiud saadi T-särkidest. T-särkidest pärinesid ka sünteetilised kiud nagu sünteetiline polüester (PES). T-särkidest saadi ka keskmist tüüpi kootud kangast ja joogipudelitest polüetüleentereftalaati (PET). Komposiitmaterjalide maatriksina kasutati polüpropüleeni (PP) kui heade mehaaniliste ja termiliste omadustega materjali. Komposiite uuriti põhjalikult erinevate meetoditega, sealhulgas diferentsiaalne skaneeriv kalorimeetria (DSC), termogravimeetriline analüüs (TGA), pinnakareduse, tõmbe- ja paindetugevuse, löögisitkuse mõõtmine ning hõõrd- ja erosioonkulumise uuringud.

Peamised tulemused näitavad, et looduslike kiududega armeeritud komposiitidel, st polüpropüleenist tarbimisjärgsetel puuvillakiududel (PP-PCCF), on kõige paremad mehaanilised omadused, nimelt jäikus ja sitkus. Tselluloosi kui puuvilla põhikomponendi suurepärased omadused ja PP-faasi kõrge kristallilisus võimaldavad saavutatud tulemusi selgitada. Teisest küljest täheldati parimat plastilisust PP-PCPESF-i puhul ja kõrgeim erosioonikindlus oli PP-PCPETF-i komposiitidel. Lisaks leiti, et TiAlN ja TiCN katetega lõikeriistad sobivad PCCF jäätmete lõikamiseks.

Käesolevas uurimuses kavandati analüüs ja viidi see läbi, järgides ringmajanduse suletud ahela lähenemist, sealhulgas alljärgnevaid etappe: kogumine, eraldamine, jahvatamine, ekstrusioon, survevalu ja kvaliteedikontroll. Alljärgneval joonisel on lühidalt näidatud suletud ahelaga ringlussevõtu etapid.



Joonis. Käesoleva uurimuse graafiline kokkuvõte

Extended list of publications

I. **Abrar Hussain**, Podgursky V, Goljandin D, Viljus M, Antonov M, Bogatov A, Krasnou Tribological and mechanical properties investigations of post-consumer cotton textiles. In Solid state phenomena 2021 (Vol. 320, pp. 97-102). Trans Tech Publications Ltd. doi.org/10.4028/www.scientific.net/SSP.320.97.

II. **Abrar Hussain**, Podgursky V, Goljandin D, Antonov M, Basit MA, Ahmad T. Mild steel tribology for circular economy of textile industries. Tribology in Industry. 2021;43(4):552. DOI: 10.24874/ti.1050.02.21.04.

III. **Abrar Hussain,** Podgursky, V., Antonov, M., Viljus, M., and Goljandin, D., 2021. TiCN coating tribology for the circular economy of textile industries. Journal of Industrial Textiles, 50 (1), 1-13, doi.org/10.1177/15280837211025726.

IV. **Abrar Hussain**, Podgursky V, Goljandin D, Antonov M. TiAlN coatings tribology for textile machinery parts. Proceedings of the Estonian Academy of Sciences. 2021 Apr 1;70(2). https://doi.org/10.3176/proc.2021.2.04.

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VI. **Abrar Hussain**, Podgursky V, Antonov M, Abbas MM, Awan MR. Tungsten carbide material tribology and circular economy relationship in polymer and composites industries. Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications. 2022 Oct;236(10):2066-73. doi.org/10.1177/1464420722109692.

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VIII. Awan, M. R., Rojas, H. A. G., Benavides, J. I. P., Hameed, S., **Hussain, A**., & Egea, A. J. S. (2022). Specific energy modeling of abrasive cut off operation based on sliding, plowing, and cutting. *journal of materials research and technology*, *18*, 3302-3310.

IX. **Abrar Hussain** and Muhammad Mujtaba Abbas. Role of Experimental Damage Mechanics for the Circular Economy Implementation in Cotton Industries. *JMN.* 2021. Vol. 1(1). DOI: 10.53964/jmn.2021004.

Sustainable Nuclear Energy Production

I. **Abrar Hussain** and Muhammad Mujtaba Abbas. A Review of Elemental Mass Origin and Fundamental Forces Unification for Nuclear and Aerospace Industries. *JMN.* 2021. Vol. 1(1). DOI: 10.53964/jmn.2021002

Collaboration Regarding Agriculture, Hospital waste, renewable clean technologies, and automotive energies production

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Appendix

Publication I

Hussain, A., Kamboj, N., Podgurski, V., Antonov, M., & Goliandin, D. (2021). Circular economy approach to recycling technologies of postconsumer textile waste in Estonia: a review. *Proceedings of the Estonian Academy of Sciences*, *70*(1), 80-90. doi.org/10.3176/proc.2021.1.07.

Proceedings of the Estonian Academy of Sciences,
2021, **70**, 1, 80–90
https://doi.org/10.3176/proc.2021.1.07
Available online at www.eap.ee/proceedings

RECYCLING

Circular economy approach to recycling technologies of post-consumer textile waste in Estonia: a review

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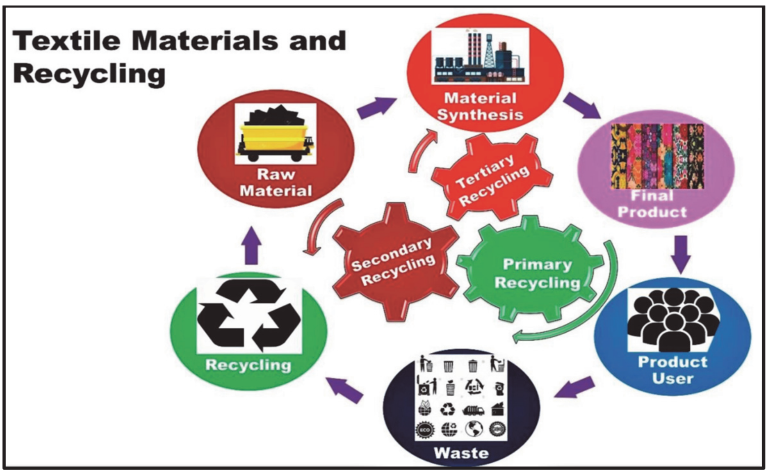
Received 31 August 2020, accepted 9 November 2020, available online 3 February 2021

Graphical abstract:



Abstract. Circular economy and recycling of post-consumer textile waste is gaining momentum. Its major obstacle is low-quality recycled products. This review article analyses commercial post-consumer textile materials, their recycling and applications. Modernization of fibre processing and recycling technology has assumed an indispensable role in the quality enhancement of post-consumer products. A futuristic overview of fabric materials, their processing, recycling and applications is presented by the example of commercial polymers. Different types of recycling – primary, secondary, tertiary, quaternary, and biological – used with ultramodern compatibilization and cross-linking are explored. Additionally, the conventional and proposed "Just-in-Time" (JIT) remanufacturing and recycling technologies for enhancing circular economy are demonstrated.

Key words: circular economy, post-consumer textile waste, textile recycling, waste management, end-waste, Just-in-Time manufacturing, end-of-life waste.

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1. INTRODUCTION

Circular economy is an industrial system that manufactures products with negligible waste [1]. It originates from the theory of a common industrial system [2], cradle to cradle recycling [3] and performance economy [4]. It has appeared as an innovative solution for the transformation of textile waste into valuable recycled products.

The global need for textile products and goods is climbing constantly. Generally, 63% of textile materials are procured from petrochemical products and 37% are extracted from natural resources [5]. Secondary textile material processing, e.g. weaving, knitting or spinning, as well as dying, finishing or printing treatments are considered to be major origins of CO_2 emissions and other types of pollution [6,7]. These provocations draw attention to recycling of textile materials, which principally enhances their service life for practical applications [8].

Natural and synthetic fibres are a prime fount of textile materials. Cotton, wool, silk and hemp are vital natural fibres used for commercial or mercantile applications. Likewise, various thermoplastics and thermosetting materials are paramount sources of synthetic fibres [9]. The quantity of the yearly produced fibres (as illustrated in Fig. 1, [10]) could be re-utilized after their initial applications.

There are two primary types of waste – pre-consumer and post-consumer waste [11,12]. Pre-consumer waste appears due to defective samples and scrap. Similarly, post-consumer waste originates because of end-use or disposed products. Representative examples of these types of waste are packaging materials (bottles, bags, etc.), abandoned goods (cars, computers, cell phones, furniture, etc.), demolished materials in construction (pipes, carpets, insulation materials, etc.) and disposable items [13,14].

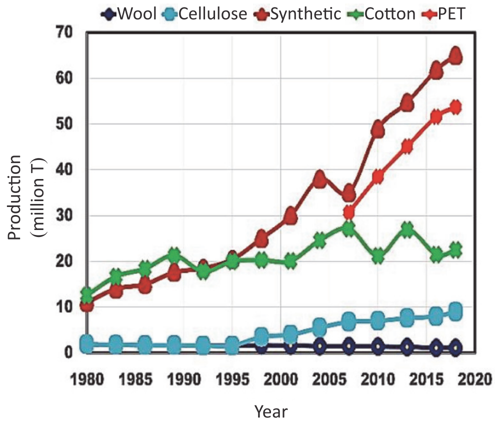

Fig. 1. Yearly quantity of world fibre production.

Recycling and value recovery from textile waste is achieved through new applications, sorting, purification, separation and sustainable processing of waste [15,16]. End-waste (EW), post-consumer (PC) and end-of-life waste (EOL) are recycled by primary, secondary, tertiary, quaternary, and biological recycling into useful products [16,17]. Polyesters (PES), polypropylene (PP), polyvinyl chloride (PVC), high-density polyethylene (HDPE),

Table 1. Commercial post-consumer waste and recycling technology

Waste type	Textile materials	Separation operation	Recycling process	Product
EW/PC/EOL	Polyesters, cotton, thermoplastics	Collection and sorting	Primary	Respective recycled product
EW	Scrap and defective materials (natural and synthetic fibres)	Manual separation and automatic sorting	Primary & secondary	Material recovery & recycled product
EW/PC	PVC, PET, PS, PP, PU	Electrostatic and magnetic separation	Tertiary	Material recovery
EW/EOL/PC	PVC, PET, PS, PP, PU	Compatibilization and cross-linking	Feedstock	Material recovery
EW/EOL/PC	Contaminated specified waste	Manual and auto	Incineration	Energy production
EW/EOL/PC	Contaminated waste	Compatibilization	Biodegradable/ quaternary	Material recovery and energy production

polyethylene terephthalate (PET), low-density polyethylene (LDPE), cotton, silk, hemp, wool and other thermoplastics are recycled commercially [18–20]. The details are shown in Table 1.

The objective of this review is to describe the processing of commercial textile waste by modern recycling technologies. A detailed description is given of the continuous recycling processing which generates a minimum amount of waste, contributing thus to circular economy and reducing textile waste. The relevant market assessment and the latest knowledge about fabric materials are also presented. Our review article focuses substantially, but not exclusively, on the recycling of textile waste that is accessible in enormous quantities or can be recycled precisely. The conventional and advanced methodologies, e.g. Just-in-Time (JIT) methodology, are also compared.

2. POST-CONSUMER TEXTILE WASTE IN ESTONIA

The total yearly utilization of textile materials in Estonia is more than 62 million tons. Of total waste, the potential for recycling is greater than 27 million tons. The entire volume of textile and apparel fabrics includes 27.212 million tons of local production, 12. 064 million tons of imported textiles for shops and user applications, and more than 22 million tons are used for export. The total material outflow and inflow capacities of Estonia are 27 million tons and 26 million tons, respectively [21].

An analysis on the composition of waste reports that municipal waste in Estonia contains 25% plastics and textiles, 3% wood, 2% ferrous metals, 1% non-ferrous metals, 1% paper, 1% glass, 1% rubber and 65% other rejected waste. Of the above-mentioned waste, 11% was used for recycling, 78% for energy production, and 11% termed as neutral waste was utilized for landfill [13,14]. Figure 2 illustrates the distribution of municipal waste in Estonia [22].

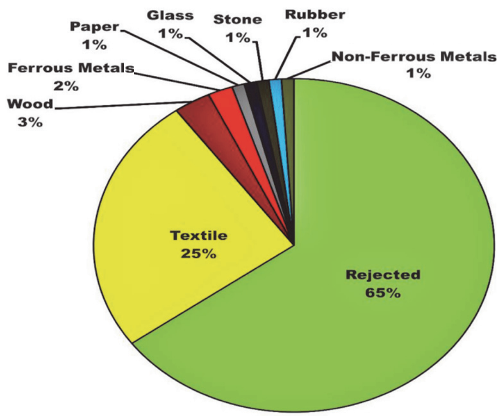

Fig. 2. Daily municipal waste distribution in Estonia.

3. TEXTILE MATERIALS

Textile materials can fundamentally be grouped into natural and synthetic materials. In textile engineering, fibre is a technical term specifying textile materials. Crops and animals are the principal origin of natural fibres [23,24]. As shown in Fig. 3, natural fibres such as cotton, hemp, sisal, coco, henequen, jute, kenaf, kapok and ramie are derived from plants. Silk, diverse hairs and wool fibres are procured from animals. Similarly, synthetic textile materials are classified into organic and inorganic textile materials. Plastics and thermosetting textile materials, for example, PVC, PE, PET, PS, HDPE, LDPE, viscous materials, modal materials and even composites, result in waste after their service life [25]. Formally, it generates end-waste, post-consumer waste and end-of-life waste. End-waste and post-consumer waste undergo primary and secondary recycling, respectively. Mostly, end-of-life polluted waste is processed through tertiary, biodegradable and incineration recycling to produce new products or energy [26,27].

4. RECYCLING TECHNOLOGY

Fibre technology is mainly concerned with apparel, industrial and home decoration applications. The reproduction of textile materials from their waste and transformation into a useful product is termed as recycling. Generally, recycling is categorized into primary, secondary, tertiary, and quaternary processing. Industrial byproducts and scrap are usually processed mechanically in primary and secondary recycling [28,29]. On the other hand, tertiary and quaternary recycling incorporates pyrolysis and burning of textile materials for energy generation. A wide range of textile materials are produced from natural and artificial fibres. These fibres, e.g. cotton, silk, nylon, rubber, and polyester will attain optimized strength, hardness, and abrasion or wear resistance when manufactured through knitting or weaving processes. Primary textile waste may be single or complex polymers that are usually easy to recycle. Post-consumer textile waste refers to fibrous materials that are discarded after use. The biodegradability of textiles depends on their natural materials. Generally, this waste is used for manufacturing clothes, carpets, belts, and composites

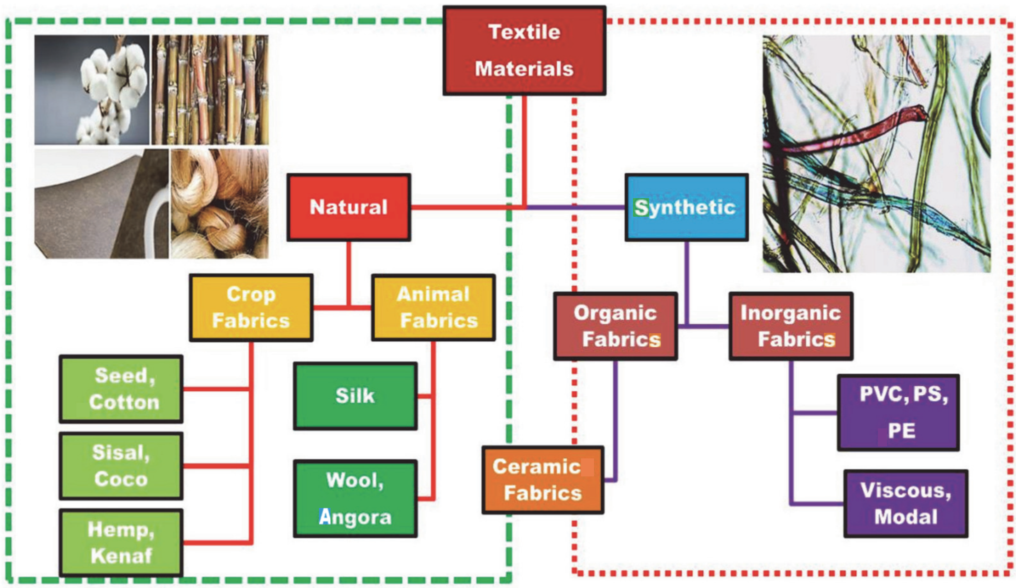

Fig. 3. Types of commercial textile materials.

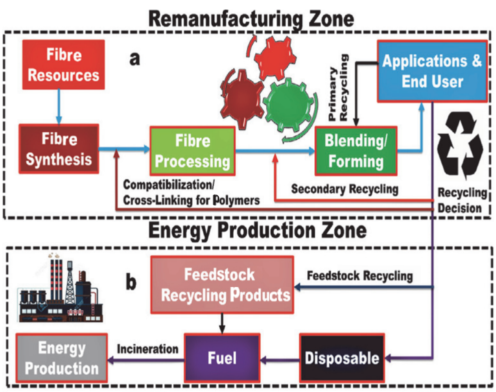

Fig. 4. Recycling flow chart for textile waste: (a) remanufacturing zone; (b) energy production zone.

[16,30–31]. Textile waste is usually shredded for reuse. Formally, automotive plastics, wool, silk, polyesters, polyethylene, and other plastic fabrics are easy to recycle when compared to thermosetting materials which cannot be re-melted or reshaped. Literature data of textile recycling and circular economy in the world is given in Table 2. The recycling flow chart proposed by the authors is demonstrated in Fig.4.

4.1. Textile waste separation and sorting

Commonly, the recycling of post-consumer waste is performed based on quality, availability, cost, the type of recycling process and the type of single or mixed textile materials [32, 33]. Before recycling of waste, the sorting process is carried out for textile material separation. Realff, Kip et al. [34,35] have developed a manual infrared spectrometer technique for wool, cotton, polyester, polyethylene, terephthalate, rubber, nylon 6,6 and other commercial polymers. In addition, a commercial automatic device was developed for separation of different textile materials. A piece of textile material is examined with the probe and transported via different tracks for identification of various fibres. The given data is estimated, analysed and displayed on the screen with the help of control units. The main objective of the separation and sorting is to separate different textile fabrics and the process is carried out either manually or preferably automatically. The recycling processing of textile waste is performed based on the sorting and separation results. After separation the waste undergoes size reduction and shredding [36].

4.2. Textile waste size reduction and shredding

After the sorting and separation process and before recycling, textile materials are shredded into small fibres and yarns. Fibres and yarns of different sizes are shredded with hardened metal blades that are attached to rotary drums. Smaller and oversize fibres and yarns are not allowed to enter the main chamber. Optimized cutting and rotational speeds along with suitable torque are used for mechanical shredding. Almost all types of commercial polymers and textile materials are shredded with modern optimized mechanical shredders. Usually, shredding is performed to enhance mixing, increase purity of the product and optimize the respective recycling process. After size reduction, the following recycling processes (introduced in subsections 4.3–4.7) are considered for the manufacturing of new products [37–41].

4.3. Primary recycling

Primary recycling, also known as closed-loop recycling, is the process of manufacturing of a new textile product from waste with desired properties [42]. Before recycling, some more processes such as collection and sorting of textile materials are required. Initially, the textile materials are crushed, ground, shredded, or milled. After that, the fine fibres or yarns are mixed with additives and other polymer materials. The elementary processing yields homogeneity and ease for purification or forming [50]. Usually molding, heat pressing, and extrusion are applied to polypropylene (PP), polyethylene (PE), polyethylene terephthalate (PET), and polyvinyl chloride (PVC) commercial thermoplastics for making different textile products. The primary recycling process can provide ease of production, impurity removal and stability to the product [51].

4.4. Secondary recycling

In secondary recycling processing, textile materials with unknown composition and purity are treated. Intially, some mechanical separation or purification treatments are employed to increase the purity. This purification and separation usually requires acids, additives, degassing and drying treatments which reduce the mechanical properties of textile materials [52]. In addition, contamination of the main polymer can decrease the mechanical properties of textile materials. Contamination increases due to the lowering of forming and blending compatibility or suitability with the matrix. Usually secondary processing is used for automotive, petrochemical and textile industries [43,53]. The composite is ground and the fine fibres are

Table 2. Literature data of textile recycling and circular economy in the world [43–50]

Serial No.	Author and publication year	Recycling process	Recycled materials	Recycled product
1	Esteve-Turrillas and de la Guardia (2017) [42]	Primary recycling	Cotton	Commercial production
2	Dahlbo et al. (2017) [43]	Shredding	Cotton, polyesters, cellulose	Fibres and yarns
3	Esteve-Turrillas and de la Guardia (2017) [42]	Cutting or shredding	Cotton	Fibres
4	Esteve-Turrillas and de la Guardia et al. (2017) [42]	Shredding	Cotton	Fibres
5	Zamani et al. (2017) [44]	Cutting and grinding	Elastane	Fibres and yarns
6	Beton et al. (2014) [45]	Primary and secondary recycling	Hemp, polyesters, cotton	Polymers and fibres
7	Glew et al. (2012) [46]	Primary recycling	Cotton, wool	Fibres
8	Palm et al. (2013) [47]	Primary recycling	Cotton, polyesters, cellulose	Fibres
9	Muthu et al. (2012) [48]	Primary and secondary recycling	Polyesters, polypropylene LDPE, HDPE, polyamide	Fibres
10	Williams et al. (2010) [49]	Primary recycling	Cotton and polyesters	Fibres

representative polymer materials for secondary recycling and are used for manufacturing of different automotive components. Commercially, different post-consumer textile and petrochemical materials such as PP, PE, HDPE, LDPE, PS, PET and PVC are mixed with each other in the presence of organic solvents like toluene, methanol and benzyl alcohol [54,55]. The nature of the secondary scrap, composition of the textile materials, purity grade of the final product, availability, types of additives, cost, and processing techniques are usually extensive factors which govern secondary recycling processing [56].

4.5. Tertiary recycling

Tertiary recycling (laboratory-scale technique) is also known as feedstock recycling. In tertiary recycling, hydrolysis, pyrolysis, gasification, condensation and hydrocracking are performed in the presence of transition metals [42,57]. Feedstock processing yields the monomers of polyurethane, PET, polylactic acid (PLA), polycarbonate (Poly.C) and wax by hydrolysis, condensation and gasification processing [58,59]. Other versatile and commercial plastics such as PP, PS, HDPE and PE are depolymerized by pyrolysis in the presence of transition metals [60]. The main shortcomings of tertiary recycling are that it constitutes only a subsidiary for industries, causes pollution and toxicity in terms of carbon or dioxin emissions, as well as high energy consumption [61]. Another major drawback is that it sets limits to large-scale production. Recently, in tertiary recycling a synthesis yielded nanotube production from PP, PS and PE polymer waste [62]. These aforesaid plastic polymers were transformed under reasonable conditions into respective carbon nanotubes. Sometimes transition metals are also employed for such a nanotube transformation process [63]. These newly formed nanomaterials have phenomenal applications in composites, electronic and biological industries [64].

4.6. Biodegradable recycling

Biodegradable recycling is especially performed for natural textile materials or fabrics. Special microorganisms and enzymes are utilized for degradation [65]. Hence, microorganism recycling is an advanced form of feedstock recycling. Diverse bacteria and fungi are applied under reasonable conditions [66]. Biological polymers such as cellulose, chitin, wood and hemp fibres are easily debased [5]. Moreover, organic compounds, e.g. butylene adipate-co-terephthalate, PET, polylactic acid (PLA), adipic dimethyl esters, 1,4-butanediol and hydrophilic lignocellulosic fibres, can be de- graded under equitable conditions [67].

4.7. Plastic incineration and quaternary recycling

Incineration is the process of producing heat and energy from post-consumer textile waste. This textile waste is highly contaminated. In Europe, the discarded waste is commonly utilized for energy and heat production. Medical and toxic waste are main raw materials for incineration. Inorganic waste is utilized mostly for road construction. The heat and energy are typically used for cement, metal melting and chemical heating applications [68,69].

5. EUROPEAN PROJECTS ON PLASTIC RECYCLING

The following information regarding projects on plastic recycling has been presented on the European Commission website [70]:

(1) German project LIFE00ENV/D/000348 for thermoplastics and wood fibre production. The product applications include house decoration;
(2) Danish project LIFE04ENV/DK/000070 for manufacturing of rubber products. Chemical additives and polymer monomers are utilized for the removal of impurities. The products are blended by using the dense phase technique. Sports and rubber industries are typical applications;
(3) German project L-FIRE undertakes the separation and recycling of Kevlar;
(4) Belgian project FP7-ENV-2010: IRCOW addresses materials recovery from construction and post-consumer waste;
(5) ECO/10/277225 SUPERTEX, SUPERCLEAN, DEVULCO2, PEGASUS and FP7-ECOMETEX are other similar innovative European projects on post-consumer textile waste, addressing the separation and recycling of automotive, polymer, chemical and electronic waste [27].

6. RECYCLED PRODUCT APPLICATIONS

In this section the commercial and economic aspects are presented. Generally, 24% of thermoplastics, thermosetting materials, coatings and textile fabrics of the world's production is manufactured in Europe. The major applications of post-consumers plastics include packages, products of electronics, textiles, construction and automotive industries. PS, PE, PET, PVC and nylon can be utilized to produce materials with HSLW (high-strength lightweight), high thermal and electrical resistance at a very low price.

Packaging materials account for 40% of the thermoplastic commercial applications. Usually, PET and PE are used. Construction materials, for instance, PVC (pipes, cables, electrical insulation), PO (carpets and textiles) and PU or PS (foams) supply 20% of the commercial applications. Generally, about 13% of the total volume of textile materials is utilized for underwear, outerwear, technical and industrial textile applications [13,14].

7. JUST-IN-TIME (JIT) MANUFACTURING MODEL FOR RECYCLING

JIT is a type of advanced manufacturing model based on continuous improvement and manufacturing. The raw materials which constitute mostly waste are delivered at the precise time they are required for the production process. In Fig. 5 the authors demonstrate the advanced model for recycling which enhances circular economy and reduces textile waste. First, the collected commercial post-consumer textile waste like cotton, polyester, cellulose, wool and PET are separated and sorted. The separation of post-consumer textile waste enables to decide immediately the desired process of recycling. Sorting and separation are applied to textile waste which has unknown purity, non-homogeneity in mixing, lower mechanical properties and difficulty in blending.

After manual separation, the good quality fabric is reused directly for suitable applications. It mostly includes outerwear and innerwear. Generally, the scrap, defective and low-quality textile waste with negligible impurity and contamination undergo primary recycling. Thus, EW and PC waste is widely recycled. If impurities and contamination account for more than 20% of a particular waste material, secondary recycling is performed to manufacture a new product. This post-consumer waste is utilized as raw materials.

Tertiary recycling is used purely for contaminated textile waste. Biodegradable and incineration recycling are performed for highly contaminated and toxic textile waste to produce a new product and energy, respectively.

JIT practices and infrastructure can improve the quality and performance. It reduces the time for recycling and the cost of remanufacturing. Since JIT involves continuous improvement of manufacturing, it adds value to manufacture excellence. Primary and secondary recycling mostly process end-waste and post-consumer waste. Therefore, our model suggests that the recycling units employing primary and secondary recycling technologies should be installed in pre-manufacturing

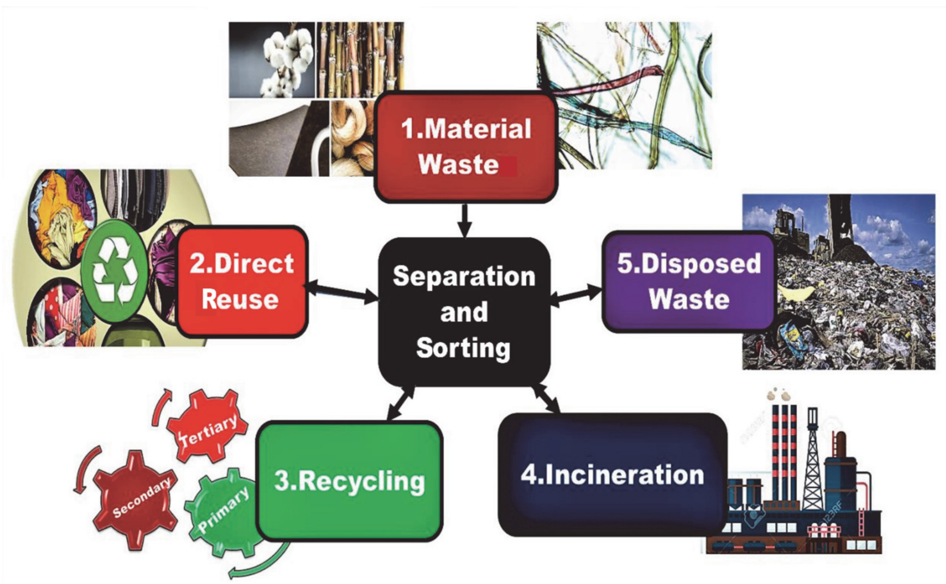

Fig. 5. Proposed JIT model for textile waste recycling.

Table 3. Terminology and abbreviations of textile materials

Abbreviation	Name of material	Abbreviation	Name of material
EPDM	Ethylene–propylene diene rubber	HDPE	High-density polyethylene
NR	Natural rubber	Poly.C	Polycarbonate
PE	Polyethylene	PET	Polyethylene terephthalate
PP	Polypropylene	PS	Polystyrene
PU	Polyurethanes	PVC	Polyvinyl chloride
EW	End waste	PC	Post-consumer
EOL	End of life waste	JIT	Just-in-Time
PES	Polyesters	LDPE	High-density polyethylene
PLA	Poly lactic acid	PAM	Polyamides

units. Tertiary recycling is still a laboratory technique. Quaternary and biological recycling mostly deal with contaminated and toxic waste. The recycling units designed for the latter should be installed outside cities and urban areas to reduce pollution and hazardous gas emissions.

8. CONCLUSIONS

The growing global demand for textile products leads to millions of tons of textile waste being disposed to landfills. A wide range of commercial post-consumer textile waste, for instance, cotton, wool, polyesters, PET, PVC, nylon, PS, and cellulose are transformed into new recycled products, mostly by using primary and secondary recycling technologies. Tertiary and biodegradable recycling technologies are mainly utilized for contaminated and polluted textile waste. Similarly, incineration can be used for disposed and even landfill waste.

In this review the authors have explored different authentic recycling technologies for circular economy. The commercial implementation of recycling technologies and the possible dependence on separation methods have been described in detail. The unsolved fundamental complications in forming, processing, collection, sorting, recycling, cost, and transportation have also been considered. Additionally, the formal and proposed models for recycling technologies to improve the quality of recycled products and reduce textile waste have been compared.

ACKNOWLEDGEMENTS

This study was financially supported by the project KIK 19019 "Development of textile waste shredding technology and innovative materials to add value to textile waste and to support circular economy". The publication costs of this article were covered by the Estonian Academy of Sciences.

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Ringmajanduslik lähenemine tarbijajärgsete tekstiilijäätmete ringlusse võtu tehnoloogiatele Eestis: ülevaade

Abrar Hussain, Nikhil Kamboj, Vitali Podgurski, Maksim Antonov ja Dmitri Goliandin

Ringmajandus ja tarbimisjärgsete tekstiilijäätmete ringluse maht kasvavad pidevalt. Tihti on tõsiseks takistuseks taasringlusse võetud toote halb kvaliteet. Selles ülevaateartiklis on analüüsitud tarbijale mõeldud kaubanduslikke tekstiilmaterjale, nende ringlusse võttu ja rakendusi. Kiudude töötlemise ja taaskasutuse tehnoloogia moderniseerimine on tarbimisjärgsete toodete kvaliteedi parandamisel muutunud asendamatuks. Kangasmaterjalide töötlemise, taaskasutuse ja nende rakenduste futuristlikku ülevaadet on illustreeritud kaubanduslike polümeermaterjalidega. Esmase, sekundaarse, tertsiaarse, kvaternaarse ja bioloogilise taaskasutuse erinevaid liike rakendatakse koos tänapäevaste ühilduvuse ning ristsidemete protsessidega. Lisaks on ringmajanduse edendamiseks esile toodud tavapäraseid ja väljapakutud "Just-in-Time"-i (täpselt õigel ajal) ümbertöötlemise ning taaskasutuse tehnoloogiaid. On tõestatud, et tavapärased ja väljapakutud "Just-in-Time"-i (JIT) ümbertöötlemise ning taaskasutuse tehnoloogiad edendavad ringmajandust.

Publication II

Hussain, A., Podgursky, V., Viljus, M., & Awan, M. R. (2023). The role of paradigms and technical strategies for implementation of the circular economy in the polymer and composite recycling industries. *Advanced Industrial and Engineering Polymer Research*, *6*(1), 1-12. doi.org/10.1016/j.aiepr.2022.10.001.

Advanced Industrial and Engineering Polymer Research 6 (2023) 1–12

Contents lists available at ScienceDirect

Advanced Industrial and Engineering Polymer Research

journal homepage: http://www.keaipublishing.com/aiepr

The role of paradigms and technical strategies for implementation of the circular economy in the polymer and composite recycling industries

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A R T I C L E I N F O

Article history:
Received 21 June 2022
Received in revised form
10 October 2022
Accepted 12 October 2022

Keywords:
Circular economy
Recycling
Polymer waste
Mechanical testing
Tribology

A B S T R A C T

The circular economy (CE) is facing a problem of industrial implementation. The models are proposed for natural types of polymers and CE. The current work reports the technical strategies and paradigms for sustainable close-loop manufacturing. The post-consumer cotton polymer is used for optimization of functions like cost analysis, material evaluation, recycling technique selection, and quality control. The presentation of the results is advanced by break-even analysis, mechanical testing, tribological investigations, and polymer surface evaluations to select a reasonable processing technique. The current experimental and theoretical approaches could be used functionally for the adaption of the circular economy in polymer industries. The article also highlights the possible procedure for sustainable production of polymer products from natural and artificial wastes.

1. Introduction

The word circular economy (CE) was first used in 1962 by the European Union (EU). Technically, CE is a collection of economic, technical, management, and quality testing [1–4]. The complexity and industrial implementation are a challenge of the twenty-first century to produce sustainable products with minimal material waste. The CE transforms the take-make-dump (open loop) system into a closed-loop system [5–7]. The dynamic transformation is based on paradigms of cost analysis, material analysis, management, and quality testing. The cost analysis is carried out before research and development to check the suitability of materials for processing. The materials analysis controls the processing, quality, performance, commercial application, and expected purity of the product [5,8,9]. The reasonable remanufacturing technique along with suitable testing procedure helps CE to emerge as an excellent solution to the ubiquitous challenges [1,9–12]. Therefore, CE presents a restorative and regenerative design for industries [13]. On the other hand, CE also provides considerable attention to accommodate the ecological systems (microsystem, macro system, chrono system) and economic growth (land, labor, capital and entrepreneurship) [14,15]. Microsystems impart cleaner production (sustainable and green technology), suitable production design and customer satisfaction. The CE is still considered a theoretical field with a set of strategies. The set of strategies only associate CE with the cradle to cradle (C2C) concept [16,17]. The C2C practically assists the circulation of polymer and composite materials for service life. The C2C assesses open systems industrially based on materials analysis, cost evaluations, energy, reutilization, and quality management testing. The industrial implementation of CE starts with cost analysis of recycling [18,19].

Currently, process costing, and job order costing are two modern techniques for manufacturing cost evaluation and determination [20]. The process and job order costing [21,22] are employed for homogeneous [23], continuous [24], and heterogeneous productions [25]. Simply, cost analysis of polymer recycling is equal to the sum of all types of costs that are required to transform the raw material into a product using the optimum recycling process [26–28]. The synthetic and natural polymers are extracted from petrochemical, plant, and animals [29]. These natural fibers [30,31] have huge potential use in modern reinforcement composites materials [32]. Similarly, polyesters (PES), polyethene (PE), poly(ethylene terephthalate) PET, 2-methylbut-1, 3-diene, poly(azanediyladipoylazanediylhexane-1, 6-diyl), poly(1-phenylethene-1, 2-diyl), poly(bisphenol A carbonate), poly(propene), and others are famous commercial synthetic

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https://doi.org/10.1016/j.aiepr.2022.10.001

Abbreviations:

CE	Circular Economy	Z	Initial amount of money
EU	European Union	C2C	Cradle to cradle (C2C)
PE	Polyethene	PES	Polyesters
SEM	Scanning electron microscope	PET	Poly (ethylene terephthalate)
SNK	Student Newman procedure	ISO	International Organization for Standardization
R_p	Maximum profile peak	R_a	Arithmetical mean height
R_t	Total height of profile	R_z	Maximum height of peak
ASTM	American Society for Testing and Materials	μm	Micrometer
COF	Coefficient of friction	mm	Millimeter
mm/s	Millimeter per second	UMT	Universal mechanical tester
m	Meter	N	Newton
TiCN	Titanium carbo-nitride	Zr_2O_3	Zirconia
cm	Centimeter	AISI	American Iron and Steel Institute
T/m	Twist per meter	g/m^2	Grams per meter
F	Load	VMHT	Vickers micro hardness tester
d	Diagonal length	HV	Vickers hardness
MgO	Magnesium oxide	X	Symbol for magnification in scanning electron microscope
°C	Degree Celsius	K	Kilo
MPa	Mega Pascals	G	Grams
BEA	Break even analysis	%	Percentage sign
FC	Fixed cost	TC	Total cost
BEP	Break even point	VC	Variable cost
		Y	Number of product or project units

polymers used for common, engineering, and technical applications [33]. In current decades, different types of reinforcement composites have been developed from virgin polymer and waste materials. Therefore, cost analysis is an advanced tool for planning and determining the success of recycling projects [34–36].

In recycling development, the operational strategies and functional paradigms of CE are instigated to reduce the consumption of material resources [37]. Recycling like primary, secondary, tertiary, and polymer incineration are established to recycle, manufacture, and energy production from respective wastes [38]. Open-loop polymer manufacturing is transformed to closed-loop processing to enhance the quality and performance of sustainable recycled products. The growth of the circular economy in polymer industries is getting momentum due to excellent consolidation of mechanical, tribological, ease of reshaping and fabrication [39,40]. The extrusion, molding, calendaring, powder metallurgy, thermoforming and additive manufacturing as primary processing often impart reasonable rigidity and toughness for many commercial applications [41]. The thermosetting polymers are generally not recycled due to their high brittleness and rigidity. Thermoplastic polymers are usually recycled due to elasticity, plastic deformation, and low rigidity. Genuinely, the performance and quality of the product developed from initial processing, whether the polymer product is a smart textile or a strategic polymer [41,42]. Therefore, the capacity to transform a product's tactile character into optimized quality can modernize product design [43–46].

In this manuscript, the CE strategies, paradigms, cost analysis, material testing, and optimum recycling are evaluated for the first time for industrial applications to produce sustainable and green products. A framework is advanced to address this issue regarding the polymer and composite industries. A newly developed method was used to demonstrate optimization of texture, abrasiveness, slipperiness, fuzziness, and tribological properties. The scanning electron microscope, optical and mechanical profilometer, and tribometer were used for surface characterization and tribological investigations. The surface roughness of metallic and polymer surfaces was measured and commensurate with scanning electron microscope characterization results. Finally, the concept of low surface roughness, higher metallic hardness, metallic and polymer surfaces with minimal surface defects is introduced to gain customer satisfaction.

2. Experimental work

2.1. Materials

Natural polymers are emerging as a replacement for synthetic materials due to their excellent physical and chemical characteristics. The natural polymer wastes are categorized into various types, see Fig. 1 The bast (kenaf, hemp, jute, cotton, coir), plant straws (corn, wheat, rice), leaf fibers (sisal, henequen, pineapple), grass fibers (bamboo fiber, switchgrass, elephant grass), and wood fibers (soft and hardwood) are major types of natural fibers. The plant fibers include silk, wool, angora, alpaca, bison, cashmere, mohair, and qiviut for commercial applications.

The post-consumer cotton polymer was selected for investigations. The subjective assessment of the cotton fabric is shown in Table 1.

2.2. Scanning electron microscopy

The surface analysis of polymer wastes, metallic materials, coatings, and tested samples were analyzed using scanning electron microscope (SEM) (Zeiss EVO® MA - 15 system, Oberkochen, Germany) with LaB6 cathode in secondary electron mode, applying an accelerating voltage of 10–15 kV and at 6.5–8.5 millimeters (mm) working distance.

2.3. Surface roughness measurements

The surface roughness of metallic and polymer surfaces was investigated using contour GT- K 3D optical microscope and

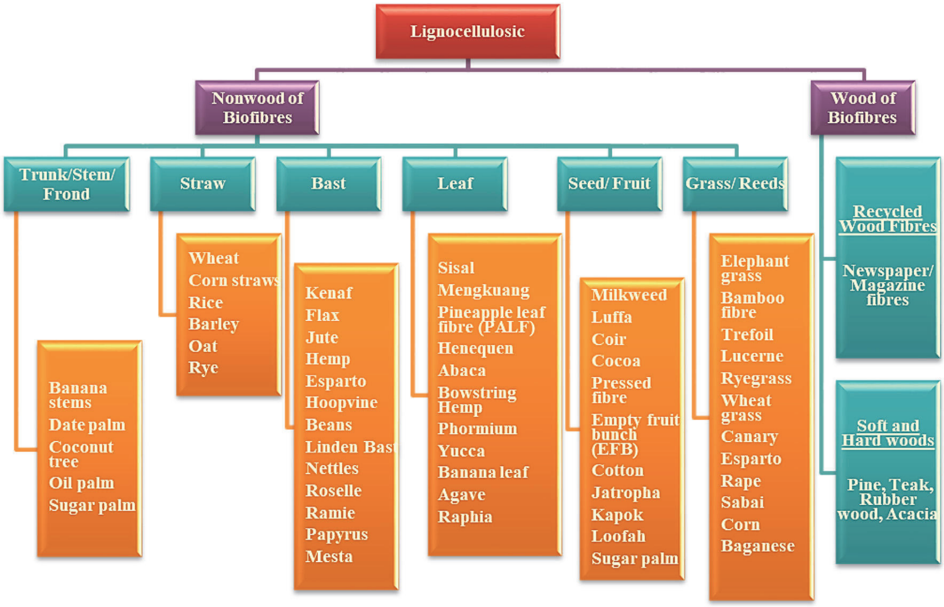

Fig. 1. Proposed types of natural polymers waste.

mechanical profilometry (Mahr Perthometer). The investigations were performed before and after tribological testing. The testing was done using the International Organization for Standardization (ISO 4287-1997) standard and Student Newman Procedure (SNK). The surface roughness parameters like arithmetical mean height (R_a), maximum profile peak (R_p), maximum height of peak (R_z) total height of a profile (R_t) was calculated. Additionally, green monochromatic laser light with 275 micrometers (μm) back scanning and 300 μm lateral scanning length was allowed to interact with samples.

2.4. Mechanical testing of cotton waste

The strip test American Society of Testing of Materials (ASTM D5035-95), grab test (ASTM D5034-95), tensile test (ASTM 4878.6-2001), tear test (ASTM D1423-83), burst test (ISO 3303-1995), universal mechanical test (ASTM D3039), bend test (ASTM D5467) and compression test (ASTM A370) is used to investigate the mechanical, flexural, bending and impact properties of thermosetting, thermoplastic polymers and composites materials [47,48].

Instron mechanical testing machine (Model 5860) was used to perform tensile tests. The upper and lower jaws have a width of 25 mm. The speed of the lower jaw and gauge length were 50 mm/min and 100 mm. The post-consumer cotton samples of dimensions of 180 × 100 mm and 180 × 50 mm were selected for grab and strip testing, respectively. Moreover, Bluehill universal material testing software was utilized to measure the tensile force, breaking force, and elongation during data analysis.

2.5. Tribological testing

The cotton fabric was cut into 25 (width) × 50 (length) mm rectangular strips. The polymer strips were mounted on mild steel (dimensions of 25 (width) × 10 (thickness) × 50 (length) mm) blocks to provide stiffness during tribological testing [49]. In our previous research work, our group reported a new innovative method for coefficient of friction (COF), wear, abrasion, and deformation evaluations of polymer materials. The CETR/Brucker universal macro materials testing device (UMT-2) was used for tribological investigations. The tribometer can slide and reciprocate with 0–200 N (N) force and 0.5–15 mm per second (mm/s) speed on a polymeric surface. However, the tribological testing was performed for 0–80 meters (m) sliding distance, 0.5–9 N force, 1–10 mm/s speed and 0–320 s time variations. Experimentally, the

Table 1
Subjective assessment of post-consumer cotton textile.

Physical Property	Unit	Value	Physical Property	Unit	Value
Woven-Weft	–	Plain	Thread diameter in weft direction	mm	0.345
Woven-Warp	–	Plain	Thread diameter in warp direction	mm	0.345
Weight	gm^{-2}	237	Twist value	T/m	800
Warp linear density	cm^{-1}	29	Thickness	mm	0.45
Weft linear density	cm^{-1}	29	Weft-Warp thread setting	cm^{-1}	18–36 Thread setting
Fabric thickness	mm	0.45	–	–	–

change in physical parameters was contemplated for optimization of COF regarding industrial applications [50]. Zirconia (Zr_2O_3) ceramic balls were used as a counter body for tribological evaluations [51]. Furthermore, American Iron and Steel Institute (AISI) 52100 steel balls, titanium carbonitride (TiCN) coated AISI 52100 steel balls and C10 alumina ceramic balls were purchased from Redhill company for COF optimization.

2.6. Micro-Vicker hardness testing

Vicker hardness tests were performed on Shimadzu type M and Leica mod, VMHT microhardness testers. The load was applied in the range of 25–5000 mN. The micro-hardness after testing was calculated as:

$$HV = 0.1891\frac{F}{d}$$

$$d = \frac{d_1 + d_2}{2}$$

where *HV*, *F*, and *d,* are Vicker hardness, load, and average diagonal length.

The hardness was calculated in a vertical direction with respect to the substrate. The loading and unloading correspond to elastic-plastic deformation of the material. The diagonal length was calculated using SEM.

3. Results and discussions

3.1. Scanning electron microscope characterization and surface damage

The purpose of cotton polymer surface characterization was to decide the quality of tactile properties. Therefore, surface evaluations were done at lower and higher magnifications. At lower magnification, SEM images Fig. 2(a) (*X* 50) and Fig. 2(b) (*X* 50) were shown the arrangement of polymer yarns from left to right (parallel weft) and bottom to top (perpendicular warp). The cotton polymer yarns and threads are composed of fibers. The longitudinal SEM micrographs manifest an irregular cylindrical shape [52] with diameters between 10 and 20 μm. The cotton fiber is made up of ordered microfibrils. These microfibrils are bounded together by hemicellulose [53]. The porous cotton fibers absorb humidity and impurities. These phenomena originated from three-dimensional random oriented fibers. Microscopically, these fibers generate unevenness and surface roughness [11,54]. Theoretically, these findings were reported as abrasiveness, fuzziness, texture, and slipperiness. The degree of fabric surface defects engendered bobs, joggling, micro pills, and distortion [55,56], see Fig. 2(c) and (d).

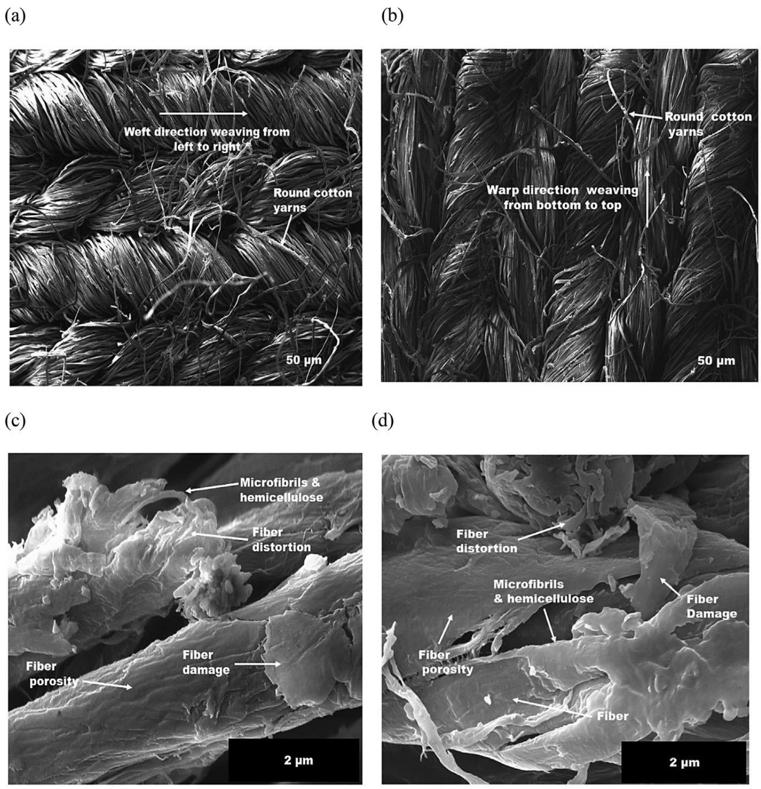

Fig. 2. SEM characterization of pure cotton post-consumer waste: (a) polymer yarn alignment in parallel (weft) direction, (b) polymer yarn alignment in vertical direction (warp), (c) fiber condition in weft direction (d) fiber condition in warp direction.

Table 2
Average metallic and cotton polymer roughness parameters.

Material	Device	Surface roughness parameters			
		R_a (μm)	R_p (μm)	R_z (μm)	R_t (μm)
Zirconia balls	Optical (non-contact method)	0.05	0.10	0.13	0.28
	Mechanical (contact method)	0.07	0.11	0.15	0.34
Cotton fabric polymer	Optical (non-contact method)	25.24	125.91	215.25	495.17
	Mechanical (contact method)	27.28	135.49	224.37	509.17

3.2. Surface roughness evaluations

Originally, for polymer fabrics, the surface roughness came into existence due to unevenness and irregular distribution of fabric yarns and threads [50,56,57]. Therefore, the values of cotton polymer surface roughness parameters in Table 2 are related to the degree of distortion and damage. Abrar et al. studied the surface roughness of post-consumer cotton polymer textile. The group had reported the R_a, R_p, R_z and R_t as 40.33, 260.57, 340.15 and 696.12 μm, respectively. The marginal increase in surface roughness parameters was reported due to the creation of new random oriented fibres. The fibres were produced due to physical and chemical impacts during the service life of cotton polymers [58].

Table 2 showed the average surface roughness parameters for zirconia balls. The zirconia balls have low surface roughness and a smooth surface. ISO 3290 G3 zirconia balls have 1240 HV10 of Vickers hardness, 6.0 gcm^{-3} of density, and 2400 °C of temperature resistance. Furthermore, zirconia balls are made up of 97% zirconia and 3% magnesium oxide (MgO). The balls have 0.53 kN mm^{-2} of rupture strength, 2 kN mm^{-2} of ultimate compressive strength, 0.05 kN mm^{-2} of ultimate tensile strength, 195 kN mm^{-2} of modulus of elasticity, and 10 MPa m^{-1} of fracture toughness. Higher hardness, low surface roughness, and reasonable mechanical properties provide better tribological properties. Moreover, these mechanical properties improve the wear resistance, corrosion resistance, erosion, and fatigue resistance of machinery parts during polymer processing [59]. Moreover, the SEM images of zirconia material are shown in Fig. 3(a) and (b). The SEM micrographs of zirconia show the same behavior before and after wear testing. Anyhow, some impurities, micro-scratches are detected on the ball's surface.

3.3. Experimental damage analysis through mechanical testing

The mechanical testing is generally performed during the processing of polymers and composite materials to assure commercial applications. The first time, the tensile grab and strip tests were performed on pure cotton polymer waste to evaluate the relationship between processing, recycling, and mechanical properties. The results of mechanical testing and SEM image characterization are shown in Fig. 4(a)–(d). The average tensile force, design force, breaking force, and extension were 180, 240, 150 N, and 28%. The tensile force is the maximum force that cotton fabric can bear during recycling. The higher value of tensile force refers to the good condition of cotton fabric for recycling [14,60–62].

3.4. Role of friction measurements in damage mechanics

The principal interaction between polymer and metallic machinery parts engenders operational problems during manufacturing. Sliding and Reciprocation principles are used to measure and optimize the coefficient of friction. The additional vital contemplation is the accomplishment of the developed manufacturing process to produce low friction polymer products for strategic, smart textile and, automobile applications [1]. The development and innovation in this field have been impeded due to the difficulty of establishing a relationship between tribological outcomes (tactical properties) and physical properties (super lubricity, low friction coefficient) [63]. The typical COF graphs of cotton polymer in warp and weft directions were depicted in Fig. 5(a)–(d). At start, the force, speed, and time have been varied to investigate COF. The results express that at a constant speed of 1 mm/s, in the case of Zirconia balls, for the value of force increasing

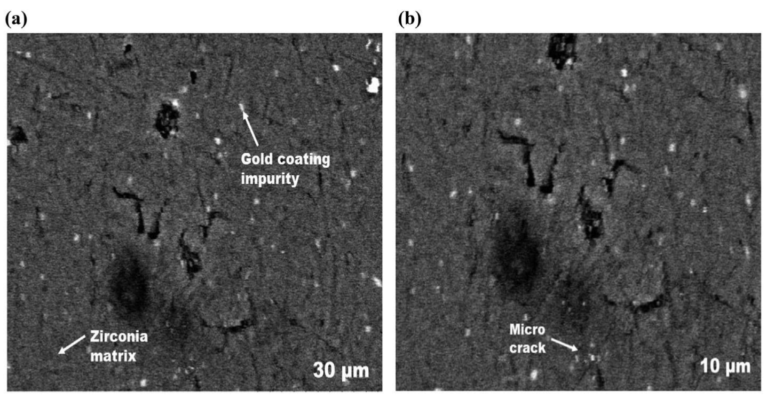

Fig. 3. SEM characterization of zirconia balls: (a) zirconia matrix and gold impurities and (b) micro-cracks on the metallic ball's surface.

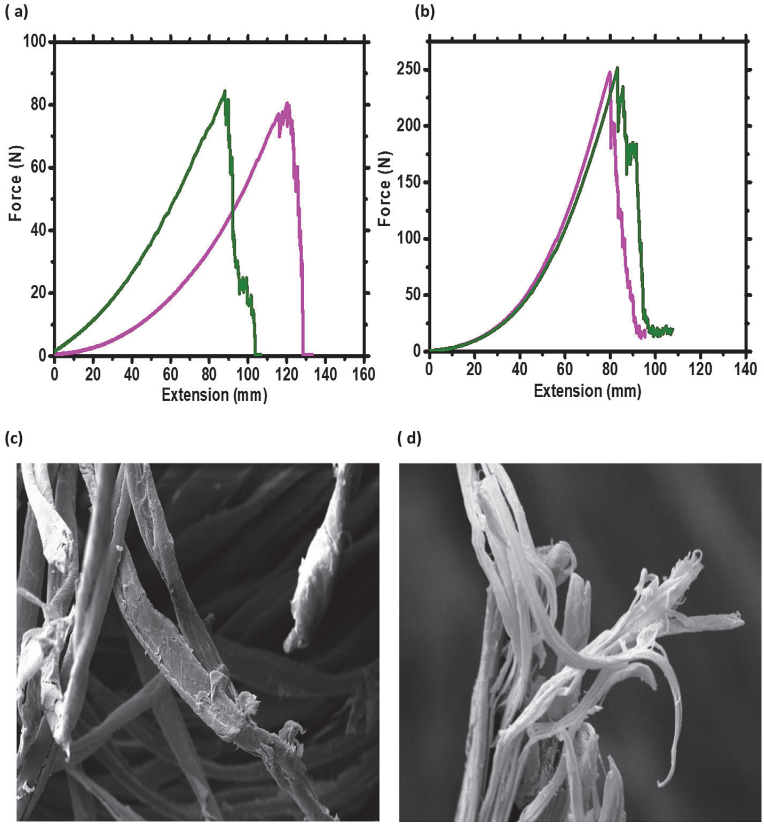

Fig. 4. Mechanical testing and SEM characterization of pure cotton post-consumer waste: (a) ASTM D5035-95 strip test, (b) ASTM D5034-95 grab test, (c) SEM micrograph of post-consumer cotton polymer after strip test and (d) SEM micrograph of post-consumer cotton polymer after grab test.

from 0.5 to 9 N, the COF value increases from 0.04 to 0.23 in warp and 0.05 to 0.18 in the weft directions. Correspondingly, at a constant speed of 8 N, in the case of zirconia balls, for the value of speed increasing from 1 to 10 mm/s, the COF value of cotton polymer increases from 0.15 to 0.17 in warp and 0.14 to 0.18 in weft directions [64,65].

The sliding motion was transformed to reciprocation motion for optimization [1,49,50,60]. The periodic linear abrasive path increased to 80 m to test COF, wear, and deformation. At a load of 8 N and 1 mm/s, for 80 m of sliding path, ISO 3290 G3 zirconia balls deformed cotton fabric polymer. There was no wear or abrasiveness detected during reciprocation. At the start, the COF values change almost exponentially with sliding distance. After 55 m of sliding distance, the COF became constant in warp and weft directions. This phenomenon is related to Fig. 6(a) and (b).

3.5. *A comprehensive analysis of surface distortion for polymer recycling industries*

Various appraisals and evaluations can be foreseeable using experimental results. The fabric descriptors and tactility (strategic properties) rely principally on fabric polymer mechanical properties, roughness, coefficient of friction, nature of metallic and polymeric materials. The cotton fabric polymeric material was chosen to enable the diversity of processing. The description of some other commercial coatings and materials responses as counter body (published by our group) is expressed in Table 3. The same experimentation was used for polymer COF, hardness, and surface roughness investigations [66].

The mechanical investigations express a reasonable role in the evaluation of waste material's response towards recycling. The recycling of polymer wastes generally includes the sorting, grinding, processing, and finishing steps for transformation into a green and sustainable product. The lowering of tensile, design, and breaking force is linked with distortion and bad conditions of polymer waste. These conditions create problems (tangling, buckling, surface wear) during recycling. However, an increase in the mentioned tensile properties enhances performance during recycling steps. The higher tensile values of cotton provide minimal surface damage, distortion, and surface roughness. The minimum surface defects provide better grip and recyclability for waste. Moreover, the subjective assessment (polymer weight, density, twist factor, composition, and weaving structure) express the reasonable influence on mechanical properties [1,50].

The surface roughness description and evaluation of fabric materials are quite different from metallic materials. The fabric surface roughness produced due to uneven orientation and arrangement of polymer threads and yarns, see Fig. 7(a). The uneven distribution is established due to fiber's individual composition and structure. This surface roughness is related to random

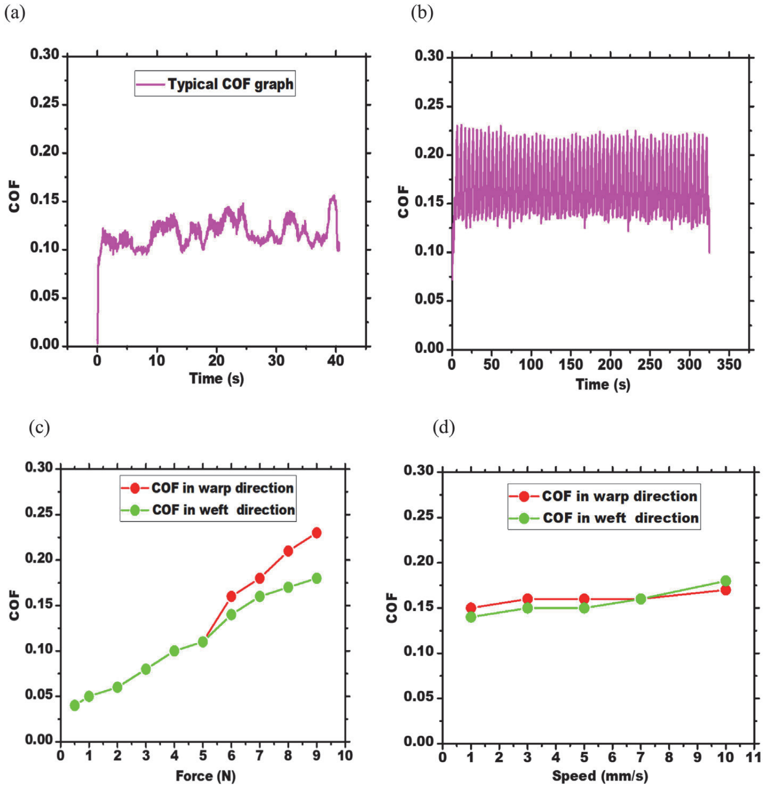

Fig. 5. Tribological investigations of post-consumer cotton polymer: (a) Typical COF graph for sliding principle, (b) Typical COF graph for reciprocation motion, (c) COF graph for force variations (d) COF graph for speed variations.

oriented fibers, jerks, bobs, surface distortion, and damage [60,67]. However, metallic surface roughness is produced due to product manufacturing and finishing operations, see Table 3.

The various materials and coatings have been considered to optimize COF for cotton processing. The experimental results were shown in Fig. 7(b). The results show that the nature of the counter body (metallic) plays a vital role. The friction coefficients for steel, zirconia, alumina and TiCN were found in the range of 0.12–0.22, 0.04–0.27, 0.11–0.22, and 0.30–0.45, respectively. Additionally, the measured micro surface hardness on the Vickers scale was 210, 1250, 1450, and 2800 HV for steel, zirconia, alumina and TiCN materials and coatings, respectively, see Fig. 7(c). All materials and coating show disparate responses during interaction with cotton polymer.

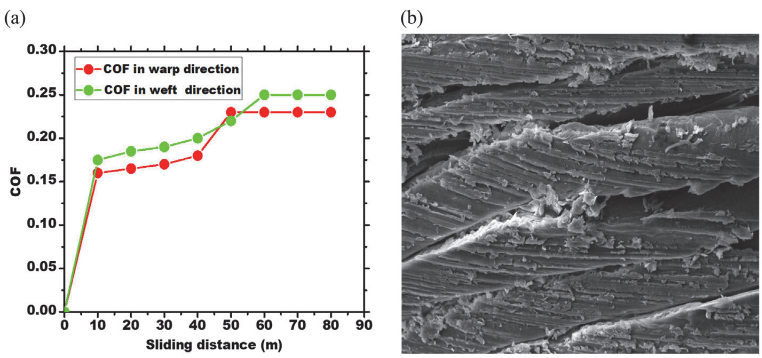

Fig. 6. Tribological and SEM investigations of post-consumer cotton polymer for industrial evaluation: (a) COF graph for sliding distance variations and (b) SEM micrograph of a damaged cotton polymer surface.

Table 3
Commercial coatings and metallic surface roughness parameters.

Material	Device	Surface roughness parameters (μm)			
		R_a	R_p	R_z	R_t
AISI 52100 mild steel balls	Optical (non-contact method)	0.34	0.56	0.67	1.60
	Mechanical (contact method)	0.24	0.25	0.50	1.10
TiCN coated steel balls	Optical (non-contact method)	0.43	0.25	0.33	1.05
	Mechanical (contact method)	0.35	0.27	0.28	0.98
C10 alumina ceramic balls	Optical (non-contact method)	0.24	0.32	0.34	0.92
	Mechanical (contact method)	0.25	0.39	0.37	0.97

4. Remanufacturing processes and damage mechanics analysis

4.1. Break even analysis

The act of division of cost summary into its components for factor evaluation is termed cost analysis. The variable cost (labor, raw materials cost, machinery cost, packaging cost, royalties), fixed cost (depreciation, rent, office salaries), total cost (fixed plus variable costs), direct costs, and indirect costs (factory overheads) are considered before processing of materials. Cost in terms of pricing governed the survival of an organization. The price as a liaison between consumer and organization regulates the success or failure of the manufacturing industry. The selling price as a bridge between supply and demand decides the production in the polymer and composites industries.

Break even analysis (BEA) is an industrial cost analysis tool. BEA allows us to predict the success rate of projects. Fig. 8 considers the sales of manufactured products as a function of revenue. The primary and post-consumer cotton wastes were provided by Estonian local industry. Therefore, we have considered the theoretical application of the BEA model. The total cost of a project is given by a formula:

$$Total\ Cost\ (TC) = Fixed\ cost\ (FC) + Variavle\ Cost\ (VC)$$

TC helps to analyze the initiation of a project. FC is the initial amount of money in terms of ‘Z’ required for the manufacturing of ‘Y’ number of units without risk of loss.

However, the propagation of the project required the inclusion of a VC. This condition creates the risk of loss. Therefore, the break-even point (BEP) came into existence. BEP is a point at which revenue is equal to the cost of the project. Hence, BEA helps to predict the period of initiation, expanding, lowering, and narrowing the business during implementation of CE.

The recycling of polymers with green and sustainable technology is a challenge of the twenty-first century. The nature of polymer

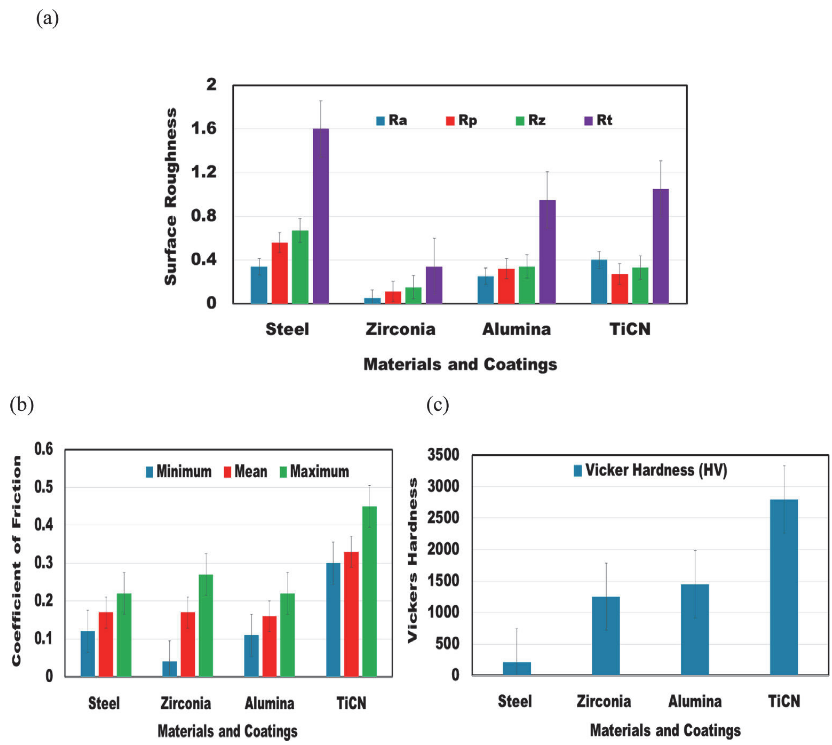

Fig. 7. Tribological and surface investigation demonstrations: (a) Surface roughness effect, (b) Typical COF value effect and (c) Vickers hardness effect.

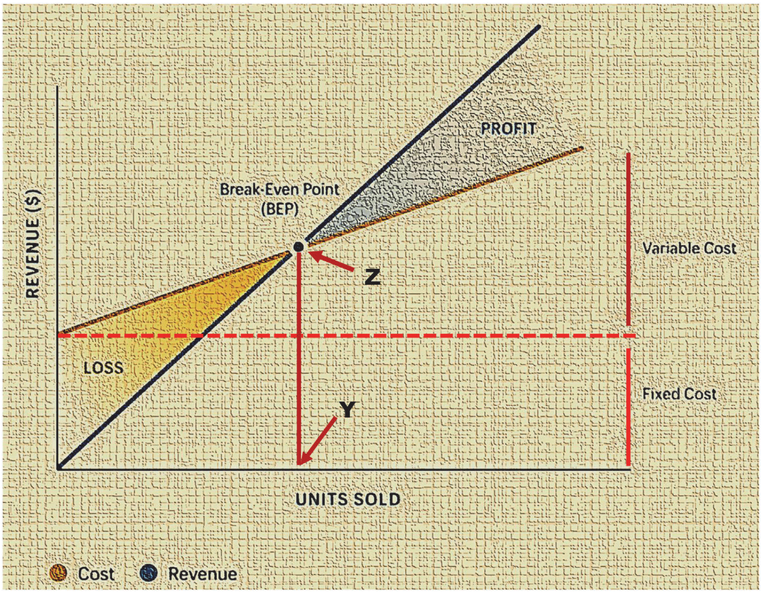

Fig. 8. BEA model.

materials plays a vital role in the optimization of tactical properties. Besides virgin polymer materials processing, the CE mainly focuses on recycling polymer wastes [68,69]. The organic, solid, liquid, recyclable and hazardous are the five main types of waste.

The increase in population, diminishing of natural resources, lack of modern recycling technologies, and variations in costs transfer the research towards the development of sustainable and green technology [70,71]. The wastes are categorized into pre-consumer, post-consumer, end waste, and end-of- life wastes for recycling. Before recycling, all types of waste are separated and sorted. The separation and sorting of polymer and composite materials is divided into manual, semiauto, and automatic techniques. The infrared spectrometers, electromagnetic conveyor belts, X-ray fluorescence sensors, electrostatic sorting, air sorting, and chemical sorting methods are used to sort thermosetting, thermoplastic, and natural polymers. The separation is done to separate different types of polymers and increase the quality of the recycled products [72]. Moreover, sorting and separation also help in the selection of recycling techniques. In the next step, the cutting, shredding, crushing, and grinding is conducted to reduce the size of polymers. The polymers are shredded using direct or indirect methods linearly and rotationally into optimum size using hardened metal blades and cutting tools. After grinding, the polymers are categorized into optimum, smaller, and oversize fibers or yarns. The pure polymer waste with minimum impurity is remanufactured through primary recycling. Secondary recycling is used for processing those polymer wastes which contain a removable number of impurities and other contamination [72–75]. The polymer wastes are purified through a sorting, purification process, and separation using acids, additives, mixtures, and degassing agents during cleaning. However, tertiary, biodegradable, and incineration techniques are introduced if impurities and contamination exceed above 10–20% of the total weight of polymer wastes [14,76–82].

In the last five decades, the extensive research has been done on the traditional processing and testing of polymers. The research depicted the mechanical (elastic modulus, yield strength, tensile strength, and fracture strength), tribological (coefficient of friction, abrasion, wear, and erosion), and surface properties of polymer and composite materials. These properties determine the commercial use of final products. However, up to best of our knowledge, no work has been reported for the investigation of the circular economy relationship with processing, testing, management, and cost analysis. Industrial implementation requires a deep understanding of engineering management, material analysis, testing, economical overview, and optimum recycling technique selection, see Fig. 9. The current study presents a set of technical strategies and paradigms for circular economy implementation in industries. The cost analysis, waste materials evaluations, recycling techniques considerations and product quality testing were used for the first time for the transformation of open system processing into closed system processing [83–85]. The direct materials cost, labor cost, and production cost help to govern and control the commencement of recycling. The polymer materials analysis along with surface investigations manifest the quality lowering in terms of impurities, surface damage, and distortion due to mechanical and chemical treatments during service life. These parameters help to find the conclusive processing technique from primary, secondary, tertiary, and quaternary recycling methods. The mechanical properties like tensile force (180 N), design force (250 N), breaking force (150 N) and extension (28%) explain the buckling, tangling, quality, and performance during processing. Similarly, the optimized low coefficient of friction (close to super lubricity) from 0.05 to 0.07 can impart reasonable tactical properties and overcome operational problems like abrasion, erosion, and fatigue. The contact and noncontact profilometer observations show the surface roughness of cotton polymer waste. In our previously reported work, we

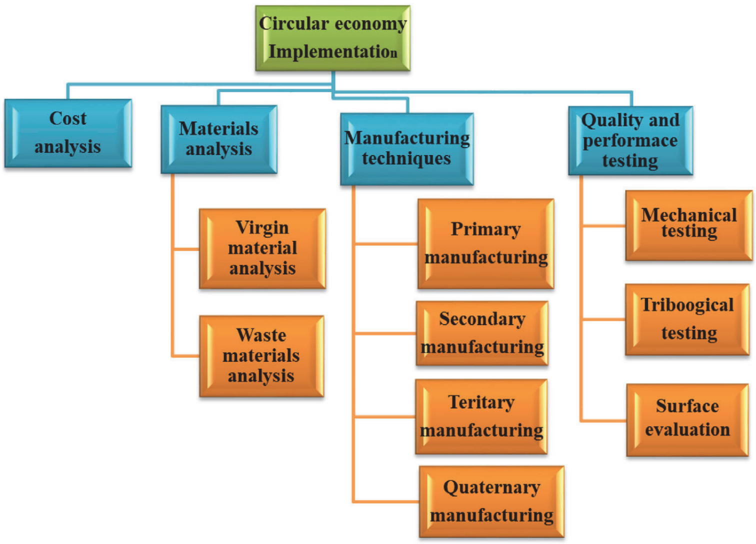

Fig. 9. Mutual technical strategies and paradigms (CE model) relationship for closed loop processing.

have proved that tactical properties of cotton products processed through recycling and manufacturing depend primarily on surface conditions, relative metallic–cotton interaction, and surface hardness. The polymer weight, density, surface pattern, temperature, mechanical properties of cotton, and polymer thickness also affect the quality and performance of polymers. During metallic–cotton relative interactions, TiCN causes the creation of plastic deformation, heavy wear, and hence local cotton material removal. Alumina and mild steel balls show almost similar behavior. Both metallic balls produced only some deformation on the cotton polymer surface. Briefly, the cost and materials analysis help to decide the suitable recycling technique during innovation and development. Moreover, recycling and testing techniques evaluations help to predict the service life, useability, suitability, and diversity of polymer composite products.

5. Conclusions

The post-consumer cotton fabric waste was selected for mechanical and tribological testing. The optical, mechanical and SEM analysis proved that cotton fabric waste has higher surface roughness, jerks, bobs, joggling, distortion and microfibrils. The defects produced tangling and buckling effects on machinery parts. Higher values of tensile (180 N), design (250 N), breaking force (150 N), and extension (28%) show the suitable condition of cotton waste for recycling. The friction coefficient for steel, zirconia, alumina and TiCN coatings were found in the range of 0.12–0.22, 0.04–0.27, 0.11–0.22, and 0.30–0.55, respectively. Cost calculations in terms of break-even analysis help to predict the commercial value of a recycling process or project. Finally, the CE model was developed to transform open-loop into closed-loop manufacturing. Factory overhead, total, fixed, direct and variable costs govern the commencement of recycling. After commencement of recycling, the material analysis of cotton waste can assist to select a suitable processing technique from primary, secondary, tertiary, and quaternary manufacturing methods. Moreover, the tested metallic materials can be used for surface modification of various industrial machinery parts.

Data availability statement

The original contributions presented in the study are included in the article and further inquiries can be directed to the corresponding author.

Authorship contribution statement

Abrar Hussain: Conceptualization, Methodology, Complete experimentation, Writing – original draft, Validation. CE industrial implementation review, open system to closed system transformation, sustainability check. **Vitali Podgursky**: Supervision review and editing, Validation. **Mart Viljus:** Cotton waste SEM Characterization-evaluation before and after testing, Review, and editing. **Muhammad Rizwan Awan:** Review and Editing.

Funding

This study has received funding from Tallinn University of Technology.

Declaration of Conflicting Interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

Acknowledgements

This research was supported by Estonian Ministry of Research and Education and Tallinn University of Technology under project KIK 19019.

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Publication III

Hussain, A., Podgursky, V., Goljandin, D., Antonov, M., Viljus, M., & Krasnou, I. (2023). Sustainable fabrication of polypropylene-postconsumer cotton composite materials: circularity, characterization, mechanical testing and tribology. *Materials Today Sustainability*, *22*, 100344. doi.org/10.1016/j.mtsust.2023.100344.

Materials Today Sustainability 22 (2023) 100344

Contents lists available at ScienceDirect

Materials Today Sustainability

journal homepage: https://www.journals.elsevier.com/materials-today-sustainability

Sustainable fabrication of polypropylene-postconsumer cotton composite materials: circularity, characterization, mechanical testing, and tribology

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ARTICLE INFO

Article history:
Received 9 November 2022
Received in revised form
21 January 2023
Accepted 29 January 2023
Available online 8 February 2023

Keywords:
Circular economy
Sustainability
Recycling
Polymer composites
Mechanical properties
Wear

ABSTRACT

The processing, microstructure, mechanical properties, characterization, and performance as paradigms play vital in determining a material's ultimate role in commercial applications. Circularity of natural polymer waste is required innovation in industrial processing. In this creative research, an industrial model is introduced for pilot fabrication of polypropylene–postconsumer cotton fiber reinforced composites. International organization for standardization (ISO 9001–2) standards are instigated for selection and categorization of manufacturing techniques and waste. In this research, we applied compression and injection molding techniques to fabricate polypropylene (PP)–cotton composites. Postconsumer cotton waste was used as a reinforcement material. Postconsumer cotton fiber (PCCF) content in the composites was 10, 30, and 40% wt. Injection molding was found to produce composites with better structure than compression molding. As compared to the PP samples, the best results were achieved with 10% wt. Similarly, American standards are established to validate the performance and quality of developed PP-PCCF composites. The PP-PCCF composite (10% wt.) exhibited good mechanical properties with a tensile strength of 26.31 MPa, modulus of elasticity of 1476 MPa, and strain of 8%. The composite of 10% wt. PP-PCCF demonstrated reasonable thermal, wear, and surface properties, including crystallinity of 44%, degradation temperature of 360 °C, wear rate of 3×10^{-6} mm^3/Nm in sliding tests against silicon carbide (SiC) sandpaper, the average weight loss of 12 mg/kg in erosion tests, and the surface roughness Ra of 0.20 μm. Theoretical models and experimental investigations (tensile, bending, impact, surface, erosion, abrasion and thermal) are established through quantitative discussion. This pilot framework provides a foundation for the implementation of close loop manufacturing of polymer composites. Moreover, this work also controlled the commercial green production of polymer composites products with negligible waste. The investigated PP-PCCF composite materials have potential applications in the automotive and construction industries.

1. Introduction

Circular economy (CE) is a set of economic, technical, management, and quality testing strategies to manufacture reliable products from virgin and waste materials [1–4]. In a broader concept, CE is a transformation of open system manufacturing (OSM) into closed system manufacturing (CSM). The OSM operates on the idea of a ‘pro-cure-manufacture-discard’ model [4]. In the CSM, the various types of waste serve as a raw material input for different recycling processes. The practical CE is a paradigm of complex analysis of the type of materials (virgin or waste), manufacturing and testing of products, and type of commercial use. Complexity lies in the uncertainties in the cost of raw materials, suitability of polymer and cotton waste for recycling, selection of the type of manufacturing process with respect to application, quality of testing, and so on. The fabrication of composites with excellent quality from synthetic and natural polymer wastes is a challenge. In addition, the operational complications of composite fabrication can reduce the quality and performance of the product [5–8]. The pultrusion and resin transfer molding have been generally used to make products for automotive, military, marine, and aerospace

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https://doi.org/10.1016/j.mtsust.2023.100344
2589-2347/

applications from thermosetting materials. On the other hand, injection molding and diaphragm forming were introduced to manufacture composites for automotive, electronic, building construction, and smart textiles applications from thermosetting polymers. The complexity of techniques, harmful gas emissions, high costs, and toxicity of fabricated products have shifted research for developing sustainable, environment-friendly green technologies. The polymer industries are facing huge engineering problems. Many functional problems like lowering in mechanical properties and poor surface finishing have remained unsolved [9]. The optimum recycling process with reasonable cost, polymer properties testing, and evaluations assists CE to develop a unique solution for the present challenges [10–12].

Natural and artificial fibers improve the strength and stiffness of fiber-reinforced composites. The addition of fibers can remarkably enhance the properties of the synthesized composites. For instance, improved wear, erosion, abrasion, mechanical, and thermal properties have been observed [13–15]. Synthetic fibers like polyester, nylon, rubber and polyethylene terephthalate, glass, carbon, and aramid are used as the reinforcement materials in several composite materials that are currently in commercial use [16,17]. Environmental pollution, high costs, toxicity, and unreliability of oil prices are guiding research into the suitability and increasing service life of natural fibers as reinforcement materials. The biodegradability, hydrophilicity, sustainability, good mechanical properties, low density, and green utilization are the main benefits of natural fibers for composites industries. The type of fibers and amount of filler content have strong influences on the quality of composites. On the other hand, a matrix holds fibers together, transfers and distributes the loads, preserves environmental impacts, and improves mechanical properties and resistance of the fibers. For instance, the amount of cellulose influences the mechanical properties of the composite's materials reinforced with cellulose; also, lignin and hemicellulose determine the properties of the composites [18,19]. However, lignin and hemicellulose are not required more than 2–6.5% and 5% wt., respectively [20–23]. The tensile strength, modulus of elasticity, and plastic deformation describe the composites and polymer reliability during service life for various applications [24–26]. Interrelation between the properties of composites and fiber recycling processes is another important factor [27–31].

Polymer composites with natural fiber reinforcement have a significant role in commercial applications [32]. Design and selection of natural composite materials (NCMs) [33] have gained momentum due to drastic reduction in reserves of petroleum. Many researchers have done a lot of work on the fabrication of NCMs. NCMs have potential applications in the automotive, packaging and construction industries. Polypropylene (PP) as a matrix (reference) phase provides good heat distortion temperature, flame resistance, design stability, better blending properties, and versatility in processing [34,35]. PP forms suitable fiber reinforced composites with natural polymeric materials like cotton, jute, kenaf, silk, hemp, and others. Besides this, the fabricated composites are also qualified easily for quality control and assurance. However, CSM requires development of new ideas [36] and technology to overcome functional problems during waste analysis, sorting, grinding, processing, finishing [37], and quality assurance steps [35].

In this novel work, a proposed recycling model (reported by our group) is utilized to categorize various recycling techniques. Polypropylene–postconsumer cotton fiber (PP-PCCF) reinforcement composites were manufactured for the first time with reported modifications for commercial applications and processing technology. The compression and injection molding techniques were associated with melting extrusion to produce test specimens from the composites. The thermogravimetric analysis (TGA) and differential scanning calorimetric (DSC) were established for thermal evaluation. The tensile (ASTM D3039), bend (ASTM D5467), and impact tests (ASTM A370) were used to assess the tensile, flexural, and fracture toughness properties. Erosion and abrasion tests were performed to evaluate coefficient of friction, abrasive, and erosive wear. The proposed recycling model and experimental evaluations are foremost applied to demonstrate pilot projects and quality development for commercial production. The research suggests a novel concept for nanofabrication, development, testing, and characterization for potential industrial implementation.

2. Materials and methods

2.1. Materials

The postconsumer cotton waste and pure PP plastic powder were used as fiber reinforcement and matrix phases. The characteristics of the postconsumer cotton waste were 1.55 g/cm3 density, 5–25% elongation range, 0.06 MPa tensile strength, 0.10 N effective tensile strength, 0.045 MPa breaking strength, and 237 g/m^2 weight [38]. PP powder was supplied by Egyeuroptene with melt flow index = 14 g/10 min @ 2.16 kg, temperature 210 °C, and 1.50 g/mL density (ASTM D 1238). Cotton-made Estonian army T-shirts was utilized as raw material for the preparation of the PCCF.

2.2. Development and selection criteria

Selection (Table 1) and development of recycling techniques (Fig. 1) rely on purity and nature of commercial application [4,39–41]. The preconsumer waste (with purity 99.5%) can be used as it is or virgin raw material [42,43]. Lowering in purity of postconsumer waste (which usually contains 90–95% valuable material) occurs due to service life [44]. Postconsumer waste is usually recycled using primary recycling techniques [45]. However, contamination requires additional techniques for removal of impurities from end-waste polymer wastes (purity usually <85%) [42,46]. Incineration is directly introduced for energy production from end-of-life polymer wastes. An end-of-life waste has a very low quantity of valuable material [47,48].

2.3. Grinding and fabrication of PP-PCCF composites

The cotton polymer waste was cut into small pieces, grounded, and milled using a developed prototype grinding machine at a speed of 300 rpm for 10 min. This direct grinding disintegration machine transforms polymeric waste into fine fibers (Fig. 2). The produced fibers were 2–5 mm in length, 11–21 μm in diameter, and with an area of 200–250 μm^2. The fabric material, especially cotton, was dried for 4 h at a temperature of 60 °C. The compounds with pure PP and 10, 30, and 40% wt. variations of cotton fiber reinforcement were fabricated. The PP and PCCF were mixed in a cylindrical mixer at a speed of 80 rpm for 15 min. The PP matrix and PCCF were compounded at 190 °C for 7 min, at a torque of 60 Nm and a speed of 80 rpm. Compounding materials were passed through different melting zones operating in the range from 120 to 200 °C. Finally, the extruded melted wires were ground and milled into fine grains and beads. In compression molding, the fine grains of the composites were fabricated into 1 mm, 3 mm, and 6 mm thick sheets using compression techniques. The time, pressure, and temperature were 7 min, 80 kg/cm^2, and 190 °C, respectively. The material was forced to move into cavity and cured for the desired shape. At the end, compressed sheets were quenched from 190 °C to room temperature for 5 min. In injection molding, the compounding mate (in the form of pellets) was heated and injected into the mold cavity at the temperature of 190 °C. The final products

Table 1
Scheme for selection of recycling techniques and waste.

Polymer waste	Conventional conditions	Processing technique	Outcome and technical comments
Preconsumer wastes	Virgin form of valuable polymer	Primary manufacturing or recycling	Manufactured polymer products for all types of applications.
Postconsumer wastes	Addition of impurities due to useability	Primary recycling	Recycled product required in depth analysis like tensile, bend, impact, SEM, optical, tribological and surface for determination of commercial applications
End-wastes	High impurity level and low quality due to service life	Secondary and primary recycling are required	Low quality products for common applications.
End-of-life wastes	Low quality and hazardous in nature	Tertiary or incineration	Energy production for industries or urban areas. Landfills.

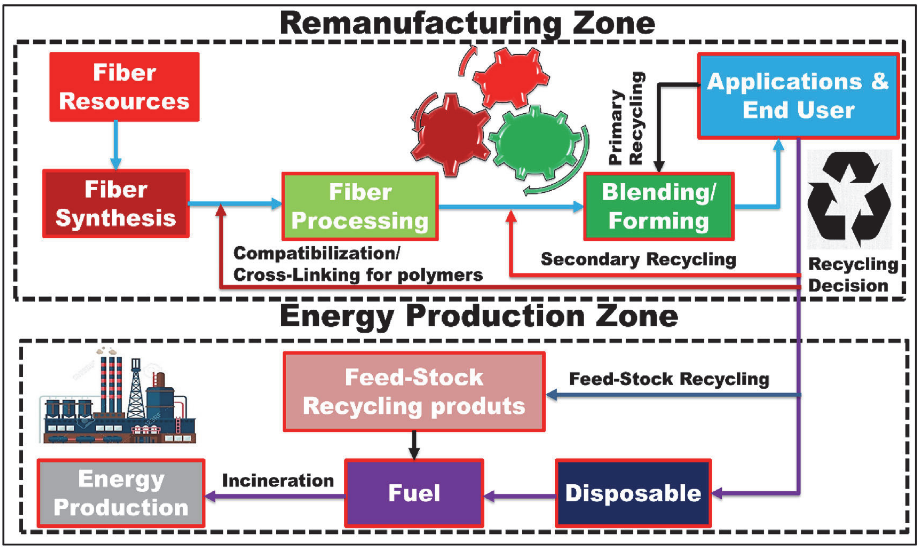


Fig. 1. Proposed and reported model of recycling.

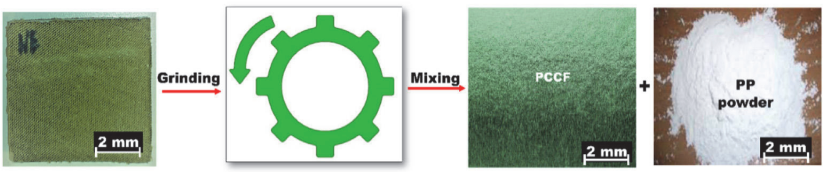


Fig. 2. Initial fabrication steps of polypropylene–postconsumer cotton fiber composites.

were cooled and hardened using the automatic cooling system of the machine.

2.4. Thermal analysis of PP-PCCF composites

The TGA and DSC methods were used to evaluate thermal properties.

2.4.1. Thermogravimetric analysis

The thermogravimetric analyzer (TGA 1000 system, Anderson Materials Evaluation, Inc.) was used for the TGA. In the TGA, ceramic crucibles were used to heat samples (10 mg each) in the nitrogen environment at the scanning rate of 10 °C/min. The temperature varied from 25 to 1000 °C during the testing.

2.4.2. Differential scanning calorimetric

A simultaneous thermal analyzer (Model STA 449 F3 Jupiter, NETZSCH Co) was utilized for DSC analysis. DSC was carried out at a heating rate of 15 °C/min. The samples (10 mg each) were heated from room temperature to 250 °C and cooled down at the same rate (15 °C/min). The melting point, crystallization temperature, heat of fusion, and percentage of crystallization were calculated.

2.5. Mechanical testing of PP-PCCF composites

A Universal Testing Machine (Model 5820, Instron Co.) was used to evaluate mechanical properties, namely tensile and bending strengths. The testing was performed according to ASTM D3039 (tensile testing) and ASTM D5467 (bending test) standards. The specimen's size was 4 (thickness) × 25.4 (width) × 150 mm (length). The gauge length was kept at 100 mm. The crosshead speed was 50 mm/min. The force—extension and stress—strain graphs were recorded using data collection and acquisition software. Additionally, ASTM A370 Charpy tests with v-notch were carried out on a local impact testing machine to study the strain rate and impact energy.

2.6. Tribological testing of PP-PCCF composites

2.6.1. Abrasion testing

Abrasion wear testing was performed on the tribometer (Model UMT-2, CETR Bruker) in zig-zag reciprocal motion mode. The samples were tested against the SiC P150 grade sandpaper. The average speed, load, and abrasive sliding path distance were 0.1 m/s^{-1}, 1 N, and 18 m, respectively.

2.6.2. Dry erosion testing

The dry erosion wear tests were done on a four-channel accelerator erosion testing machine. The test was performed in a sequence of three steps, and the weight loss was measured after each step; in the following text, the steps are indicated as first, second and third weight losses. The impact angle, time of each step, and weight of the sand (abrasive) used in the test was 300, 30 min, and 6 kg, respectively. The weight loss (M) of the composite materials was calculated by the following formula:

$$M = \frac{\Delta m}{Gv} \tag{1}$$

where M is the specific weight loss (mg/kg) of the sample, G is the weight of sand, and v is the share of sand per sample. Δm (mg) is the weight loss of each composite sample during the experiment. Δm was measured before and after the erosion test using Mettler Toledo ME204 balance.

2.7. Surface evaluation of PP-PCCF composites

A mechanical profilometer (Mahr Perthometer PGK120) and optical profilometer (Contour GT-K0+ 3D) were used to measure surface roughness. Surface morphology was investigated by the scanning electron microscope (SEM) (Zeiss EVO® MA-15 system, Oberkochen, Germany) with LaB6 cathode in the secondary electron mode, applying an accelerating voltage of 10—15 kV at 6.5—8.5 mm working distance.

3. Results and discussion

3.1. Thermal analysis

Fig. 3a—b shows the results of the DSC and TGA. The material produced after the compounding process (see Materials and methods) was under investigation. The crystallinity of pure PP and composites (10, 30 and 40% wt.) was 42, 44, 33, and 23%, respectively. The extent of crystallization was calculated by the following equation:

$$\%Xc = \frac{\Delta H_f}{\Delta H_f^o} \times \frac{100}{w} \tag{2}$$

where Xc, w, ΔH_f and ΔH_f^o are the amount of fraction, weight, the heat of fusion of the composite material, and the heat of fusion of a 100% reference material, respectively. The melting and crystallization temperatures of PP and PP-PCCF composites (10, 30, and 40% wt.) show an opposite trend with the increase of the PCCF content, namely melting temperature changes as follows: 169 °C, 174 °C, 173 °C, and 168.60 °C, contrary to crystallization—118 °C, 105 °C, 108 °C, and 117 °C. In other words, melting temperature increases for 10% wt. in contrast to the PP, with the following decrease for other PP-PCCF compounds. In the case of the crystallization temperature, it decreases by 10%wt. PP-PCCF compounds and increases for other PP-PCCF compounds.

The degradation temperature (Fig. 3b) of pure PP and composites decreases from 480 to 450 °C due to water removal and weakening of C—C bonding [49,50]. The composite degrades due to the processes occurring in the matrix and fibers [50]. In general, the degradation temperature decreases due to the decomposition of saturated and unsaturated hydrocarbons, depolymerization, and removal of volatile materials like hemicellulose and pectin. In addition, the decrease in crystallinity and melting and degradation temperatures with increasing fiber content can occur due to the presence of nucleation, weak bonding, and impurities [51—53]. Moreover, crystallization temperature also shows variations in behavior. The behavior follows the 'decrease—increase' cycle (Fig. 3a). The crystallization resembles the regular arrangement of polymer crystals. Therefore, the higher crystallization temperature of PP is due to the presence of higher nucleating sites (that expand crystallinity in all directions). However, PCCF addition can act as a barrier (smaller PCCF addition) and promotor (higher PCCF addition). This effect can cause a decrease in the crystallization temperature.

Fig. 3a and b represent DSC and TGA of PP-PCCF composites. Pure PP material always has sharp values for melting, crystallization, and degradation temperature. However, the addition of cotton fibers as a reinforcement phase (impurity) changes the thermodynamic conditions of developed PP-PCCF composite materials. The PP-PCCF composites materials melt, crystallize, and degrade at a specific range of temperatures. The variations occurred due to differences in thermodynamic values. That is why the increase or decrease in thermodynamic values came into existence. The same phenomena were shown in Fig. 3a and b.

Our represented results of TGA and DSC analyses match with literature values [43—47]. The decrease in thermal stability of PP-PCCF composites is negligible. However, thermal stability can be stabilized using binding materials and mixtures.

3.2. Surface and cross-sectional microscopic analysis

The surface morphology of the composite materials produced by compression molding is shown in Fig. 4a—d. The surface of pure PP (reference material) was found smoother than that of PP-PCCF composites. Cotton fibers can be seen on the surface of the PP-PCCF composites (with 10, 30, and 40% wt. fiber loadings). The increase in the fiber content causes the creation of surface defects like asperities and microcracks. These defects make composite surfaces rough, see Fig. 4 b-d.

Fig. 5 a-d shows the surface morphology of the composites prepared by injection molding. The surface of the produced materials is smoother than that of the composites prepared by compression molding.

The characterization of cotton fibers was performed before and after mechanical and tribological testing. The diameter, length, and area of cotton fibers were in the range of 2—5 mm, 11—21 μm, and 200—250 μm^2, respectively (Fig. 6a, numbers 1, 2, and 3).

Cotton fibers consist of microfibrils that are bound with the core of the fiber through lignin, see Fig. 6b. Therefore, such the structure of cotton fibers contributes to porosity [54,55] and random orientation of fibers [56—58] during the production of composite materials.

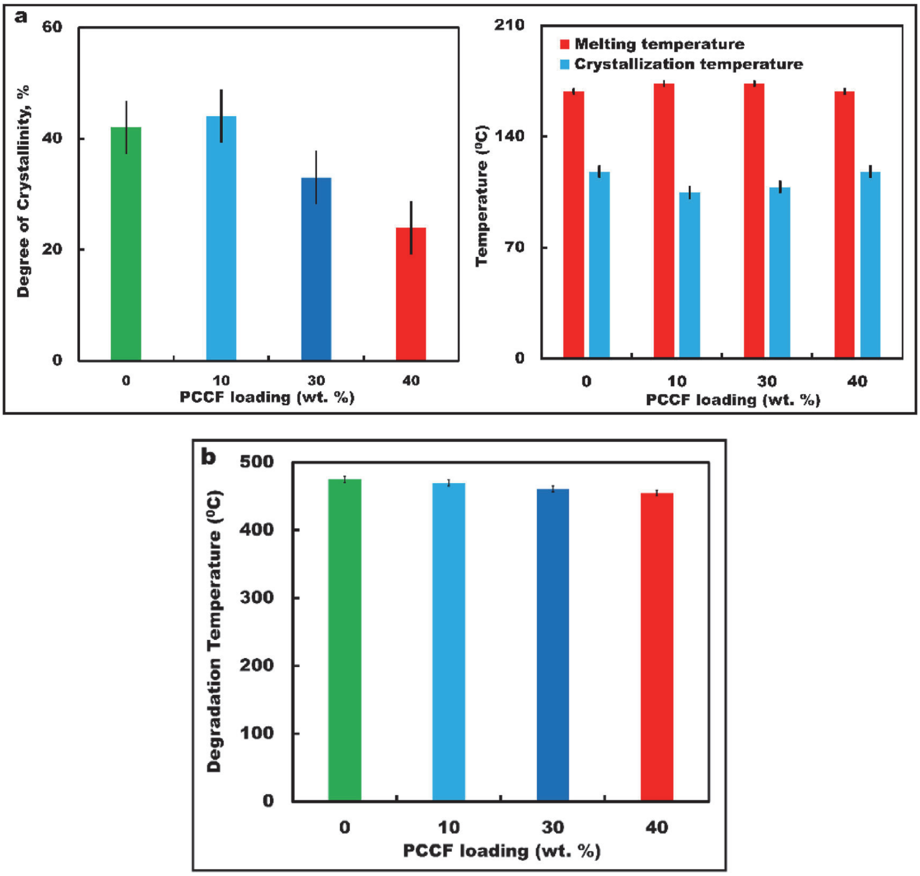


Fig. 3. Thermal properties of pure PP and PP-PCCF composites: (a) DSC and (b) TGA for thermal stability. Abbreviations: PP-PPCF, polypropylene–postconsumer cotton fiber; TGA, thermogravimetric analysis; DSC, differential scanning calorimetric analysis.

The SEM images and surface roughness parameters of pure PP and composite sheets produced by compression molding are shown in Fig. 7 a-e. A smooth surface of pure PP and 10% wt. PP-PCCF composite can be seen (Fig. 7a and b). The line-like asperities formed on the surface of the composite (Fig. 7 b). Line-like asperities and highly rough areas can be observed in Fig. 7c and d for 30% and 40% wt. PP-PCCF composites. Due to these defects, the surface roughness parameters were higher in contrast to the PP and 10% wt. PP-PCCF composite (Fig. 7e).

However, the same composites fabricated by injection molding have a smoother surface and a smaller number of defects, see Fig. 8 a-e. The evaluation of the surface roughness parameters of the manufactured composites is shown in Fig. 8e. The surface smoothness and homogeneity of fiber distribution decrease with an increase in fiber loading as reinforcement materials. The increase in fiber loading reduces the bonding area between the PP matrix and cotton fibers, resulting in defect formation and boost of a surface roughness increase [59,60].

The composites produced by compression and injection molding were cut, and the cross-section area was investigated by SEM, see Figs. 9 and 10. The surface debris, asperities, and void were observed in compounds produced by compression molding. In the case of injection molding, the voids formation was observed only in a composite with 40% wt. fiber loading.

Insufficient manual control of fabrication parameters, especially temperature, pressure, and time during compression molding, can lead to a decrease in the wettability of the matrix phase with cotton fibers (Fig. 9 b-d). For instance, a variation of pressure along the sample surface during compression can cause poor compaction of composites during compression, resulting in defect formation in the composite. The lowering in wettability caused poor adhesion between the CF and PP matrix. Due to these factors, the surface roughness of composite materials produced by compression molding (Fig. 7 e) is higher than that of the composites produced by injection molding (Fig. 8 e).

No voids or other defects were detected in the reference material (pure PP) and composites (10%, 30% wt.) fabricated by injection molding, see Fig. 10 a-c.

Fig. 10 d shows the formation of small voids due to the increase in the PCCF content to 40% wt. The increase in the fiber content enhances the hardness of the composites produced by injection molding. The hardness of PP and PP-PCCF composites was 33 HR15W (PP), 13 HR15W (PP-PCCF 10% wt.), 19 HR15W (PP-PCCF 30% wt.), and 23 HR15W (PP-PCCF 40% wt.), respectively (Rockwell HR15W scale).

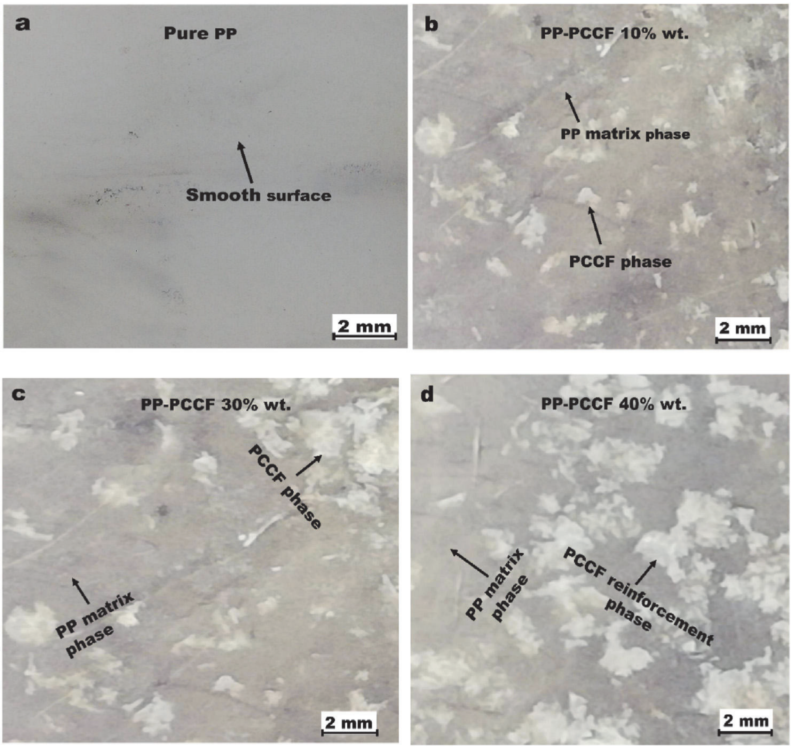


Fig. 4. Optical micrograph images of the composites produced by compression molding (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt., and (d) PP-PCCF 40% wt. Abbreviation: PP-PPCF, polypropylene–postconsumer cotton fiber.

To summarize, optical microscopy, SEM, and surface roughness analysis showed considerably fewer defects in the structure of composites produced by injection molding than in the composites produced by compression molding. These outcomes lead to studies of composites produced by injection molding using tensile, compression, and impact testing, as well as erosion and abrasion techniques.

3.3. Mechanical investigations

The results of mechanical properties of composites produced by injection molding for various fiber loadings are shown in Table 2. Fig. 11a presents samples of the stress–strain diagram. The data indicate that tensile stress, tensile strain, and flexural strain decrease with the increase in the fiber content. Increase in the fiber content decreases adhesion between the PP matrix–PCCF interface; thus, PP-PCCF composite (especially PP-PCCF 30% wt. and PP-PCCF 40% wt.) fracture occurs under smaller deformation in tensile tests. Poor adhesion can cause the fiber pullout in composites materials, see Fig. 11b. On the other hand, according to the results in Table 2, the modulus of elasticity E in tensile and flexural tests increases with an increase in PCCF loading. The increase in the fiber content that is directly proportional to the increase in the value of the modulus of elasticity indicates the influence of elastic properties of PCCF on the properties of composites. It was mentioned above that the increase in the hardness of the composite with the increase of fiber loading was observed as well, indicating simultaneously an increase in the elasticity modulus and hardness. This fact could explain the observation of the highest value of impact energy for PP-PCCF 40% wt. composite, that is, the increase in the notch toughness of the material with the highest hardness. Usually, materials with higher hardness tend to show lower toughness.

Despite statistics, a more complex response, especially for flexural strength and impact energy, was found in the case of the PP-PCCF composite 30% wt.; the reason probably is a transition from more ductile/plastic (like for PP) to more elastic/brittle behavior with an increase in the fiber loading. In other words, under bending conditions, this transition is manifested more noticeably than under tension. Moreover, the embrittlement of composite materials under bending conditions is more essential than under tension.

3.4. Tribological analysis

The wear rates of pure PP and PP-PCCF composites prepared by injection molding against SiC P150 grade sandpaper are shown in Fig. 12a. The wear rate values of pure PP and PP-PCCF with 10, 30, and 40% wt. fiber fractions were found 3.09×10^{-6} mm^3/Nm, 3.54×10^{-6} mm^3/Nm, 6.25×10^{-6} mm^3/Nm, and 6.38×10^{-6} mm^3/Nm, respectively. The adhesion between the PP matrix and fibers decreases with an increase in the fiber loading, leading to the

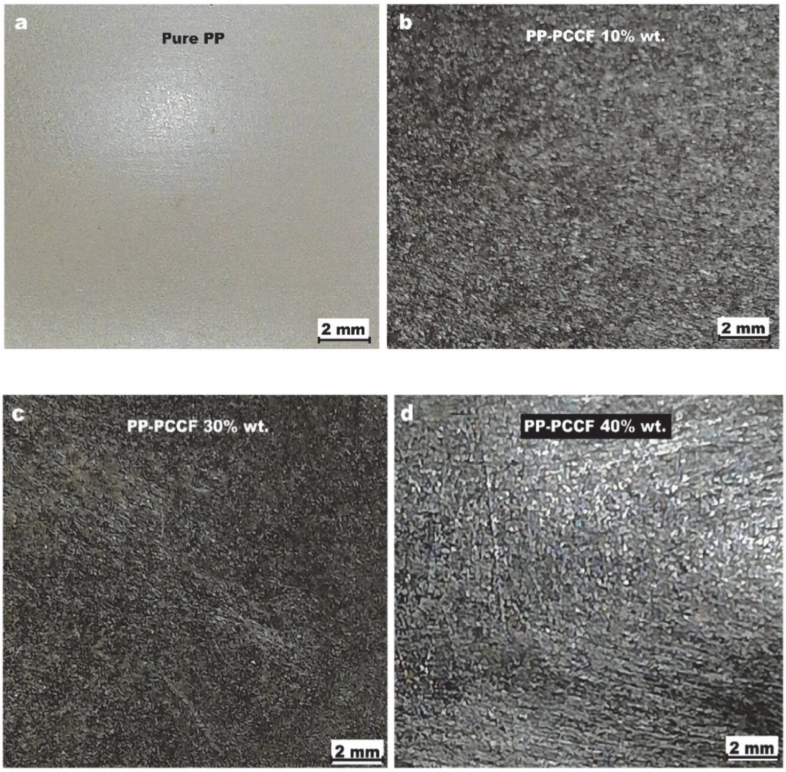


Fig. 5. Optical micrograph images of the composites produced by injection molding (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt., and (d) PP-PCCF 40% wt. Abbreviation: PP-PPCF, polypropylene–postconsumer cotton fiber.

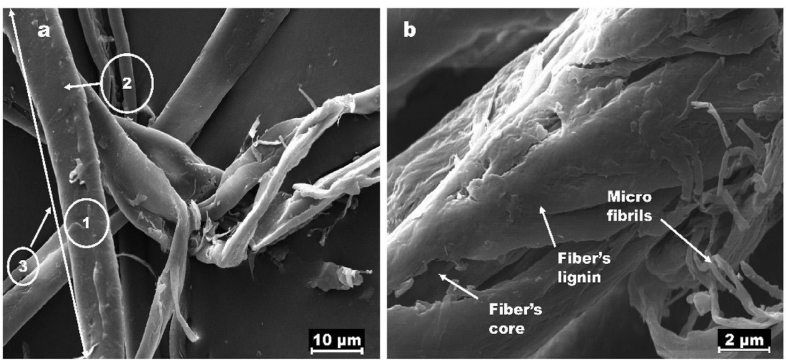
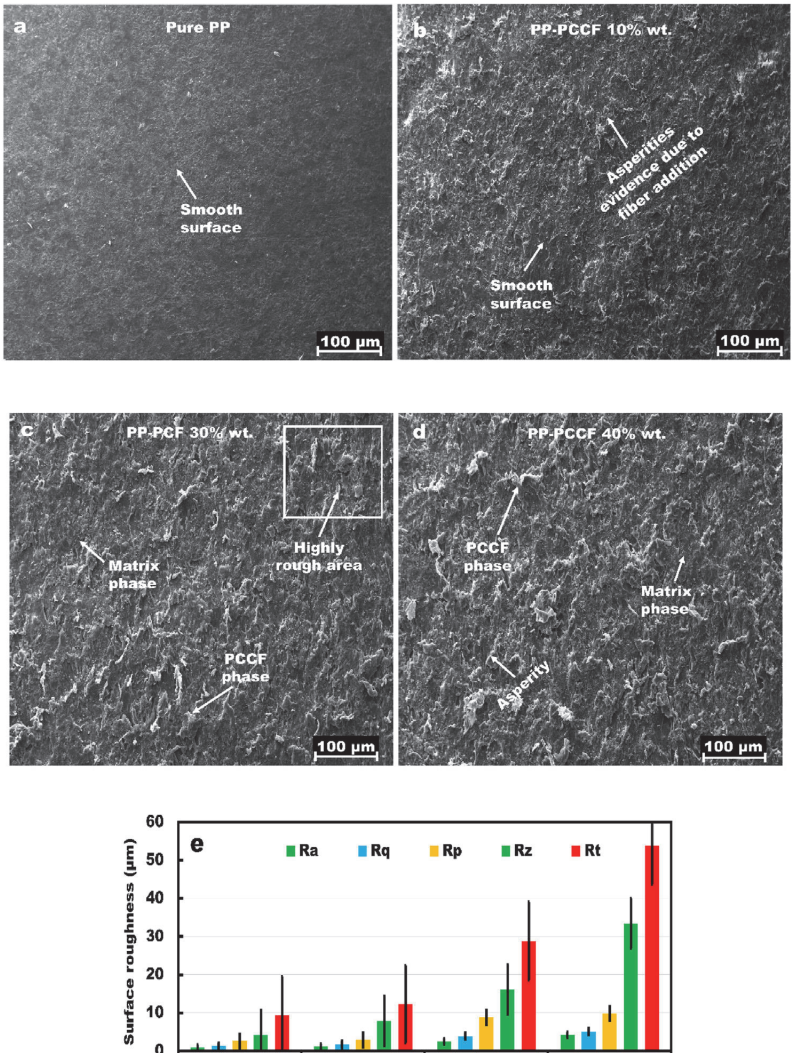


Fig. 6. SEM images of PP-PCCF: (a) diameter (1), length (2), area (3) of fibers and (b) core, microfibrils and lignin are observed in postconsumer cotton fibers. Abbreviations: PP-PPCF, polypropylene–postconsumer cotton fiber; SEM, scanning electron microscopy.

formation of defects, including line-like asperities, porosity, and debris, which finally results in a wear rate increase.

The coefficient of friction (COF) was studied between SiC sandpaper and composite materials, see Fig. 12b. The average COF values were in the range from 0.63 to 0.93. The increase in the COF value with an increase in fiber loading could be due to the increase in surface roughness (Figs. 7e and 8e) [61]. SEM images of the pure PP and composite with 10% wt. fiber content after abrasion tests are shown in Fig. 12 c, d. The plastic deformation occurs on the composite-to-SiC sandpaper surfaces due to shear stress. Moreover,



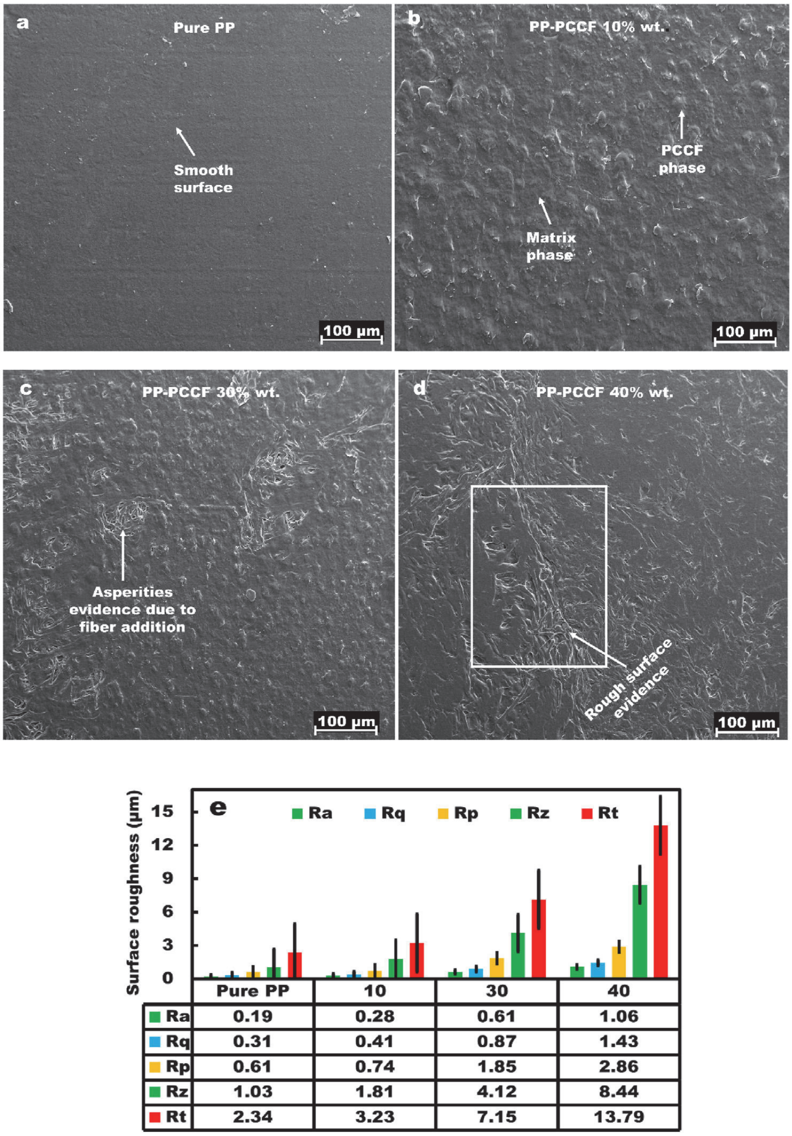
	Pure PP	10	30	40
Ra	0.89	1.2	2.5	4.16
Rq	1.31	1.71	3.88	5.05
Rp	2.61	2.94	8.85	9.86
Rz	4.13	7.81	16.1	33.44
Rt	9.34	12.23	28.8	53.8

Fig. 7. SEM images of the composites fabricated by compression molding: (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt., (d) PP-PCCF 40% wt., and (e) surface roughness parameters. Abbreviation: PP-PPCF, polypropylene–postconsumer cotton fiber. Abbreviations: PP-PPCF, polypropylene–postconsumer cotton fiber; SEM, scanning electron microscopy.

interaction between the hard particles of SiC sandpaper and the composite produces cutting and plowing. In other words, removal (failure) of material occurs on the composite's surface in the form of abrasion wear.

Fig. 13 shows the results of the erosion tests on the composites fabricated by injection molding. The weight loss is the removal of the composite material during each three steps of the erosion test (see Materials and methods). The results show considerably higher weight loss (erosion rate) of composites with 30 and 40% wt. fiber loading. The pure PP and the composite with 10% wt. fiber show relatively good erosion resistance probably due to reasonable fiber bonding with the PP matrix and higher crystallization, see Fig. 13a.

The SEM micrographs of the pure PP and PP-PCCF composite with 10% wt. fiber content after erosion tests are shown in Fig. 13 b,



	Pure PP	10	30	40
Ra	0.19	0.28	0.61	1.06
Rq	0.31	0.41	0.87	1.43
Rp	0.61	0.74	1.85	2.86
Rz	1.03	1.81	4.12	8.44
Rt	2.34	3.23	7.15	13.79

Fig. 8. SEM images of the composites fabricated by injection molding: (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt., (d) PP-PCCF 40% wt., and (e) surface roughness parameters of composites. Abbreviations: PP-PPCF, polypropylene–postconsumer cotton fiber; SEM, scanning electron microscopy.

c. The SEM image (Fig. 13b) proves the minimum fiber pull out and hence the sustainability of pure PP and 10% wt. composite materials. A similar response was shown by a composite (like abrasion testing) with 40% wt. fiber loading.

3.5. Quantitative discussion and potential commercial applications

The circularity of natural polymers is gaining momentum [62]. Use of polymer composites produced by means of green and sustainable technology is a challenge of the 21st century. The alteration in processing techniques is always required due to a lowering in mechanical properties. Lowering in mechanical properties occurred due to poor quality of natural polymer wastes after end use [63]. Commercial injection and compression molding manufacturing processes in association with extrusion were introduced to fabricate reinforced composites (Table 3). Extrusion of matrix and fiber phases helps to get maximum homogeneity and mixing. It produces an intermediate product called 'mate' [64–66].

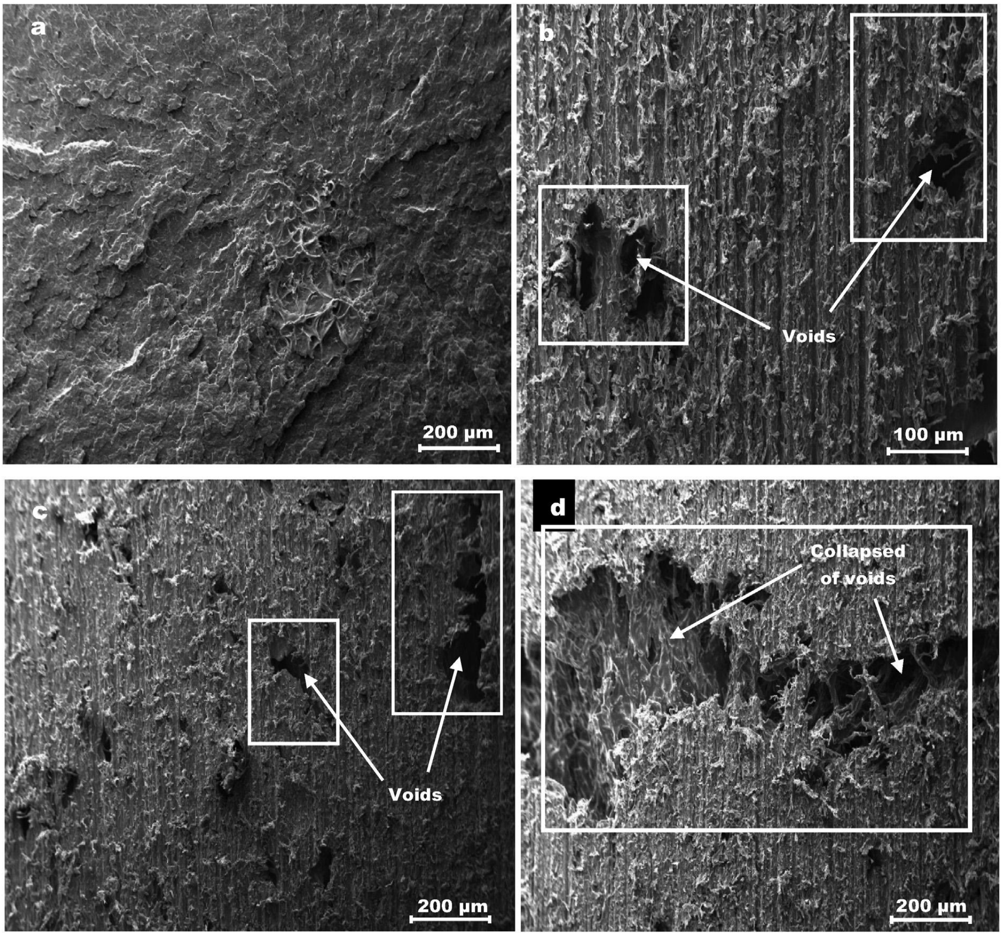


Fig. 9. SEM images of the cross-sectional area of the composites fabricated by compression molding: (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt., and (d) PP-PCCF 40% wt. Abbreviations: PP-PPCF, polypropylene–postconsumer cotton fiber; SEM, scanning electron microscopy.

Therefore, extrusion plays a vital role in regaining strategic mechanical properties [85]. After extrusion, secondary heating (heating for injection and compression molding processing) also assists in homogenizing the remaining impurities and residue. During and after processing, mostly chemical, thermal and mechanical testing is required to check the performance of recycled products [86,87].

Transformation of natural polymer waste into sustainable product is required systematic and meticulous investigations. In this research, we applied compression and injection molding techniques to fabricate PP-cotton composites. Postconsumer cotton waste was used as a reinforcement material. PCCF content in the composites was 10, 30, and 40% wt. Injection molding was found to produce composites with better structure than compression molding. As compared to the PP samples, best results were achieved with 10% wt. Similar results were also reported for other natural reinforced composites [79,88–91]. The PP-PCCF composite exhibited good mechanical properties with a tensile strength of 26.62 MPa, modulus of elasticity of 2525 MPa, and strain of 8%. The composite of 10% wt. PP-PCCF demonstrated reasonable thermal, wear, and surface properties, including crystallinity of 44%, degradation temperature of 360 °C, wear rate of 3×10^{-6} mm^3/Nm in sliding tests against silicon carbide (SiC) sandpaper, the average weight loss of 12 mg/kg in erosion tests, and the surface roughness Ra of 0.20 μm. The investigated PP-PCCF composite materials have potential applications in the automotive and construction industries.

The DSC, TGA, and SEM analyses justified the possibility of manufacturing compact, defect-free composites (especially with 10 and 30% wt. cotton fiber loadings) using injection molding. The composites studies showed low surface roughness and the values of crystallization, degradation temperature and thermal stability are in

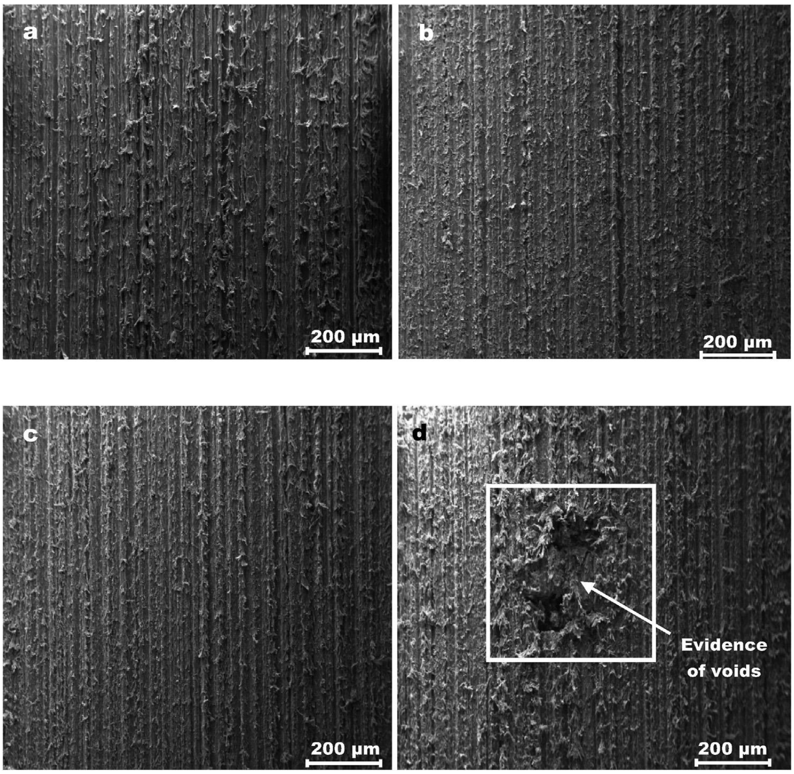


Fig. 10. SEM images of the cross-sectional area of the composites fabricated by injection molding: (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt., and (d) PP-PCCF 40% wt. Abbreviations: PP-PPCF, polypropylene—postconsumer cotton fiber; SEM, scanning electron microscopy.

Table 2
Mechanical properties of PP and polypropylene—postconsumer cotton fiber (PP-PCCF) composites produced by injection molding.

Composite	Tensile testing			Bend test			Impact test
PCCF content (% wt.)	σ(MPa)	Strain (%)	E (MPa)	σ(MPa)	Strain (%)	E (MPa)	Impact energy (kJ/m^2)
PP - 0	31.16	9.55	1226	41.97	13.77	1172	4.9
PP-PCCF - 10	26.31	8.09	1476	51.03	11.54	2069	4.8
PP-PCCF - 30	24.13	4.25	2521	34.25	9.15	2130	4.5
PP-PCCF - 40	22.16	3.55	2751	56.77	4.25	3780	5.5

good agreement with the previous results. The defects can produce stress concentration, which can reduce the properties of PP-PCCF composites. The porosity and voids presented in the composites produced by compression molding suggest that such composites could be used for thermal, acoustic, and construction applications [92,93]. In the case of composites produced by injection molding, the flexibility, toughness, tensile strength, flexural properties, deformation, and impact energy of the composites were verified using tensile, compression, and impact testing. The reference material (PP) and composites with 10 and 30% wt. PCCF contents showed reasonable mechanical and tribological properties. Therefore, the developed composites produced by injection molding can be suitable candidates for automotive, medical, and smart textile applications.

Reported model can help to select reasonable recycling technique [94]. Compression, injection molding in association with extrusion, helps to transform open loop into close loop manufacturing [95]. Thermal, mechanical, tribological, and surface evaluations governed the quality control and assurance. Collectively, the theoretical model and experimental investigations could be used for conversion of open loop into sustainable closed loop manufacturing.

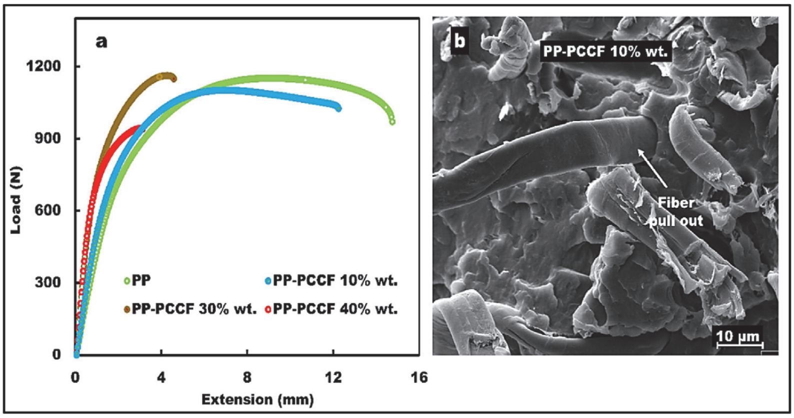


Fig. 11. The results of (a) tensile testing of composites (produced by injection molding) and (b) SEM image of fiber pull out after tensile testing. Abbreviation: SEM, scanning electron microscopy.

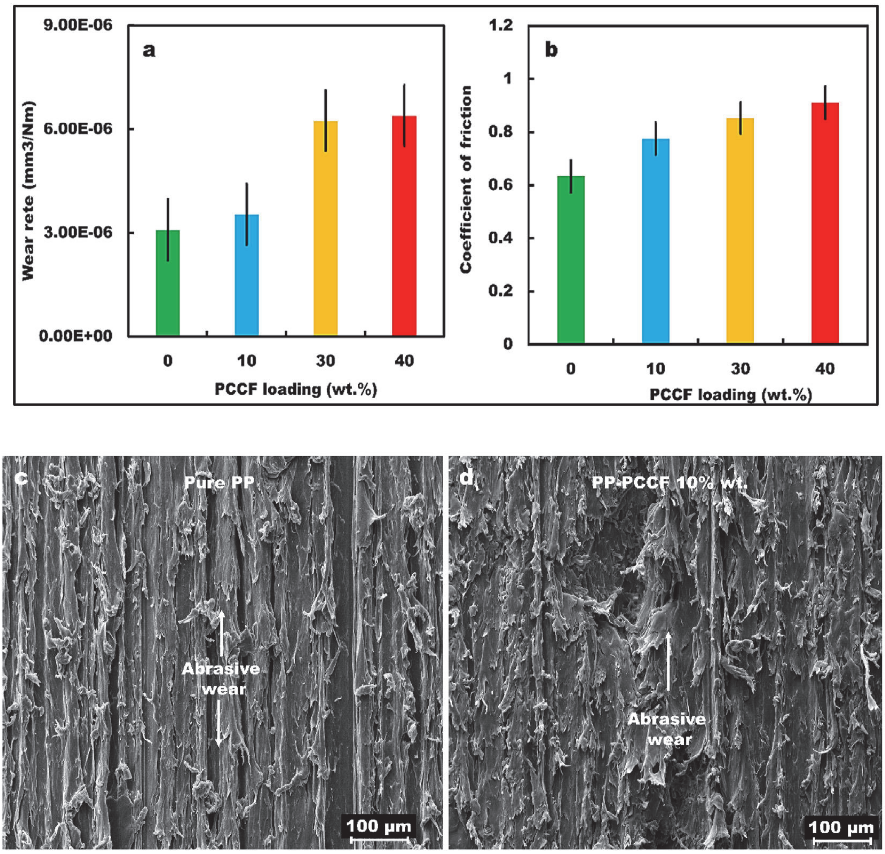


Fig. 12. Wear rate and COF were evaluated in abrasion tests and SEM images taken within the wear scar on PP and PP-PCCF composites developed by injection molding: (a) wear rates, (b) COF values, (c) SEM image of pure PP specimen, and (d) SEM on PP-PCCF 10% wt. composite. Abbreviations: PP-PPCF, polypropylene—postconsumer cotton fiber; SEM, scanning electron microscopy.

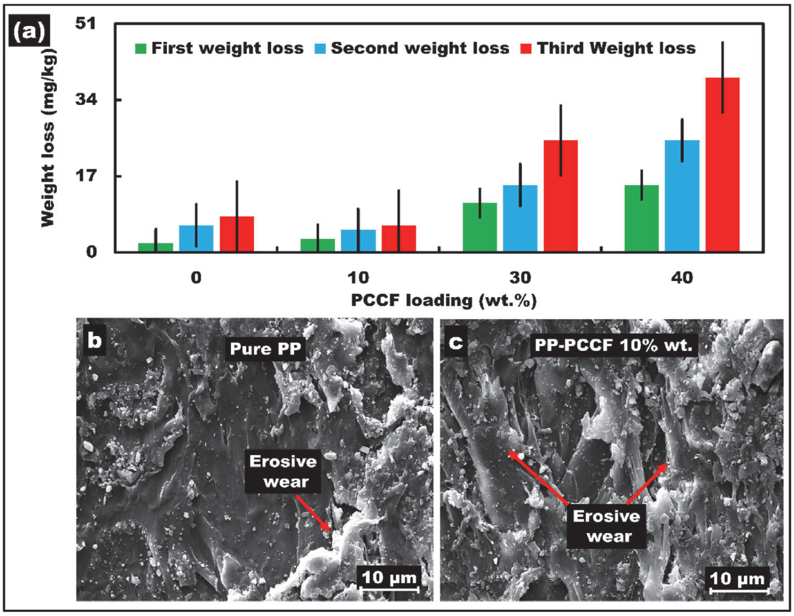


Fig. 13. Erosion test and SEM characterization of pure PP and PP-PCCF composites developed by injection molding: (a) weight loss after erosion test, (b) SEM image of pure PP after erosion test, and (c) SEM image of PP-PCCF 10% wt. composite after erosion tests. Abbreviations: PP-PPCF, polypropylene—postconsumer cotton fiber; SEM, scanning electron microscopy.

Table 3
Quantitative proof of natural reinforced composites.

Composite	Manufacturing process	Performance
PP-PCCF reinforced composites (current work)	Compression molding	Composites, suitable for acoustic and thermal applications
PP-PCCF reinforce composites (current work)	Injection molding	Composites, suitable for medical and automotive and construction applications
PP-wood fiber composites [67]	Circularity of wood natural polymers using extrusion process	Composites for commercial applications
PP-hemp fiber composites [68]	Closed loop manufacturing using compounding	Pilot scale production at laboratory
PP-cellulose reinforced composites [69]	Circularity of natural polymer composites using compression molding	Pilot production for industries
PP-flax fiber reinforced composites [70]	Sustainable production using hot pressing	Laboratory scale production
PP-kenaf fiber reinforced composites [71]	Sustainable commercial production	Composites for commercial applications
Epoxy-banana reinforced composites [72]	Compression molding	Compatible mechanical properties
PP-wood reinforced fibers composites [73]	Injection molding	Improved tensile and flexural properties for construction application
Epoxy-jute fiber reinforced composites [74]	Hot pressing	Mechanical, impact and flexural properties are improved. Suitable for commercial application
Polylactic acid-sisal fiber reinforced composites [75]	Injection molding	Increased tensile strength, modulus of elasticity and impact strength. Good interfacial binding
Polylactic acid-kenaf fiber reinforced composites [76]	Compression molding	The fabricated composites were found reasonable for commercial applications
PP-palm reinforced composites [77]	Compression and injection molding	Improved and almost same (for our reported work) thermal, mechanical, and chemical properties
PP-silk reinforced composites [78]	Compression and injection molding	Our reported work shows better mechanical properties
PP-bamboo reinforced composites [79]	Injection molding	Reported work exhibits comparable mechanical and thermal stability
PP-lignocellulosic fiber reinforced composites [80]	Compounding	Quality testing techniques for laboratory production of composites
PP-sisal fiber reinforced composites [81]	Injection molding	Quality assurance of fabricated composites for pilot production
PP-kapok fibers composites [82]	Compression molding	Quality control of composites using mechanical testing
Bio composites [83]	Hydrothermal processing	Performance assurance of composites using thermal, mechanical, and chemical testing
Natural fiber composites [84]	Hydrothermal processing	Quality assurance of composites using mechanical and characterization techniques

4. Conclusions

In summary, PP-PCCF reinforced composites were recycled successfully using compression and injection molding. Thermally, the developed composites were found stable for variation of fiber loadings. Semiautomatic control of compression molding, distortion, and random incorporation of cotton fibers produced line cracks, asperities, and high surface roughness. However, the composite materials produced by injection molding have a smoother surface, lower number of internal defects, and smaller surface roughness. Mechanical investigations proved that PP-PCCF with 10% wt. of reinforcement can be used operationally for construction, medical, and automotive application due to flexibility in physical properties. However, an increase in fiber loading improved the tensile flexural and elastic modulus, flexural strength, and impact energy of PP-PCCF composites. Therefore, PP-PCCF with 40% wt. of reinforcement can be used for static-load applications due to an exponential decrease in ductility. The PP-PCCF composite with 30 wt.% of fiber loading showed peculiar behavior, probably because of the transition from ductile/plastic to elastic/brittle behavior. Besides the presence of surface and cross-sectional defects, all developed composites have manifested good erosive and abrasive behavior. Finally, the proposed recycling model and scheme of selection of polymer waste can be used for CSM. Moreover, testing techniques like mechanical, tribological, and surface characterization can also be utilized as quality tools for customer satisfaction.

Credit author statement

Abrar Hussain: Conceptualization, Methodology, Composite development, TGA, DSC analysis, Tensile testing, Bend testing, Impact testing, Abrasion and erosion testing, SEM analysis, review, and editing, Complete experimentation, Writing – original draft, Validation. Moreover, CE industrial implementation review, open system to closed system transformation, sustainability check. **Vitali Podgursky:** Overall main supervision, review, and editing, Validation, Conceptualization of composites fabrication. **Dmitri Goljandin:** Cutting, grinding of cotton waste. **Maksim Antonov:** Main supervision during tribological investigations – erosion and abrasion testing, review, and editing. **Mart Viljus:** Cotton waste and composite SEM characterization before and after testing. **Illia Krasnou:** Supervision during composite fabrication and testing, review, and editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Publication IV

Abrar Hussain, Podgursky V., Goljandin D., Antonov, M., Sergejev F., Krasnou I. Circular Production, Designing and Mechanical Testing of Polypropylene-Based Reinforced Composite Materials: Statistical Analysis for Potential Automotive and Nuclear Applications. Polymers,2023, 15, 3410. doi.org/10.3390/polym15163410.

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Circular Production, Designing, and Mechanical Testing of Polypropylene-Based Reinforced Composite Materials: Statistical Analysis for Potential Automotive and Nuclear Applications

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Abstract: The circularity of polymer waste is an emerging field of research in Europe. In the present research, the thermal, surface, mechanical, and tribological properties of polypropylene (PP)-based composite produced by injection molding were studied. The pure PP matrix was reinforced with 10, 30, and 40% wt. of pure cotton, synthetic polyester, and polyethylene terephthalate post-consumer fibers using a combination of direct extrusion and injection molding techniques. Results indicate that PP-PCPESF-10% wt. exhibits the highest value of tensile strength (29 MPa). However, the values of tensile and flexural strain were lowered with an increase in fiber content due to the presence of micro-defects. Similarly, the values of modulus of elasticity, flexural modulus, flexural strength, and impact energy were enhanced due to an increase in the amount of fiber. The PP-PCCF-40% wt. shows the highest values of flexural constant (2780 MPa) and strength (57 MPa). Additionally, the increase in fiber loadings is directly proportional to the creation of micro-defects, surface roughness, abrasive wear, coefficient of friction, and erosive wear. The lowest average absolute arithmetic surface roughness value (R_a) of PP and PP-PCCF, 10% wt., were 0.19 μm and 0.28 μm. The lowest abrasive wear value of 3.09×10^{-6} mm^3/Nm was found for pure PP. The erosive wear value (35 mm^3/kg) of PP-PCCF 40% wt. composite material was 2 to 17 times higher than all other composite materials. Finally, the single-step analysis of variance predicts reasonable results in terms of the *p*-values of each composite material for commercial applications.

Keywords: circularity and processing; fiber-reinforced composites and injection molding; micro characterization and surface roughness measurement; macro mechanical testing and green surface tribology; sustainability

Citation: Hussain, A.; Podgursky, V.; Goljandin, D.; Antonov, M.; Sergejev, F.; Krasnou, I. Circular Production, Designing, and Mechanical Testing of Polypropylene-Based Reinforced Composite Materials: Statistical Analysis for Potential Automotive and Nuclear Applications. *Polymers* **2023**, *15*, 3410. https://doi.org/10.3390/polym15163410

Academic Editors: Markus Gahleitner, Heidis Cano and Joaquín Hernández-Fernández

Received: 20 July 2023
Revised: 4 August 2023
Accepted: 13 August 2023
Published: 15 August 2023

1. Introduction

Circularity is an emerging field for the conversion of raw materials into green and sustainable products [1,2]. Innovation and new technological development have transformed open-loop manufacturing into closed-loop manufacturing [3,4]. The processing industries are focusing on the development of sustainable products [5]. Environmental pollution [6], energy crises [7], a decrease in materials' natural resources [8], and an increase in the population [9] have enhanced the use of recycled products [10]. Potentially, the utilization of polymer materials produces pre-consumer [11], post-consumer [12], end-waste [13], and end-of-life waste [14]. Pre- and post-consumer waste, as end streams, contained more than 95% of valuable material [15]. These types of commercial waste can be introduced for the fabrication of fiber-reinforced, sandwich, and particulate composite materials [7,16–18].

Traditionally, composite materials are made up of matrix and fiber phases [19]. The matrix phase fundamentally controls fiber dispersion, transferring of load, and protection from environmental impacts [20]. Epoxy resins like polyester (PES) [21], polypropylene (PP) [22], and polyethylene terephthalate (PET) [23] are commercial types of synthetic waste that show good adhesion properties, productivity, mechanical properties (flexural, tensile, tribological, impact, and hardness), and moisture resistance [24,25]. Principally, the fiber phase can enhance the quality and performance of composite materials [26]. Better hybridization with reinforcement fibers (glass, carbon, ashes, cotton, jute, kenaf, etc.), [27–32] improved the strength, toughness, and the commercial use of epoxy materials in the automotive, electronic, medical, and aerospace industries [33–35]. Due to unsaturation, bonding ability, plasticity, recyclability, versatility in utilization and mixing, mostly manufacturing techniques like compression molding [36], hand lay-up [37], resin transfer [38], and injection molding [39] are used for the transformation of waste materials into a product [18,40].

PP is a thermoplastic material commercially used for various applications [41,42]. Chemical stability, high mechanical strength, flexibility, toughness, and resistance to heat are salient features of PP [18,43–45]. Polyolefins hydrocarbon materials are the main source of its production [46]. Commercial use of these materials produces huge waste pollution in the environment [13,47]. Similarly, PET is a type of smart engineering material introduced as a replacement for glass in the packaging industry due to its excellent fracture strength and low weight [48]. The post-consumer PET (more than 1000 tons) waste is collected using curbside collections and deposit systems [49]. Bottle-to-bottle (for low contamination) and super-clean recycling (for high contaminated PET bottles) techniques have been introduced for the synthesis of post-consumer PET bottles [50,51]. PES synthetic polymer is cheaper and readily available as compared to other materials [52]. Simple (C-C) and complex (C=C) PESs present good bonding ability [53], low moisture absorption, suitable adhesiveness with natural fiber enforcement, and minimal binders for composite fabrication [54]. On the other hand, natural polymers are basically derived from plants and animals [55]. Plant polymers are made of cellulose, microfibrils, lignin, and hemicellulose [56]. Cotton [57], sisal [58], ramie [59], flax [60], hemp [61], and so on, are cheaper, commercial, and readily available materials [62]. Moisture absorption, low dimensional stability, and complexity in composition are major drawbacks of natural polymeric materials [63].

The well-established injection molding technique is mostly used commercially for the recycling of polymer wastes [64,65]. This technique enables the processing of different thermoplastics [66], thermosetting [67], and elastomers [67,68]. However, parameters like impurities in wastes [69], moisture [70], heating time [71], chemical reactions [72], and thermodynamic functions cause operational problems [73]. Poor control of these parameters lowered the engineering performance of manufactured products [74]. Extrusion techniques are mostly introduced to overcome these operational problems [2]. In extrusion, the mixed (matrix and fiber materials) are pushed through a heated cylinder [75]. The compound is squeezed and passed through various heating zones. The solid compound is converted into liquid. The die shapes the liquid compound into the desired shape [76]. PP-flax [77], PP-nut fibers [78], PP-jute [79], PP-glass fiber [80], and PP-carbon fibers [81] are some examples of manufacturing fiber-reinforced composite materials from the literature. Conventionally, the produced polymer products from virgin raw materials possess optimum physical properties [82]. PP as a matrix material also helps reinforce the fabric phase to gain interfacial interaction. The presence of interfacial interactions enhances the mechanical properties of fabricated composite materials. Circularity is the utilization of polymer waste to manufacture reasonable new commercial materials [83]. The mechanical properties of recycled composite materials are manifested lower in value due to low quality and performance of polymer waste [84]. The remelting and processing of extruded products through injection molding usually enhances the performance of polymer materials [85]. In injection molding, the compound mate is fed into a chamber through a hopper. The melting chamber mostly consists of four different zones. These zones operate at different temperatures and change compound mate into liquid [18,40]. A variety of polymers, like thermoplastics and thermosetting, can be processed for high or low volumes at low cost [86,87]. Thermoplastic materials are initially melted (first step) and forced into a mold cavity (second step) through an orifice [88]. The mold is kept cold. However, due to a polymer nature, thermosetting materials are melted and forced into a hot mold cavity through an orifice [67]. Modern innovation in molding technologies has made injection molding and extrusion techniques suitable for recycling due to excellent control of physical parameters (temperature, pressure, speed, time, etc.) and fast curing [89]. PP-sisal [90], PP-wood fibers [91], PP-hemp + glass fibers [92], and PP-nylon [93] are common examples of fabrication of composite materials. After development of composite materials, the quality and performance are inspected using thermal analysis, tensile, bend, impact, abrasive, and erosive testing for commercial applications [94–98]. Tensile properties ensure the withstand ability, fracture ability, stiffness, and percentage of plastic deformation [99,100]. Usually, these properties are correlated with structural applications. In parallel, bend testing explored the flexural properties of recycled composite materials under compressive conditions [101]. Maximum flexural stress, strain, and elongation of plastics [102], thermosetting [103], elastomers [104], composites, and metals are investigated for customer satisfaction [105]. The degree of surface finishing is evaluated using erosion [106], fatigue, creep testing, and surface characterization [107]. Mostly, line cracks, asperities, rough regions, pits, and grooves exist on the surface of composite materials [108]. These defects simulate material failure [109] and are mostly controlled using tribological investigations [110]. Similarly, materials characterization like scanning electron microscope analysis and surface profilometer measurements [111] provide immediate results to optimize manufacturing of polymeric materials for commercial applications. Collectively, mechanical, thermal, and surface techniques assured customer satisfaction and diversity [112].

The motive of this study is to introduce the concept of circularity in polymer industries for the fabrication of smart recycled composite materials. The post-consumer (PC) cotton fabric (CF), synthetic polyester fabric (PESF), and polyethylene terephthalate fabric (PETF) waste are used as reinforced fibers to fabricate polypropylene (PP) based PP-PCCF, PP-PCPESF, and PP-PCPETF composite materials. Traditional single-step direct extrusion in association with injection molding is introduced as a processing technique to enhance the physical and thermal properties of manufactured products. The fiber loadings (PCCF, PCPESF, and PCPESF) of 0 (pure PP as reference material), 10, 30, and 40% are reported for potential commercial structural and environmental applications. A scanning electron microscope (SEM) and optical and mechanical profilometers are utilized to characterize composite materials qualitatively and quantitatively for surface and cross-sectional defects. The quality and performance of recycled composite materials are analyzed using thermal, surface, mechanical, and tribological techniques for potential commercial applications and customer satisfaction. The statistical single-factor analysis of variance is established to manifest the effect of each property of an individual composite material for commercial application. Finally, the proposed framework of closed-loop pilot production of composite manufacturing can be started as a concept of circularity (recycling of polymers with almost negligible waste) in polymer recycling industries.

2. Materials and Methods

2.1. Selection Criteria and Specification of Polymer Waste Materials

Pure polypropylene (PP) powder was used as a matrix phase. The PP powder was purchased from Egyeuroptene (Jorvas, Finland). According to the American Society for Testing and Materials (ASTM) D 1238 testing method, PP has a melt-flow index of 14 g/10 min at 2.16 kg and 210 °C at melting temperature. These properties of PP were provided by the company. The PP provides excellent adhesion, interfacial strength, moisture resistance, thermal resistance, and fiber wettability. The selected material is shown in Figure 1a.

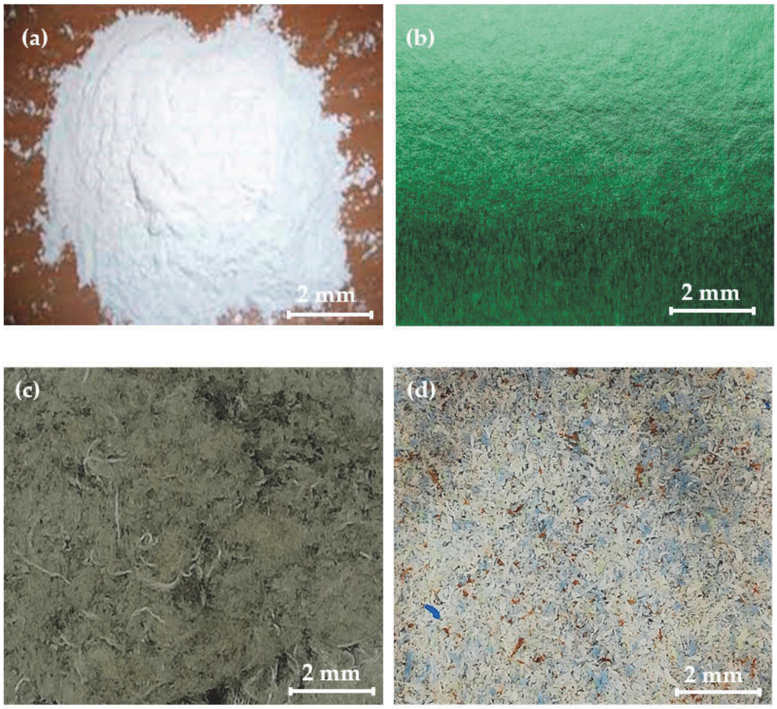


Figure 1. PP and ground post-consumer polymer fabric materials: (**a**) Pure PP, (**b**) PCCF, (**c**) PCPESF, and (**d**) PCPETF.

The subjective assessment (manual), ASTM D5034-08 (Grab test) [113], and ASTM D5034-06 (Strip test) [113] are used to investigate the physical and tensile properties of PCCF, PCPESF, and PCPETF materials [113].

PCCF (Figure 1b) was supplied by the local industry of Estonia. The PCCF has a density of 1.55 g/cm^3, an elongation range of 5–25% ± 1.5%, tensile strength of 0.06 MPa ± 0.001, design strength of 0.10 ± 0.004 MPa, breaking strength of 0.04 ± 0.002 MPa, and a weight of 237 g/m^2. The PCCF was heated at 60 °C for 70 min to remove the moisture.

PCPESF PCDT (poly-1.4 c yclohexyl-di-methylene terephthalate) (Figure 1c) was also supplied by the local industry of Estonia. The PCPESF represents post-consumer polyester fibers. The PCPES has a density of 1.45 g/cm^3, an elongation range of 8–30% ± 1.7, tensile strength of 0.08 ± 0.004 MPa, design strength of 0.13 ± 0.004 MPa, breaking strength of 0.06 ± 0.002 MPa and a weight of 230 g/m^2.

PCPETF (Figure 1d) was derived from post-consumer beverage bottles. The PCPETF has a density of 1.45 g/cm^3, an elongation of 6–12 ± 2%, and a tensile strength of 90 ± 10 MPa.

Before processing, all materials were dried in an oven at 60 °C for 4 h to remove moisture and humidity.

2.2. Sorting, Separation, and Grinding of Post-Consumer Waste

After collection, the manual separation and sorting of post-consumer fabric waste (PCCF, PCPESF, and PCPETF) were performed, see Figure 2. The fabric waste in a purity range of 95–99.99% was selected for further processing. Before grinding, the fabric waste is cut into small pieces. The direct grinding of these small pieces was performed to transform fabric waste into fine fibers (PCCF and PCPESF) and flakes (PCPETF) at a speed and time of 300 rpm and 10 min, respectively [114]. The step of grinding was repeated four times to get a uniform fine-sized distribution of ground fine fibers in terms of length, diameter, and area. The SEM was used to calculate the length, diameter, and area of waste fibers. A bunch of 50 fibers (individually each PCCF, PCPESF, and PCPETF) were selected and coated with gold (Au) using a physical vapor deposition technique. A gold thin film of 2 nm was deposited, and the waste fibers were characterized. The average size distribution of waste fibers (Table 1) was measured using SEM.

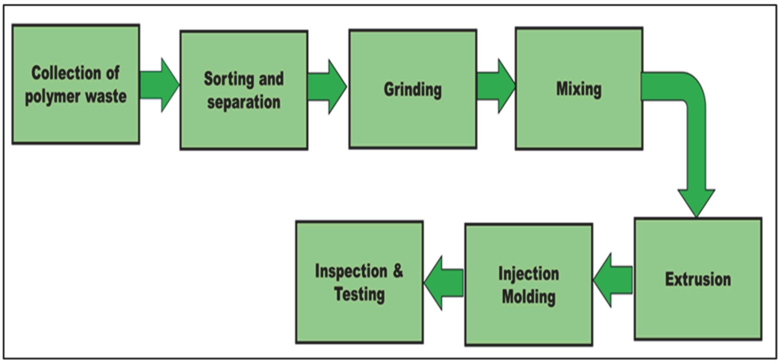


Figure 2. Steps of fabrication of fiber-reinforced and particulate composite materials.

Table 1. The average length, diameter, and area measurement of PCCF, PCPESF, and PCPETF ground fibers.

Post-Consumer Reinforced Fiber	Length (mm)	Diameter (μm)	Area (μm^2)
PCCF	3.000 ± 0.001	18.000 ± 0.002	250.000 ± 0.004
PCPESF	3.500 ± 0.001	17.000 ± 0.002	245.000 ± 0.004
PCPETF	0.310 ± 0.0002	0.010 ± 0.002	0.190 ± 0.0004

2.3. Fabrication of Fiber-Reinforced and Particulate Composite Materials

At the start, the PP was mixed using a locally manufactured semiauto cylindrical mixer with 10, 30, and 40 wt.% fiber (PCCF, PCPESF, and PCPETF) loadings before compounding. The time and speed of mixing were 15 min and 80 rpm, respectively. In the second step, mixed polymer materials (matrix and fibers) were compounded using a twin-screw compounder Brabender extrusion machine (PLE 651-plasic corder) at temperature, speed, torque, and time of 190 °C, 40 rpm, 60 Nm, and 7 min, respectively. The extruder was operated at a temperature of 175 °C in the first zone, 180 °C in the second zone, and 190 °C in the third and fourth temperature zones. The extruded mate was shaped into long cylindrical wires.

The fabrication steps are shown in Figure 2. These wires were ground into pellets or beads of size 2 mm and used as raw material for injection molding. The pellets were dried for 3 h at a temperature of 60 °C. The injection molding machine (Battenfeld BA 230A) can operate at different temperature zones. Therefore, the pellets were passed through different temperature zones (120, 150, 180, and 190 °C) for melting. Finally, small pellets were injected into the mold cavity and heated at a temperature of 190 °C. The injection, cooling, and molding open times were 8 s, 25 s, and 30 s, respectively. All composite materials were designed into ASTM test specimens of size 150 mm (length) × 25 mm (width) × 4 mm (thick) products for mechanical, abrasion, surface, and erosion testing, see Figure 3a,b.

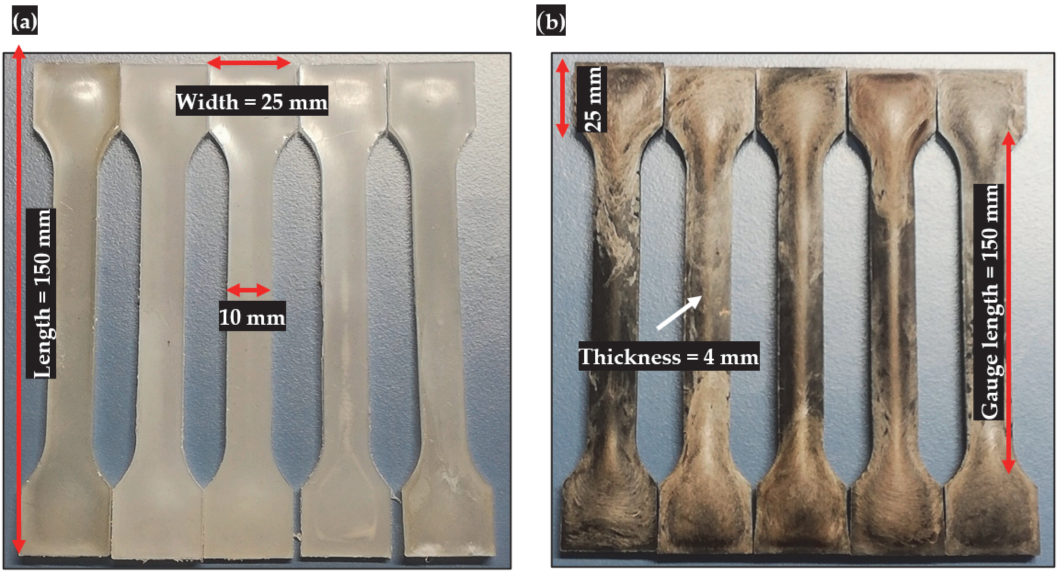


Figure 3. The representative images of fabricated composite materials: (**a**) Pure PP and (**b**) PP-PCCF fiber-reinforced composite materials.

2.4. Quality and Performance Evaluations of Recycled Composite Materials

The quality and performance of recycled composite materials were evaluated using thermal analysis, mechanical testing, tribological investigations, surface testing, and SEM characterization. The inspection and testing of composite materials were performed for customer satisfaction and commercial applications. The quality control diagram for the testing of materials is shown in Figure 4.

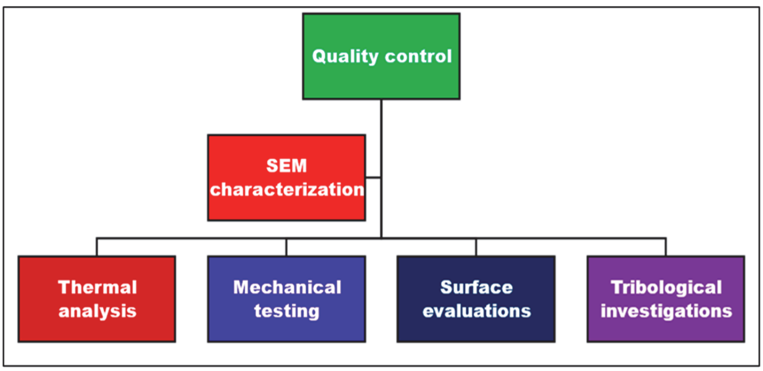


Figure 4. Industrial quality control and testing of materials.

2.4.1. Thermal Evaluations

The thermal differential scanning calorimetric (DSC) tests were performed using simultaneous thermal analyzers (Model STA 449 F3 Jupiter, NETZSCH Co., Houston, TX, USA). In the DSC thermal test, 10 mg of each individual composite material was heated and cooled from 0 °C to 250 °C and 250 °C to 0, respectively. The heating and cooling rate was kept constant at 15 °C/min. Moreover, pure nitrogen was used as a medium for testing at a rate of 50 mL/min. The melting temperature, degree of crystallinity, and crystallization temperature of developed composite materials were measured during changes in endothermic fusion and exothermic cooling curves. The DSC results are shown in Figure 5a–c.

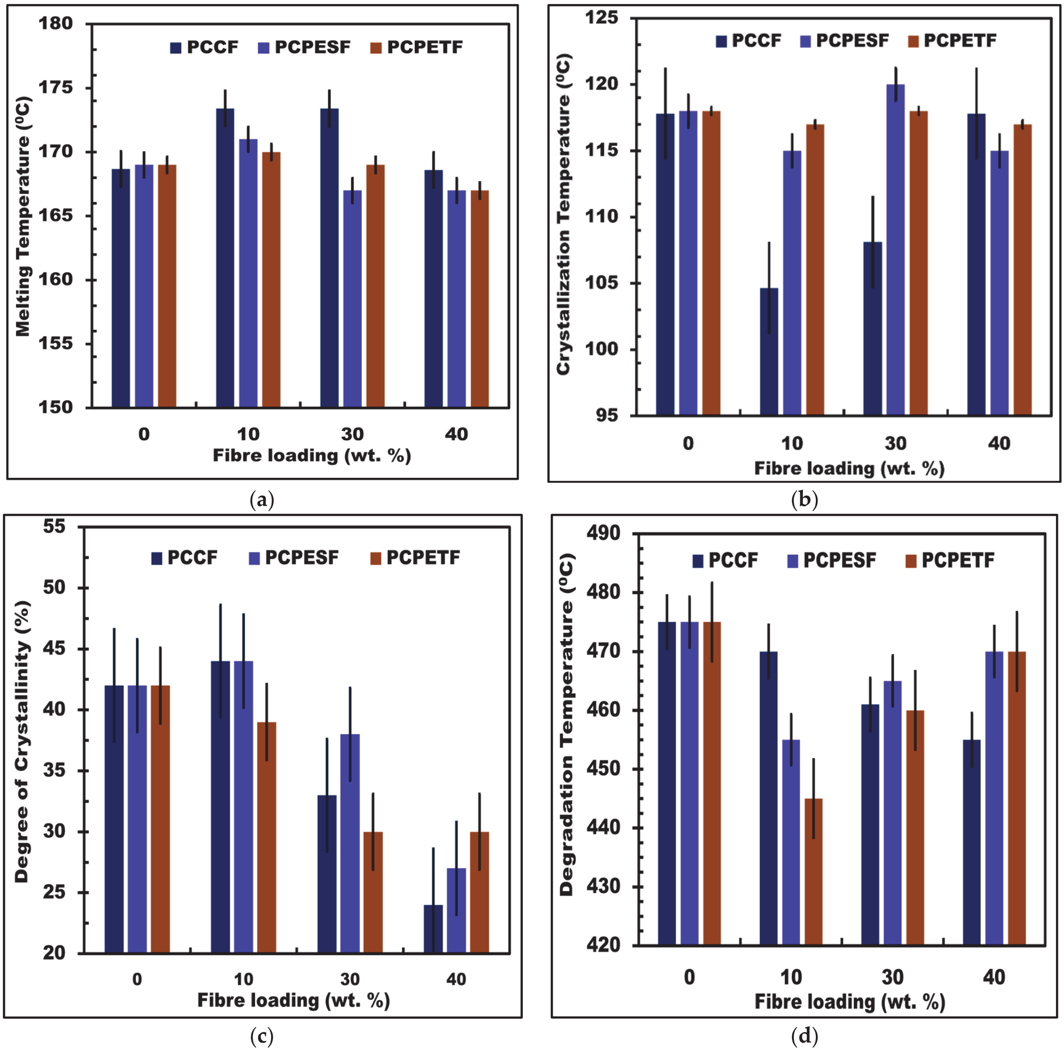


Figure 5. Results of DSC and TGA analysis of PP-PCESF and PP-PCPETF fiber-reinforced and particulate composite materials: (**a**) melting temperature, (**b**) crystallization temperature, (**c**) degree of crystallinity, and (**d**) degradation temperature.

Similarly, the thermogravimetric (TGA) tests were carried out using a thermogravimetric analyzer (TGA 1000 system, Anderson Materials Evaluation, Inc., Columbia, MD, USA). In the TGA thermal test, 10 mg of each individual composite material was heated from 0 °C to 600 °C at a heating rate of 10 °C/min in alumina ceramic crucibles. The balance and sample purges of pure nitrogen at the rates of 20 mL/min and 50 mL/min were used as a medium of testing. In this study, the degradation temperature of recycled composite materials was observed. The average values of each individual composite material were measured to quantify the thermal degradation for practical applications. The TGA results are depicted in Figure 5d.

2.4.2. Mechanical Testing

The tensile and bending properties of manufactured composites were observed using the Universal Testing Machine (UTM Model 5820, Instron Co., Norwood, MA, USA). The tensile and flexural tests were carried out according to ASTM D3039 [115] and ASTM D5467 [116] standards at a rate of 50 mm/min. The specimens of size 4 (thickness) × 25.4 (width) × 150 mm (length) were manufactured using injection molding. The machine comprises the lower (fixed) and upper jaws (moveable). The distance between the two jaws was kept equal to the gauge length (100 mm) of the specimen. The length under analysis is known as gauge length. The gauge length of bending and tensile tests was 100 mm. The data were recorded and analyzed using Acquisition software. At least twenty tests were performed for each individual composite material. Moreover, ASTM A370 impact tests were performed to measure the impact energy of composite materials. The quantitative and qualitative analysis in terms of tensile strength, modulus of elasticity, plastic deformation, flexural strength, flexural constant, flexural strain, and impact toughness help in the prediction of performance and quality of recycled composite materials. The results are presented in Table 3 and Figures 11–13.

2.4.3. Tribological Testing

The CETR Bruker UMT-2 tribometer was used to calculate the abrasive wear of developed composites. The SiC (P150 grade) sandpaper was used for the abrasion of samples. The composite pins of 4 (thickness) × 5 (width) × 25 mm (length) were slid at 0.1 m/s speed and 1 N force as a counter body on SiC sandpaper. The abrasive wear was measured for 18 m of sliding distance. The wear rate (***W***) was calculated using the following equation:

$$\boldsymbol{W} = \frac{\boldsymbol{V}}{\boldsymbol{L} \times \boldsymbol{S}} \quad (1)$$

where ***V*** is the volumetric wear loss (mm^3), ***L*** is the normal load (N) applied during the test, and ***S*** is the total sliding distance (m).

The COF graph and average COF value were calculated by CETR/Bruker UMT Viewer software (https://www.bruker.com/en/products-and-solutions/test-and-measurement/tribometers-and-mechanical-testers/umt-tribolab.html, accessed on 2 July 2023). The results are shown in Figure 14 and Table 4.

2.4.4. Erosion Testing

The locally manufactured four-channel accelerator erosion machine was used for determining the erosive wear of manufactured composite materials. Silica sand (SiO_2) of 6 kg in quantity was used as an erosive medium. The size of silica sand particles was in the range of 0.1–0.6 mm. The erosion tests were repeated three times to measure the weight loss of composite materials before and after each test. The weight loss was measured using a Mettler Toledo ME204 balance with an accuracy of 0.10 mg. Moreover, impact angle, velocity, and time during the tests were 30°, 30 m/s, and 30 min, respectively. The temperature was kept at 25 °C. The results are shown in Figure 15 and Table 4. The specific weight loss M (mg/kg) at each step can be calculated using the following formula:

$$\boldsymbol{M} = \frac{\Delta \boldsymbol{m}}{\boldsymbol{G} \times \boldsymbol{v}} \quad (2)$$

where $\Delta\boldsymbol{m}$, ***G***, and ***v*** are the weight loss of each sample, weight of sand, and share of sand per sample.

After the determination of ***M***, erosive wear (volumetric loss) ***E*** can be formulated as:

$$\boldsymbol{E} = \frac{\boldsymbol{M}}{\boldsymbol{\rho}} \quad (3)$$

where $\boldsymbol{\rho}$ is the density (mg/mm^3) of the sample.

2.4.5. Surface Characterization

The surface morphology of composite materials before and after testing was investigated by the scanning electron microscope (SEM) (Zeiss EVO® MA-15 system, Oberkochen, Germany) with LaB6 cathode in the secondary electron mode, applying an accelerating voltage of 10–15 kV at a 6.5–8.5 mm working distance. About 20 fibers were selected and coated with gold using physical vapor deposition. The thickness of the gold coating was 2 nm. The fibers were characterized using SEM. The length, diameter, and area were measured digitally using specially installed software. The calculated values are shown in Table 1.

Additionally, a mechanical profilometer (Mahr Perthometer PGK120) and an optical profilometer (Contour GT-K0+ 3D) were used to measure the surface roughness of recycled composites. The average surface roughness R_a (µm), root mean square roughness R_q (µm), maximum profile peak height R_p (µm), average maximum height of the profile R_z (µm), and maximum height of the profile R_t (µm) were measured and correlated with SEM micrographs. The results are shown in Figures 7–10.

3. Results

3.1. DSC and TGA Thermal Analysis of Composites

Figure 5a–d and Table 2 express the results of the DSC and TGA investigations. The composite materials produced after injection molding were under consideration. The melting, crystallization, degradation temperature, and crystallinity of pure PP were 169 °C, 119 °C, 480 °C, and 42%, respectively. Pure polymers always show sharpness in values of physical parameters (like temperature, force, stress, energy, etc.) during the investigation due to the highest purity [117].

Table 2. The demonstration of numerical values of melting, crystallization, degradation temperature, and degree of crystallinity of all developed composite materials.

Composite Family	Melting Temperature (°C)	Crystallization Temperature (°C)	Degradation Temperature (°C)	Degree of Crystallinity (%)
Pure PP	169 ± 2	118 ± 4	475 ± 5	42 ± 5
PP-PCCF 10% wt.	173 ± 2	105 ± 4	470 ± 5	44 ± 5
PP-PCCF 30% wt.	173 ± 2	108 ± 5	461 ± 6	33 ± 4
PP-PCCF 40% wt.	169 ± 1	118 ± 5	455 ± 7	24 ± 5
PP-PCPESF 10% wt.	171 ± 2	115 ± 3	455 ± 5	44 ± 3
PP-PCPESF 30% wt.	167 ± 2	120 ± 2	465 ± 4	38 ± 4
PP-PCPESF 40% wt.	167 ± 2	115 ± 3	470 ± 4	27 ± 4
PP-PCPETF 10% wt.	170 ± 1	117 ± 1	445 ± 7	39 ± 4
PP-PCPETF 30% wt.	169 ± 1	118 ± 1	460 ± 8	30 ± 3
PP-PCPETF 40% wt.	167 ± 1	117 ± 1	470 ± 8	30 ± 4

However, fiber addition acts as an impurity. Therefore, PP-PCCF, PP-PCPESF, and PP-PCPETF (with 10, 30, and 40% fiber loadings) composite materials melt, crystallize, and degrade within a specific range of temperature [118]. The nature of fiber, length, diameter, area, density, weight, and amount of fiber affect the thermal, tensile, bending, impact [119], abrasive, and erosion properties of composite materials [120]. The degree of crystallinity of fiber-reinforced and particulate composites is calculated by using the following formula:

$$\%Xc = \frac{\Delta H_f}{\Delta H_f^o} \times \frac{100}{w} \tag{4}$$

where Xc, w, ΔH_f, and ΔH_f^o are the amount of fraction, weight, the heat of fusion of the composite material, and the heat of fusion of a 100% reference material, respectively.

The crystallization temperature of PP-PCPESF and PP-PCPETF composites with 10 and 40% fiber amounts was constant with a value of 115 °C. The constant value of crystallization temperature appeared due to the presence of the matrix phase in higher quantities [121,122]. However, the PP-PCCF, PP-PCPESF, and PP-PCPETF composites with 30% fiber addition exhibit complex behavior due to the presence of different phases (amorphous, crystalline, and semi-crystalline), see Figures 5b and 6. Similarly, the degree of crystallinity was demonstrated in Figure 5c. The level of crystallinity decreases with an increase in fiber content. Fiber nature, length, and random orientation produced amorphous and crystalline phases [123]. At the lower addition of fibers, nucleation sites come into existence. These sites increase the level of crystallinity (Figure 6). Mechanical properties fundamentally rely on the extent of crystallinity (orientation of crystals in a specific direction) [124]. Therefore, pure PP, PP-PCCF, PP-PCPESF, and PP-PCPETF composites with 10% wt. impart good mechanical properties; see Figures 11–13 and Table 3. However, a decrease in crystallinity produces brittleness, especially in manufactured composites between 30 and 40% wt. PCCF, PCPESF, and PCPETF loadings; see Figure 5c. PP-PCCF, PP-PCPESF, and PP-PCPETF composites (with 40% wt. fiber loadings) expressed the lowest crystallinity and highest brittleness; see Figures 11–13 and Table 3. An increase in crystallinity with the enhancement of 10% wt. of fiber-reinforced materials was observed. However, an increase in fiber content produces micro defects. These defects decreased the adhesion between the matrix–fiber interface and interstitial sites. Moreover, the random orientation of fibers, the nature of reinforced materials, deformation of the surface of fibers due to periodic grinding, and retention in cooling rates also caused the decrease in crystallinity of recycled composite materials between 30 and 40% wt. of fiber loadings (PCCF, PCPESF, and PCPETF). The thermal capacity and withstanding ability of produced composites are shown in Figure 5d. The degradation temperature of pure PP was 475 °C. The addition of PCCF, PCPESF, and PCPETF caused the lowering of degradation temperature. The PP-PCCF group of composite materials manifested degradability variations from 452 °C to 475 °C. The PP-PCPESF composite family possesses thermal withstand ability in the range from 455 °C to 470 °C. On the other hand, all types of PP-PCPETF composites expressed thermal capacity in the range from 445 to 470 °C. Closeness in the values of degradation temperature is due to the presence of a major PP matrix phase, matrix (PP)-fiber (PCCF, PCPESF, and PCPETF) interactions, and difference in C-C bonding [125].

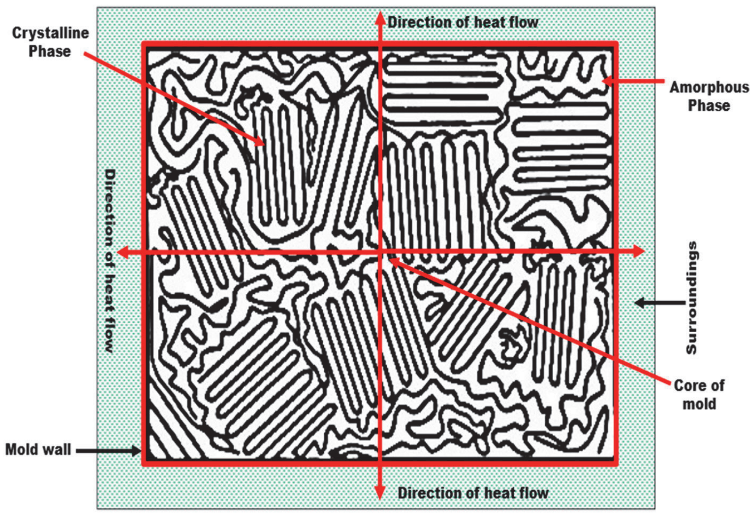


Figure 6. Solidification mechanism of pure PP and all recycled composite materials.

Table 3. The results of tensile, flexural, and impact properties of natural and synthetic-reinforced developed composite materials.

Composite	Tensile Testing			Bend Test			Impact Test
PCCF Content (% wt.)	**Tensile Strength σ(MPa)**	**Tensile Strain (%)**	**Modulus of Elasticity E(MPa)**	**Flexural Strength σ(MPa)**	**Flexural Strain (%)**	**Flexural Constant E(MPa)**	**Impact Energy (kJ/m^2)**
PP	31 ± 2	10 ± 1.75	1226 ± 325	42 ± 6	14 ± 2	1172 ± 475	4.9 ± 0.30
PP-PCCF 10% wt.	26 ± 2	8 ± 1.50	1476 ± 400	51 ± 6	12 ± 2	2069 ± 475	4.8 ± 0.30
PP-PCCF 30% wt.	24 ± 2	4 ± 1.75	2521 ± 425	35 ± 6	10 ± 2	2130 ± 475	4.4 ± 0.30
PP-PCCF 40% wt.	22 ± 2	4 ± 1.75	2751 ± 430	57 ± 6	5 ± 2	3780 ± 475	5.5 ± 0.30
PP-PCPESF 10% wt.	29 ± 2	10 ± 2	1401 ± 125	41 ± 1	13 ± 1.25	1820 ± 325	2.80 ± 0.60
PP-PCPESF 30% wt.	25 ± 2	5 ± 2	1526 ± 125	43 ± 1	12 ± 1.25	2119 ± 325	2.81 ± 0.60
PP-PCPESF 40% wt.	23 ± 2	4 ± 2	1591 ± 125	41 ± 1	9 ± 1.25	2205 ± 325	3 ± 0.60
PP-PCPETF 10% wt.	25 ± 2	5 ± 2	1167 ± 85	40 ± 2	12 ± 1.50	1909 ± 275	2.60 ± 0.60
PP-PCPETF 30% wt.	22 ± 2	4 ± 2	1117 ± 85	40 ± 2	10 ± 1.50	2150 ± 275	3 ± 0.60
PP-PCPETF 40% wt.	22 ± 2	3 ± 2	790 ± 85	39 ± 2	9 ± 1.50	2140 ± 275	3.4 ± 0.60

3.2. Solidification of Composites

Figure 6 represents the cooling model (solidification) of recycled composite materials. At a melting point of 190 °C, the melting compound mate exists in liquid form.

Thermodynamically, the heat in the form of energy flows from the core of the mold to its surroundings. Due to the flow of energy, the molecules of polymer materials start to solidify. The fibers (PCCF, PCPESF, and PCPETF) provide micro-sites to molecules of PP for solidification. Therefore, matrix–fiber interface and crystalline phases came into existence. According to Figure 6, a mostly amorphous phase exists at the boundary of the mold wall and surroundings due to heat retention. The retention of heat energy kept the temperature at a higher value at the melt mate-mold wall junction. The access to heat energy is produced by thermal barriers and heat gradients. These conditions prolonged the crystallization time of composite materials. Moreover, thermal barriers and heat gradients cause re-melting of the embryo, displacement of molecules, and random placement of reinforced (PCCF and PCPESF) and particulate fibers (PCPETF flakes). Therefore, the amorphous phase comes into existence with various surface defects, see Figures 7–10. These micro defects play an important role in the decreasing mechanical properties of recycled composite materials (Figures 11–15 and Table 3). However, the crystallization of all polymeric composite materials starts from the core of the mold. The degree of crystallinity is mentioned in Figure 5c and Table 2. The degree of crystallinity is directly proportional to the enhancement of mechanical properties of composite materials; see Figures 11–15 and Table 3.

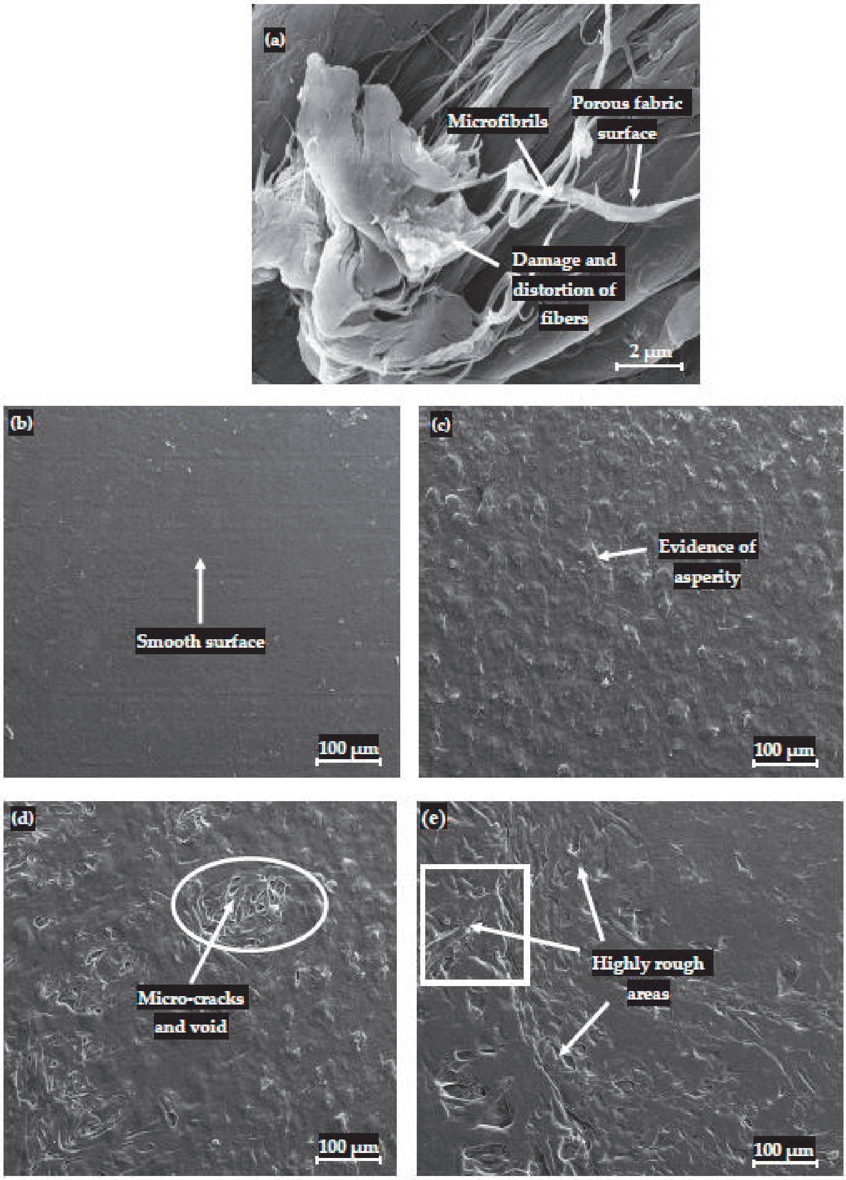


Figure 7. SEM analysis and surface characterization of pure cotton, PP, and PP-PCCF composites materials: (**a**) PCCF, (**b**) pure PP, (**c**) PP-PCCF 10% wt., (**d**) PP-PCCF 30% wt., and (**e**) PP-PCCF 40% wt.

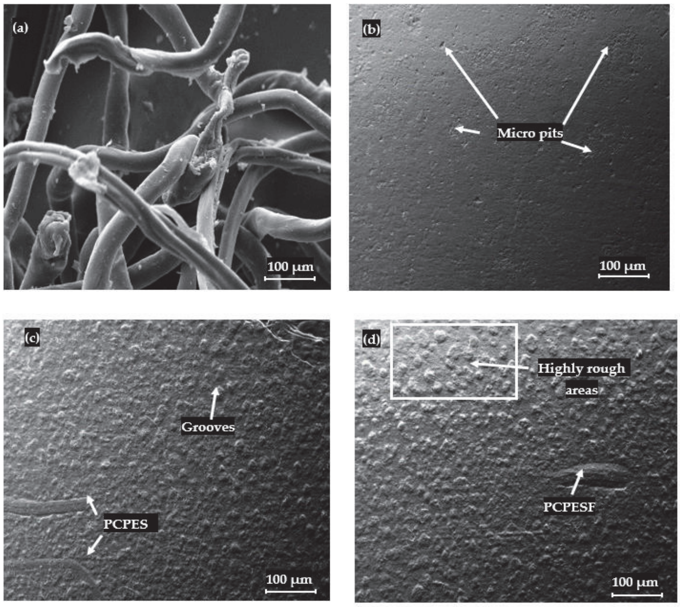


Figure 8. SEM analysis and surface characterization of synthetic polyester fibers and PP-PCPESF composites materials: (**a**) PCPESF, (**b**) PP-PCPESF 10% wt., (**c**) PP-PCPESF 30% wt., and (**d**) PP-PCPESF 40% wt.

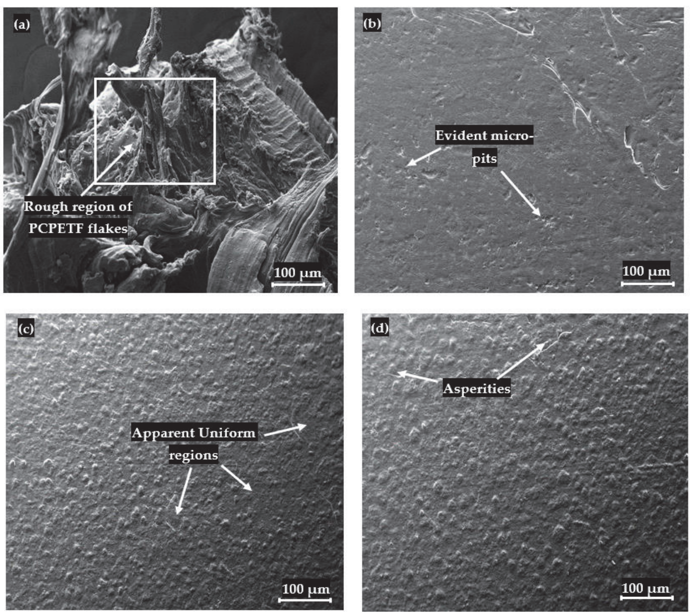
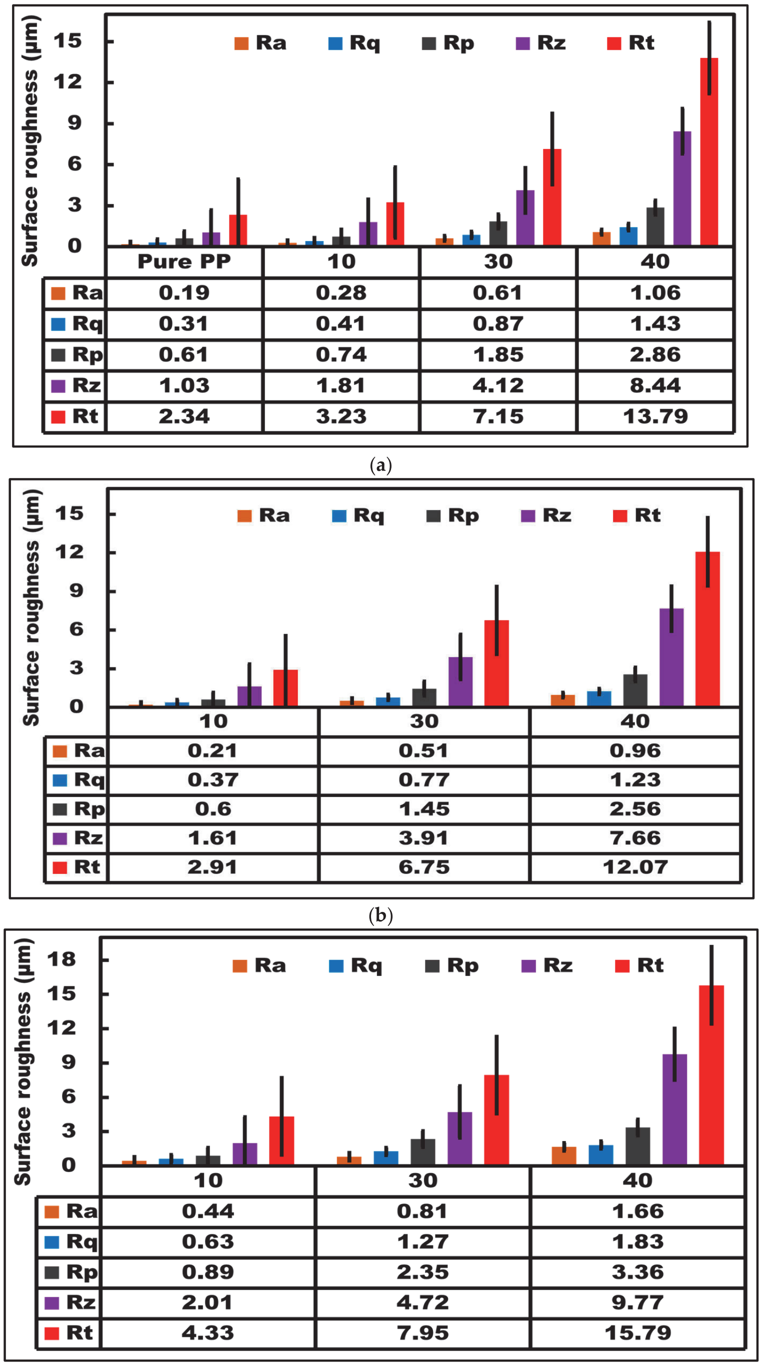


Figure 9. SEM analysis and surface characterization of pure polyethylene terephthalate fibers and PP-PCPETF composites materials: (**a**) PCPETF, (**b**) PP-PCPETF 10% wt., (**c**) PP-PCPETF 30% wt., and (**d**) PP-PCPETF 40% wt.



(a) (b) (c)

Figure 10. Quantitative surface analysis and average roughness parameters of developed composite materials: (**a**) PP-PCCF composite materials, (**b**) PP-PCPESF composite materials, and (**c**) PP-PCPETF composite materials.

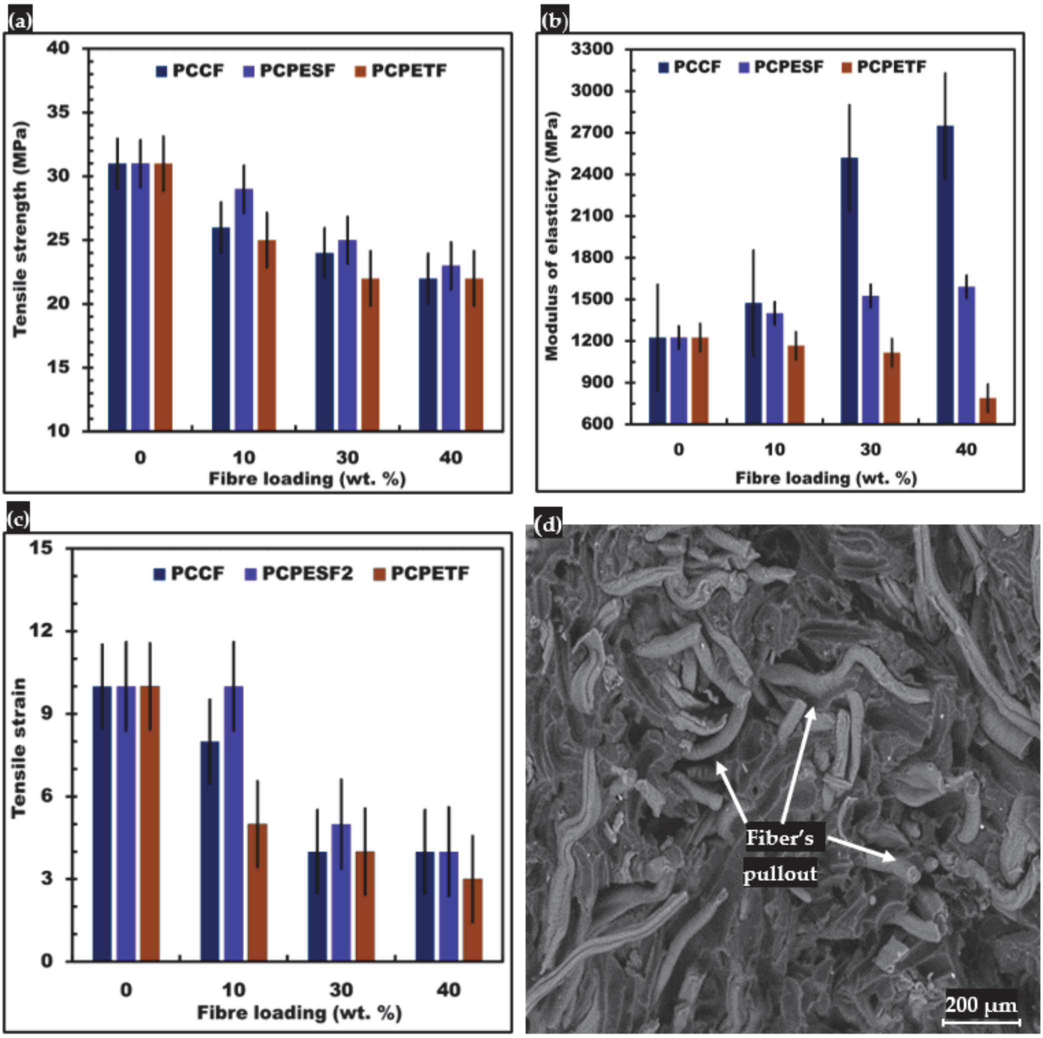


Figure 11. Comparative results of tensile properties of developed composite materials: (**a**) Tensile strength, (**b**) modulus of elasticity, (**c**) tensile strain, and (**d**) SEM image of fibers pull out of PP-PCPESF 40% wt. composite material after tensile test and fracture.

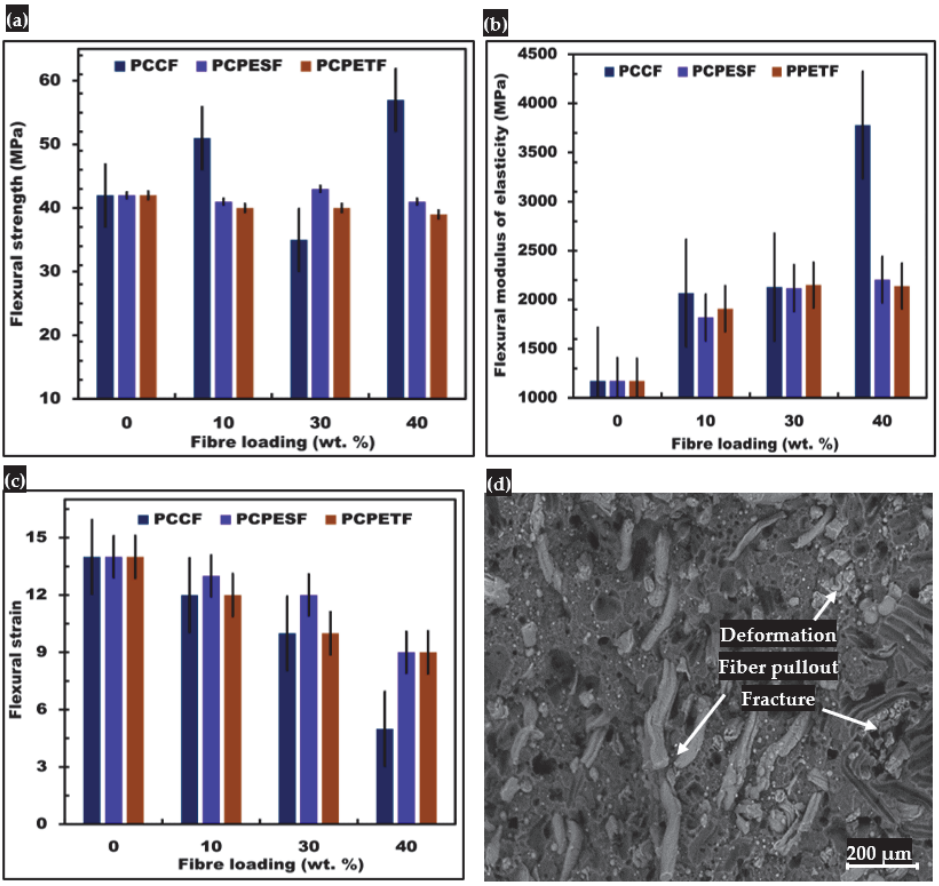


Figure 12. Comparative results of flexural properties of developed composite materials: (**a**) flexural strength, (**b**) flexural constant, (**c**) flexural strain, and (**d**) SEM image of fractured PP-PCPESF 40% wt. composite material after the flexural test.

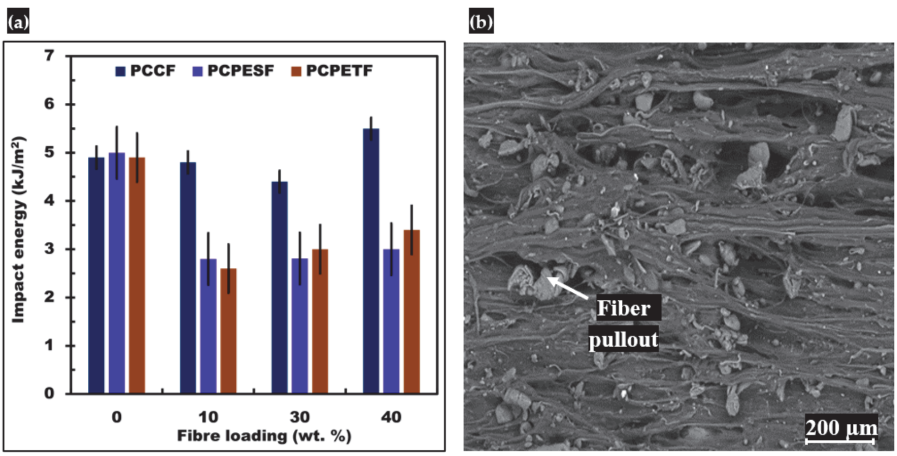


Figure 13. Comparative results of impact energy of developed composite materials: (**a**) impact energy, (**b**) SEM image of deformed PP-PCPESF 40% wt. composite material after impact test.

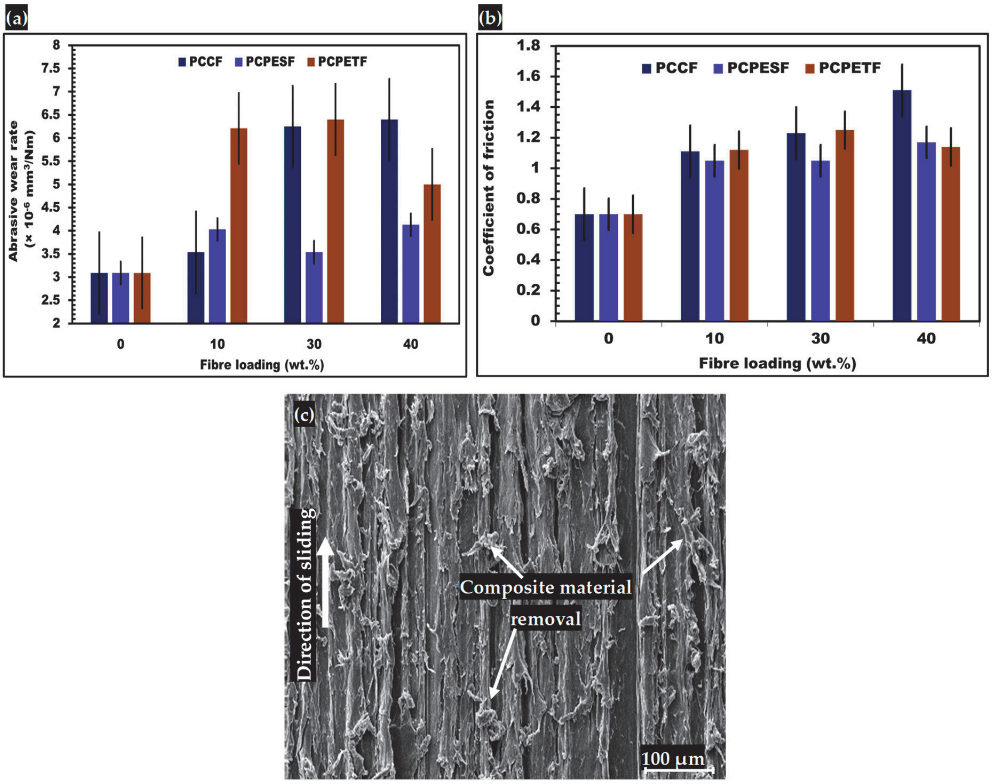


Figure 14. Results of abrasive wear, COF, and SEM characterization of manufactured composite materials: (**a**) results of abrasive wear rate values of PP, PP-PCCF, PP-PCPESF, and PP-PCPETF developed composite materials, (**b**) results of COF values of PP, PP-PCCF, PP-PCPESF, and PP-PCPETF fabricated composite materials, and (**c**) representative SEM image of pure PP after abrasion test.

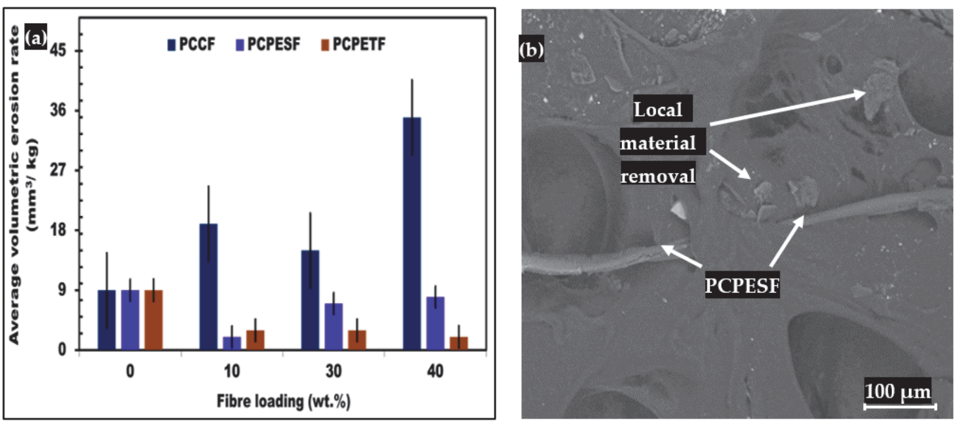


Figure 15. Results erosive wear and SEM characterization of recycled composites: (**a**) erosive wear rates of PP, PP-PCCF, PP-PCPESF, and PP-PCPETF developed composite materials, and (**b**) representative SEM image of PP-PCPESF-40% wt. composite material.

3.3. SEM Characterization of Composites

The representative SEM micrographs of PP-PCCF, PP-PCPESF, and PP-PET are presented in Figure 7, Figure 8, and Figure 9, respectively. Figure 7a shows a SEM image of pure PCCF. Microfibrils, porosity, distortion, and damage appear on the surface of fibers. These defects can contribute to lowering the quality and performance of recycled composite materials. The pure PP's surface appeared smooth; see also Figures 6 and 10. In any case, composites (with 10, 30, and 40% wt. fiber loadings) expressed the presence of PCCF, PCPESF, and PCPETF fibers. The existence of fibers produces surface defects like micro cracks and asperities. In pure PP, particles of equal size are compressed under high temperatures. PP powder's particles of the same nature are cooled at the same rate to form an embryo. Hence, a PP polymer product with a uniform surface is formed (Figures 6 and 7b). Figure 7c shows the asperities and evidence of PCCF fibers on the surface of PP-PCCF-10% wt. composite material. Micro-cracks and voids appeared on the surface (Figure 7d) of PP-PCCF-30% wt. composite material due to the PP-PCCF interface, poor adhesion, and the nature of cotton fiber. PCCF increment makes composites hard and brittle. Therefore, surface defects become prominent, see Figure 7e.

The addition of another material (for instance, PCPESF and PCPETF) as a reinforced phase changes the thermodynamic [126], chemical, and physical properties of composites [127]. Thermally, the synthesized composites melt, degrade, and recrystallize within a specific range of temperature [128]. The oriented and random incorporation of fiber phase (for instance, PCPESF, see Figure 8a) allows composites to cure at different cooling rates. The difference in temperature as a thermal gradient produces expansion or contraction in composite materials. Therefore, it appeared as micro pits on the surface of PP-PCPESF with 10% wt. composite material, see Figures 6 and 8b. Figure 8c represents the appearance of PCPESF and matrix–fiber poor adhesion, grooves, and highly rough areas on the surface of PP-PCPESF-30% wt. recycled composite. The degree of the mentioned micro defects has become prominent on the surface of PP-PCPESF-40% wt. fabricated composite (Figure 8d) due to the highest amount of PCPESF and other parameters. The PCPETF in the form of flakes was used for the fabrication of PP-PCPETF composite materials. The PCPETF flakes exhibit surface deformation and rough regions (Figure 9a). The plastic deformation and distorted regions can cause poor adhesion between the PP and PCPETF interface. The micro pits (Figure 9b) become more evident due to the flake-like shape and size of PCPETF. Figure 9c has manifested some uniform regions on the surface due to crystallinity and mutual PP-PCPETF compound (composite) formation. However, conventional asperities and line cracks were observed on the surface of PP-PCPETF-40% wt. composite material, see Figure 9d [127].

3.4. Surface Roughness Evaluations of Composites

The quantitative surface analysis of recycled composite groups is shown in Figure 10. The minimum values of surface roughness parameters were found for pure PP (Figure 10a). Naturally, the increase in fiber addition (i.e., PCCF, PCPESF, and PCPETF) produced various surface defects (Figures 6–9). Therefore, according to Figure 10a–c, the values of Ra, Rq, Rp, Rz, and Rt enhance individually for each composite material with an increase in fiber loading from 0 to 40%. All composite materials with 30% wt. fiber loading manifested complex behavior. The surface-roughness parameters of PP-PCPETF composite materials are greater than the PP-PCPESF and the PP-PP-PCCF composite family.

3.5. Mechanical Testing and SEM Characterization of Composites

3.5.1. Tensile Testing and SEM Analysis of Fracture Surface

The results of tensile properties of PP-PCCF, PP-PCPETF, and PP-PCPES composites with different fiber loading are shown in Figure 11. The experimental data indicate that tensile strength (Figure 11a) and tensile strain (Figure 11c) decrease with an increase in PCCF, PCPETF, and PCPESF fiber contents. The increment of fiber loadings (all PCCF, PCPESF, and PCPETF) decrease the adhesion between fiber–matrix interfaces [129,130]. Additionally, the surface and cross-sectional defects can offer hurdles to transferring of load [131]; see Figures 6–10 and Table 3. Therefore, these conditions decrease the performance and quality of developed composites [132]. On the other hand, the modulus of elasticity (Figure 11c) increases naturally with an increase in the amount of fiber in composite materials [133]. As fiber-reinforced composites are a combination of two unusual polymers, hence matrix and fiber phases help to resist environmental impacts and transfer of loads, respectively, see Figure 6.

The ability of composites to withstand impact loads is improved with a rise in fiber amounts, see Table 3. The improvement in impact energy for PP-PCPETF composites is observed more compared to PP-PCPESF composites.

According to dynamic mechanics, the stretching force propagates in composites through fibers from one matrix phase to another. The application of force appeared as deformation (change in length) in composite materials. Pure PP and composite materials (PP-PCCF, PP-PCPETF, and PP-PCPESF) consist of crystalline and amorphous phases, see Figure 6. The PP-PCCF, PP-PCPES, and PP-PCPET composites with 10% wt. show the highest level of crystallinity. Crystallinity is an indication of good adhesion between matrix and fiber phases. The crystalline phases resist the creation of deformation and ease the load-transferring phenomenon [134]. However, the increase in fiber content resists the transfer of load [135]. Therefore, all composites with 30 and 40% fiber loadings have the highest level of resistance toward load application. Additionally, the higher number of amorphous phases and lower level of crystallinity also enhanced brittleness and lowered ductility. Finally, the fracture appeared in the form of fibers pullout, see Figure 11d.

3.5.2. Flexural Testing and SEM Analysis of Fracture Surface

The results of flexural properties of PP-PCCF, PP-PCPETF, and PP-PCPES composites with different loading fiber loading are shown in Figure 12. According to Figure 12a and Table 3, the PP-PCCF 40% wt. composite material expresses the highest value of flexural strength of 57 MPa. Similarly, pure PP, PP-PCPESF 40% wt. and PP-PCPETF 40% wt. have a value of flexural strength of 42 MPa, 41 MPa, and 39 MPa. The higher value of flexural strength of PP-PCCF 40% wt. may be due to the nature of PCCF and PP material and the direction of application of force. However, in comparison, the PP-PCCF 30% wt. shows the lowest value of flexural strength of 35 MPa. Furthermore, composite materials with 30% fiber loadings (PCCF, PCPESF, and PCPETF) have complexity in mechanical behavior. That is why the other two, the PP-PCPESF 30% wt. and PP-PCPETF 30% wt. composite materials have values of flexural strength of 43 MPa and 40 MPa, respectively. Besides this, the PP-PCCF 10% wt., PP-PCPESF 10% wt., and PP-PCPETF composite materials show reasonable values of flexural strength of 51 MPa, 41 MPa, and 40 MPa, respectively.

Figure 12b and Table 3 represent the values of the flexural constant of various manufactured composite materials; the value of the flexural constant increases with an increase in the amount of fiber loadings. The addition of reinforced materials (PCCF, PCPESF, and PCPETF) forms special compounds with the PP matrix [136]. The composite materials with 30% fiber loadings present peculiar behavior due to the transformation of ductile to brittle behavior. According to Figure 12c and Table 3, the ductility was found to be higher for PP-PCCF 10% wt. and PP-PCPESF 10% wt. composite materials due to flexural strain values of 14 and 13, respectively. All other composite materials (30 and 40% wt. fiber loadings) show brittle fracture. Moreover, tensile investigations do not support the ductile behavior of composite materials with 30 and 40% wt. fiber loadings, see Figure 11a–d and Table 3.

Figure 12d expresses the representative SEM image of flexural failure of PP-PCPESF 40% wt. composite material. In this case, the failure occurs in compressive conditions due to reversion of direction of force, plastic deformation, fiber pullout, and hence fracture of composite material. Initially, at and above the yield point, the deformation of reinforced fibers becomes permanent. After that, at a point of flexural strength (Figure 12a and Table 3), the composite materials withstand the highest compressive strength, and fiber pullout comes into existence. The flexural constant (Figure 12b) helps to evaluate the failure mechanism (ductile or brittle) of composite materials. Finally, the fracture of composite materials occurs at a specific point.

3.5.3. Impact Testing and SEM Analysis of Fracture Surface

The results of the impact energy of all composite materials are shown in Figure 13a. The highest value was found for PP-PCCF 40% wt. composite material. In the comparison of the impact energy of developed composite materials, the PP-PCCF-10% wt., PP-PCCF 30% wt., and PP-PCCF 40% wt. composite materials show the highest values of impact energy of 4.8 kJ/m^2, 4.4 kJ/m^2, and 5.5 kJ/m^2, respectively. However, the values of impact energy of all composite materials with PCPESF reinforcement and 10, 30, and 40% variations were 2.80 kJ/m^2, 2.81 kJ/m^2, and 3 kJ/m^2, respectively. These values are lower than PCCF-reinforced-based composite materials, mainly due to the nature of fiber materials. The values of impact energy of PCPETF reinforced-based composite materials were intermediate between PCCF and PCPESF reinforced-based composites; see Figure 13a and Table 3. The sudden load transfer from fibers to the matrix phase causes fiber pullout, deformation, and fracture of the PP-PCPESF 40% wt. composite material collectively, as can be seen on the SEM micrograph, see Figure 13b.

3.6. Tribological Investigations and SEM Characterization of Composites

3.6.1. Abrasion Testing and SEM Analysis of Abrasive Surfaces

The results of abrasive wear rates of PP-PCCF, PP-PCPESF, and PP-PCPETF composites are shown in Figure 14a and Table 4. Pure PP offers maximum resistance towards cutting and shearing (abrasive wear of PP is 3.09×10^{-6} mm^3/Nm). Adhesion of PP particles, high rate of crystallinity (Figure 5c, Table 2, and Figure 6), and low surface roughness (Figures 7b and 10a) also enhanced the tribological properties. The addition of fibers (PCCF, PCPESF, and PCPETF) produced micro pits, line defects, and microcracks (especially in PP-PCCF/ PP-PCPESF/PP-PCPETF with 10 wt.% composites); see Figures 7c, 8b and 9b. These defects provide stress concentration sites for deformation creation [137]. Therefore, it increases the value of abrasive wear rates. The values of wear rates of PP-PCCF, PP-PCPESF, and PP-PCPETF (with 10 wt.% fiber loading) composite materials were 3.54×10^{-6} mm^3/Nm, 4.0×10^{-6} mm^3/Nm, and 6.21×10^{-6} mm^3/Nm, respectively. For further increase in the content of fibers, the surface defects (that appeared in PP-PCCF/PP-PCPESF/PP-PCPETF with 10 wt.% composites) transformed into surface asperities, high roughness areas and poor adhesion between matrix and fiber interface. The PP-PCPESF composites with 30 wt.% show abnormal behavior.

Table 4. Results of abrasive wear, erosive wear, and COF of natural and synthetic reinforced composite materials.

Composite Family	Abrasive Wear (mm^3/Nm)	Erosive Wear (mm^3/kg)	COF
Pure PP	$3.09 \times 10^{-6} \pm 0.10 \times 10^{-6}$	9 ± 6	0.70 ± 0.15
PP-PCCF 10% wt.	$3.54 \times 10^{-6} \pm 0.10 \times 10^{-6}$	19 ± 7	1.11 ± 0.20
PP-PCCF 30% wt.	$6.25 \times 10^{-6} \pm 0.10 \times 10^{-6}$	15 ± 7	1.23 ± 0.20
PP-PCCF 40% wt.	$6.39 \times 10^{-6} \pm 0.10 \times 10^{-6}$	35 ± 7	1.51 ± 0.20
PP-PCPESF 10% wt.	$4.03 \times 10^{-6} \pm 0.05 \times 10^{-6}$	2 ± 2	1.05 ± 0.10
PP-PCPESF 30% wt.	$3.54 \times 10^{-6} \pm 0.05 \times 10^{-6}$	7 ± 2	1.05 ± 0.10
PP-PCPESF 40% wt.	$4.13 \times 10^{-6} \pm 0.05 \times 10^{-6}$	8 ± 2	1.17 ± 0.10
PP-PCPETF 10% wt.	$6.21 \times 10^{-6} \pm 0.10 \times 10^{-6}$	3 ± 2.5	1,12 ± 0.15
PP-PCPETF 30% wt.	$6.40 \times 10^{-6} \pm 0.10 \times 10^{-6}$	3 ± 2.5	1.25 ± 0.15
PP-PCPETF 40% wt.	$6.21 \times 10^{-6} \pm 0.10 \times 10^{-6}$	2 ± 2.5	1.14 ± 0.15

On the other hand, the behavior of PP-PCPETF composites with 30 wt.% is found in accordance with other values. The values of wear rates of PP-PCCF, PP-PCPESF, and PP-PCPETF (with 30% wt. fiber loadings) composite materials were 6.39×10^{-6} mm^3/Nm,

3.54×10^{-6} mm^3/Nm, and 6.40×10^{-6} mm^3/Nm, respectively. The peculiar behavior of these composites is due to the transition from ductile to brittle behavior, see Figures 11–13 and Table 3. At the extreme level of fiber addition (PP-PCCF/PP-PCPESF/PP-PCPETF with 40 wt.% fiber loadings), the micro defects (mentioned above) become prominent on the surface of composite materials, see Figures 6, 7e, 8d, 9d and 10. The composites are transformed into brittle materials permanently because of increases in strength, hardness, and other mechanical properties (Figures 11–13 and Table 3). The wear rate values of such investigated PP-PCCF, PP-PCPESF, and PP-PCPETF composite materials with 40 wt.% fiber loadings were 6.39×10^{-6} mm^3/Nm, 4.13×10^{-6} mm^3/Nm, and 6.21×10^{-6} mm^3/Nm, respectively, see Table 4.

The interaction between PP-PCCF/PC-PCPESF/PCPCPETF composites materials and SiC P150 grade sandpaper expressed a large variation in the values of coefficient of friction, see Figure 14b and Table 4. The average COF value of PP was 0.70. The applied normal load helps to produce adhesion between the polymer and counter-metallic surface. The hard particles of SiC material interact with a polymeric surface. The degree of adhesion relies on the apparent conditions of PP-PCCF/PP-PCPESF/PCPCPETF composite materials. During the mechanism, the applied load is transformed into energy dissipation and shear phenomena. Initially, the average COF values of PP-PCCF, PP-PCPESF, and PCPCPETF composite materials (with 10% wt. fiber addition of PCCF, PCPESF, and PCPETF) were increased due to asperities, line-like micro-cracks, stress concentration sites and poor adhesion at matrix–fiber interface. The shear between interacting surfaces causes elastic and plastic deformation. The highest value of COF (1.51) was observed for PP-PCCF composites with 40% wt. fiber loading. At the climax, the shear, tear (cutting), and plowing engender fracture of the composite. In SEM analysis (Figure 14c), the fracture of pure PP (reference material) appeared in the form of abrasive wear. Formally, during sliding, the moving composite pins encounter static hard particles of SiC sandpaper. Adhesion comes into existence at the composite pin surface-SiC hard particle interface. The surface defects (Figures 6–10) act as a stress concentrator.

3.6.2. Erosion Testing and SEM Analysis of Erosive Surfaces

The results of erosive wear rates of all recycled composite materials are shown in Figure 15 and Table 4. The minimum erosive wear rates of PP-PCPESF-10% wt. and PP-PCPETF-40% wt. were found to be 2 mm^3/kg due to lower surface defects (Figures 7c and 10a), and the nature of materials (PP and PCPETF), respectively. The erosive wear rate values of 7 mm^3/kg and 8 mm^3/kg belonged to PP-PCPESF-30% wt. and PP-PCPESF-40% wt., respectively. The minor increase in values was due to enhancement in fiber addition and surface defects. According to Figure 15a, the highest values were measured for the PP-PCCF group of composite materials. The PP-PCCF-10% wt., PP-PCCF-30% wt., and PP-PCCF-40% wt. composite materials corresponded to erosive wear rates of 19 mm^3/kg, 15 mm^3/kg, and 35 mm^3/kg, respectively. Besides surface defects, the nature of sand particles and PCCF (lignin, hemicellulose, and microfibrils' individual effects) also have affected the COF values. However, the lowest values were observed for the PP-PCPETF group of composite materials. The PP-PCPETF-10% wt., PP-PCPETF-30% wt., and PP-PCPETF-40% wt. composite materials were related to wear rate values of 3 mm^3/kg, 3 mm^3/kg, and 2 mm^3/kg, respectively. The lowering in erosive wear rate values can be expected due to PP-PCPETF interfacial adhesion, the nature of polymer materials, and PCPETF flake structure. Briefly, the lowest, intermediate, and highest values of erosive wear are associated with PP-PCPETF, PP-PCPESF, and PP-PCCF types of composite materials.

Figure 15b shows the representative SEM micrograph of PP-PCPESF-40% wt. In the erosion mechanism, initially, the impact collision of sand particles produced deformation on the surface of composite materials. The hard sand particles caused the shear and cutting of composite materials. Finally, cutting of composite material appeared as weight loss and erosive wear. The cutting of composite materials relies on physical parameters (like force, angle of cutting, speed, temperature, etc.), the nature of hard materials, soft materials, and other environmental functions.

3.7. Technical Aspects of Circularity and Commercial Applications

Circularity is still a theoretical concept in nature. In our previous research, Hussain et al. [138] presented paradigms and technical strategies for the implementation of circularity in polymer composite industries. The decrease in quality and performance of polymer waste occurs due to extensive use during service life. During service life, polymeric materials face numerous chemical and physical treatments. In chemical treatments and interactions, polymeric materials react with chemicals and cause corrosion, fatigue, and other phenomena. Similarly, continuous, and periodic mechanical interactions produce slip and shear on the surface of polymeric materials. The shear process initiates plastic deformation, surface damage, distortion, and even fracture [138]. At the end of the service life of polymeric products, the performance and quality decreased. Physical assessment, mechanical testing, tribological investigation, SEM evaluations, and surface roughness measurements can help in the selection of a suitable recycling technique. After selection and initial physical testing, the PC waste of natural and synthetic polymers is cut and ground into fine fibers. Fine fibers, as a reinforced phase, impart mechanical properties to composite materials. PP PCCF, PCPESF, and PCPETF are commercial polymer materials. Similarly, injection molding is also an industrial processing technique. Therefore, the manufacturing of PP-PCCF, PP-PCPESF, and PP-PCPETF smart composite materials can be considered commercial for various applications.

The performance and quality of recycled composite materials are tested using various analytical techniques. The DSC and TGA confirmed that all recycled composite materials could withstand room and optimum higher temperatures during service life, see Figure 5a–d, respectively. A single-factor ANOVA was also conducted regarding melting point, crystallization temperature, degree of crystallinity, and degradation temperature. The results are shown in Table 5. The *p*-value and F-value of all composite materials confirm thermal stability. According to Figure 10, micro defects exist mostly on composite materials with 30 and 40% fiber loadings. According to Table 5, the average surface roughness, especially Ra, was significant with an F-value and a *p*-value of 0.77 and 0.644 (Table 5), respectively. The critical suitability of composite materials for commercial applications is analyzed using tensile testing. PP-PCCF-10% wt. and PP-PCPESF-10% wt. exhibit good flexibility (Figure 11c) (in terms of strain and hence deformation), stiffness (elastic modulus), and with standability at higher loads, see Figure 11b,c. Such types of smart composite materials can have potential applications in the automotive, civil, aerospace, and nuclear industries. The PP-based heavy and low-weight composite materials are used for shielding from gamma rays in the energy range of 59.5–1332.5 keV. The pure PP and composite mate can be mixed with heavy metals, polymer virgin, and recyclable materials [139–141]. The ductile to brittle transition of developed composite materials appeared at 30% fiber loading. However, the modulus of elasticity increased, and tensile strength showed fluctuations in the mentioned values. Therefore, the PP-PCCF-30% wt., PP-PCPESF-30% wt., and PP-PCPETF-30% composite materials are only suitable for static loads and environmental impact applications. The brittleness has become constant for PP-PCCF-40% wt., PP-PCPESF-40% wt., and PP-PCPETF-40% wt. composite materials. Such hard and stiff composite materials can be utilized for insulation, tableware, marine boats, electrical fittings, domestic appliances, and other products. The tensile strength, elastic modulus, and tensile strain yield an effect size of 89.6%, 0.2%, and 4×10^{-4}% with *p*-values of 0.896, 0.002, and 4×10^{-6}, respectively. However, F-values of composite materials for tensile strength, elastic modulus, and tensile strain behavior were 0.50, 3.14, and 6.51, respectively [142,143].

Table 5. Single-factor ANOVA test of thermal, surface roughness, tensile, bending, abrasion, and erosion properties of natural and synthetic reinforced composite materials.

Source of Variance	Composite Material's Behavior	Sum of Square (SS)	Degree of Freedom (df)	F Value	*p*-Value
Between groups	Melting point	170.79	9	0.004	1
Between groups	Crystallization temperature	986.67	9	0.060	0.999
Between groups	Degree of crystallinity	205.37	9	1.298	0.262
Between groups	Degradation temperature	3057.08	9	0.01	1
Between groups	Arithmetic average surface roughness value	34.70	9	0.770	0.644
Between groups	Tensile strength	330.15	9	0.50	0.869
Between groups	Modulus of elasticity	14,203,574.67	9	3.410	0.002
Between groups	Tensile strain	276.95	9	6.51	0.000004
Between groups	Flexural strength	1251.4	9	0.51	0.860
Between groups	Flexural constant	15,374,265.15	9	2.035	0.054
Between groups	Flexural strain	226.04	9	2.891	0.008
Between groups	Impact energy	20.545	9	0.823	0.60
Between groups	Abrasive wear	13.75	9	0.238	0.987
Between groups	COF	17.318	9	0.428	0.914
Between groups	Erosive wear	3733.504	9	11.80	1.07×10^{-9}

Besides tensile, bend testing is also considered important for further investigations and confirmation of commercial applications of composite materials, see Figure 12 and Table 3. In the reverse application of load (compressive), the values of flexural strength of all composite materials were found to be higher than tensile strength. Similarly, the values of the flexural constant of all composite family materials were also higher than the tensile modulus of elasticity. Higher strength and flexural constant are an indication of thin restoration and stiffness in flexed conditions. It was also noted that flexural deformation (flexural strain) of polymer composite materials was also higher than that of tensile strain.

Additionally, flexural strength, constant, and strain yield an effect size of 86%, 5%, and 0.80% with *p*-values of 0.86, 0.05, and 0.008, respectively. Similarly, F values of flexural strength, constant and strain behavior were 0.51, 2.035, and 2.891, respectively (Table 5). The flexural tests also confirmed the potential applications of recycled composite materials.

Sometimes, composite materials also face impact loads during service life during static or dynamic conditions. Therefore, impact tests measure the impact energy of composite materials for such conditions, see Figure 13a,b and Table 3. The impact energy of composite materials creates an effect size of 60% with a *p*-value of 0.60. Moreover, the F-value of impact energy behavior was 0.823. The impact test and ANOVA statistical analysis justified the potential use of fabricated composite materials for various applications, see Table 5.

The surface performance and quality of composite materials are subjected to tribological tests. Mostly, abrasive wear occurs between manufactured composite materials and hard particles of silica sandpaper. Besides surface defects, all composite materials show good abrasion resistance values in the range of 3.09×10^{-6} mm^3/Nm to 6.39×10^{-6} mm^3/Nm, see Figure 14a and Table 4. Therefore, it can face environmental impacts during service life. The environmental impacts can also appear in the form of fatigue, creep, corrosion, or erosion mechanisms. The statistical analysis was also performed with a 13.75 sum of squares and 9 degrees of freedom, see Table 5. The abrasive wear of composite materials creates an effect of 98.7% with a *p*-value of 0.987. Furthermore, the F-value of the abrasive behavior of composite materials was 0.238.

The interaction between the composite material and silica sandpaper also produces heat energy; see Figure 14b,c and Table 4. Amorphous surface, surface defects, and surface roughness reduce the adhesion between surfaces of composite materials and hard silica sand particles, see Figures 6–10. Heat energy enhances the temperature of composite materials and softens them. The higher values of COF are an indication of the production of heat energy. However, lower values of COF are a sign of good adhesion between two interacting bodies during various motions. According to ANOVA (Table 5), the COF values of composite materials yield an effect of 91.4% under the influence of a *p*-value of 0.914. Similarly, the F-value of the COF behavior of composite materials was 0.428. The surface quality can be increased using binders, mixers, heat treatments, and surface finishing techniques.

The quality of recycled composite materials against environmental impacts like water, humidity, elevated temperature, chemicals, and mechanical stresses is tested using erosion tests, see Table 4 and Figure 15b. The value of erosive wear of recycled composite materials varies from 9 mm^3/kg to 35 mm^3/kg. The excellent resistance to environmental impacts is due to the PP matrix material. PP imparts good temperature resistance, resistance to humidity, and other chemicals. According to statistical analysis (Table 5), the erosive wear yields an effect of 1.07×10^{-7}% with a *p*-value of 1.07×10^{-9}. Moreover, the composite materials impart erosive behavior with an F-value of 11.80. The results of our fabricated composites were also compared with the outcomes of other reinforced virgin composite materials. A reasonable match was found regarding tensile, bending, impact, and other properties [144].

4. Conclusions

In this study, PP-PCCF, PP-PCPESF, and PP-PCPETF post-consumer fiber-reinforced composite materials were fabricated using injection molding with 0, 10, 30, and 40% fiber loadings. The developed composite materials were found to be thermally stable. Subsequently, the surface, mechanical, and tribological properties are as follows:

- All composite materials with 10% wt. fiber loadings exhibit lower surface roughness values, smooth surface, and minimum micro defects. However, voids, pits, microcracks, and rough areas appeared on the surface of PP-PCCF, PP-PCPESF, and PP-PCPETF composite materials with 30 and 40% wt. fiber loadings. Moreover, Ra, Rq, Rp, Rz, and Rt surface roughness parameters were also higher;
- The tensile, impact, and flexural properties of produced composites are linearly related to the nature, amount, and size of fibers. PP-PCPESF 10% wt. shows the highest values of tensile strength (29 MPa) and strain (10%) with the reasonable value of modulus of elasticity (1401 MPa). Similarly, tensile strength, strain, modulus of elasticity, flexural strength, strain, impact energy, and flexural constant of all other composite materials were found reasonable for potential commercial application;
- Collectively, composite materials with 10% wt. fiber loadings show suitability for structural applications due to good ductility, plastic deformation, stiffness, and standability at higher loads. At 30% wt. fiber loadings ductile to brittle transition occur due to the complex behavior of composite materials. However, composite materials with 40% fiber loadings exhibit suitability for environmental applications due to higher brittleness and stiffness, and impact energy;
- The values of abrasive wear with values in the range of 3×10^{-6} mm^3/Nm to 6.5×10^{-6} mm^3/Nm have manifested very good surface quality of fabricated composite materials. Similarly, manufactured composite materials can also withstand environmental impacts due to minimum values of erosive wear in the range of 2 mm^3/kg to 35 mm^3/kg;
- The statistical ANOVA predicts the potential use of recycled composite materials in various structural and environmental applications.

Author Contributions: Designing, experimentation, composite development, TGA analysis, DSC analysis, tensile testing, bend testing, impact testing, abrasion and erosion testing, SEM analysis, review, and editing, complete experimentation, writing—original draft, validation. Moreover, CE industrial implementation review, open system to closed system transformation, sustainability check, ANOVA analysis for composites commercial applications A.H.; overall main supervision, review, and editing, validation, conceptualization of composites fabrication, V.P.; main supervision during cutting, grinding of fiber waste, size analysis of fiber reinforcement materials, D.G.; main supervision during tribological investigations, erosion, and abrasion testing, review, and editing, M.A.; review, editing and funding of manuscript, F.S.; supervision during composite fabrication and mechanical testing, review, and editing, I.K. All authors have read and agreed to the published version of the manuscript.

Funding: This research was supported by the Estonian Ministry of Research and Education, Tallinn University of Technology under project KIK 19019).

Institutional Review Board Statement: Not applicable.

Data Availability Statement: The raw/processed data required to reproduce these findings cannot be shared at this time as the data also form part of an ongoing study.

Acknowledgments: The authors gratefully acknowledge the financial support from the Estonian Research Council grants (PRG643), EIT Raw Materials ("RENEW" VHE22005), and the M-ERA.Net project ("DuplexCER" MNHA22040).

Conflicts of Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Publication V

Abrar Hussain, Podgursky V., Goljandin D., Antonov M., 2023. Industrial approach to circularity of polymer composites: Processing, characterization, mechanical testing and wear regression. Journal of Reinforced Plastics and Composites, 0 (0), 1-17, DOI: 10.1177/07316844231164563.

Original article

JOURNAL OF REINFORCED PLASTICS & COMPOSITES

Industrial approach to circularity of polymer composites: Processing, characterization, mechanical testing, and wear regression

Journal of Reinforced Plastics and Composites
2023, Vol. 0(0) 1–17

DOI: 10.1177/07316844231164563
journals.sagepub.com/home/jrp
SAGE

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Abstract

Cotton, polyester, and polyethylene terephthalate are the most common types of polymers that produce huge wastes. The circularity of these post-consumer (PC) waste faces operational problems during processing. In this innovative research, the relationship between circularity, surface characterization, mechanical and tribological testing of fiber reinforced (cotton, polyester), and particulate (polyethylene terephthalate) composites is explored for industrial pilot production. Cutting model can control the size of fibers during grinding. The fiber reinforced composites (FRCs) with 10% (by weight) fiber loadings are found as operational candidates for structural, automotive, and medical applications due to suitable tensile strength (26–29 MPa), percentage of extension (10%) and abrasive wear (3×10^{-6} mm^3/Nm). An increase in fiber content produces micro-defects like asperities, rough areas, voids, cracks, and pits in recycled composites. Therefore, the particulate and FRCs with 40% (by weight) fiber loadings become hard and brittle. However, these composites (especially with 40% wt. fiber loadings) exhibit reasonable elastic modulus (1526–2751 MPa) and abrasive wear (6.5×10^{-6} mm^3/Nm). The ductile to brittle transition effect has appeared in all composites (with 30% wt. fiber content) due to continuous fiber addition, micro-defects creation and dual phase presence. In conclusion, natural and synthetic PC wastes can be utilized for sustainable processing of commercial polymer composites. Moreover, injection molding, polymer characterization, tensile testing, abrasion evaluation, and regression analysis can be introduced for the transformation of open-loop into closed-loop manufacturing.

Keywords

polymer circularity, recycling, fiber reinforced composites, particulate composites, mechanical testing, polymer tribology, wear regression

Introduction

Circular economy (CE) is a set of technical strategies that creates a path for recycling of green and sustainable products. Recycling of polymer wastes mostly involves sorting, separation, grinding, processing, and finishing.[1] The utilization of polymers in medical, construction, military, smart textile, automobile, and electronic applications produces huge wastes.[2] These wastes are pre-consumer (defective pieces obtained during manufacturing), post-consumer (PC) (damaged products), end waste and end of life waste in nature that require versatile recycling techniques for processing.[3] Generally, open molding, closed molding, casting process and additive manufacturing are utilized to recycle wastes into reasonable composite materials.[4] Most precisely, pultrusion molding and resin transfer molding are used for the forming of thermosetting matrix composites. Moreover, injection molding and diaphragm processes are used for thermoplastic matrix composites. Composites are composed of matrix and fiber materials. Recycling helps to manufacture green products with minimal waste.[5]

The process of waste's conversion into product is known as recycling. Recycling starts with separation and sorting of natural and synthetic types of waste. After separation, polymer waste of high purity is transformed into fine fibers or powder using grinding.[5,6] On the other hand, in addition to grinding, impure waste is usually required secondary processing techniques to enhance the amount of valuable polymer.[7] Pure cotton (more than 25

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million tons), synthetic polyester (61 million metric tons) and polyethylene terephthalate (35 million metric tons) are commercial types of natural and man-made types of polymers.[8,9] Therefore, waste of these materials is available in abundance.[10] According to literature, these wastes are mostly present in PC wastes (with huge amount of valuable material).[11] Extrusion in association with injection, compression, pultrusion, and other cost-effective processes to recycle polymer products.[12–14] Besides this, solution treatment, pyrolysis, and other chemical processes are mostly used for removal of impurities from secondary, tertiary, and other hazardous polymer wastes. During and after recycling, quality control and assurance of recycled products is performed to check the performance for customer satisfaction. Finally, gel coating and painting are used to enhance the quality of composite surfaces.[15] A few articles have been reported regarding recycling of natural and synthetic polymeric composites. Mostly, sugarcane, cotton,[16] jute, palm, bamboo, flax, sisal, and kenaf are used as natural fiber reinforcement materials.[16–18] Natural fibers as reinforcement materials help in transferring loads and wettability during processing of composites.[19] High strength to low weight, flexibility, fracture toughness, elasticity, higher modulus or flexural constants, and resistance to environmental impacts are main features of composites.[20]

Mechanical, surface characterization, and tribological studies of natural and synthetic polymer hybrid composites are required during recycling due to their extensive use in the construction and automobile industries.[21] Tensile strength, modulus of elasticity, and flexibility assured the withstand ability, stiffness, and plasticity of polymer composites during service life.[22] On the other hand, mostly, oxides, carbides, nitrides, and diamond-like materials are used as counter bodies to investigate wear, coefficient of friction (COF), erosion and even deformation.[23] The COF values rely on the nature of composite material, fibers, nature of the counter body, metallic and polymer surface conditions, and other physical and environmental parameters.[24] However, the natural fibers as reinforcement materials provide different adhesion to the matrix phase. Therefore, formation of composites with different natural fibers furnishes different wear, abrasive, erosive and fatigue properties. Besides this, variations in physical parameters like force, temperature, time, and speed also influence the tribological properties of polymer composites. Experimentation and simulation work is usually introduced to optimize polymer products for commercial applications. Scanning electron microscopes (SEM) and regression are very common tools to analyze recycled polymer composites.[25]

In the present era, regressions are being used extensively to predict and optimize the responses in tribological investigations. Sardar et al.[26] modeled and optimized surface roughness, COF, and wear rate using integrated artificial neural network (ANN) and regression techniques. Similarly, Padhi and Satapathy[27] used multi-layered feed forward ANN to predict the tribological behavior of epoxy composites with short glass fibers (SGF) and micro-sized blast furnaces (BFS) particles. Egala et al.[28] employed ANN regression with single and multi-hidden layers to predict the tribological behavior of epoxy composites and concluded that ANN with multi-hidden layers predicted the tribological behavior of epoxy composites using regression in ANN with single and multi-hidden layers.[29] predicted the COF of treated bitternut fiber reinforced polyester (T-BFRP) composite using ANN on large data sets at load variation from 5 N to 30 N. The sliding distance was also varied from zero to 6.72 km for three different fiber material orientations. His findings showed that ANN predictions using Levenberg-Marquardt training functions were very close to the experimental results. Similarly, Nasir et al. used ANN to predict COF of polymeric composites with varying loads, sliding distance, and sliding speeds for large 7389 datasets. Their work showed that single layered models having many neurons using the Levenberg-Marquardt function provide high accuracy in prediction.[30]

This work reports the direct grinding, composite fabrication, comprehensive morphological analysis, tribological investigation, single-factor analysis of variance (ANOVA) and ANN regression for circularity of PC polymer waste. Initially, a free-body diagram and model were presented to control the grinding of PC natural and synthetic polymer waste. The developed prototype direct grinding machine was used to ground post-consumer cotton fibers (PCCF), post-consumer polyester fibers (PCPESF), and post-consumer polyethylene terephthalate fiber (PCPETF). The combination of extrusion and injection molding is used to develop polypropylene (PP)-PCCF, PP-PCPESF, and PP-PCPETF fiber reinforced composite materials. The fiber (PCCF, PCPESF, and PCPETF) loading of 0, 10, 30, and 40% is used to study the tensile, abrasive and surface properties of recycled composites. The service life, useability and suitability of fabricated composite materials is analyzed using the American Society of Testing of Materials (ASTM) D3039 tensile test. Abrasive wear and COF of composite materials are investigated using the ASTM G132-96 abrasion test. Moreover, ANN regression has been employed to predict and optimize COF at a force of 1 N. All PC waste and fabricated composites are characterized using a SEM before and after mechanical testing to observe the micro-defects and other mechanical phenomena.

Materials and methods

Materials

Pure PP was used as a matrix material. PP powder was supplied by Egyeuroptene with melt flow index

(MFI) = 14 g/10 min @ 2.16 kg, temperature 210°C, 1.50 g/mL density (ASTM D 1238). The PP matrix phase binds fibers together, provides shield for fiber damage, responds at low processing temperature, and furnishes reasonable fiber wettability.

Pure cotton for T-shirts in PC form was supplied by the Estonian army and used as a reinforced material. The mechanical properties of the PCCF were 1.55 g/cm^3 density, 5–25 ± 1.5% elongation range, 0.06 ± 0.001 MPa, tensile strength, 0.10 ± 0.004 MPa effective tensile strength, 0.045 ± 0.002 MPa breaking strength, and 237 g per square meter g/m^2 (GSM) weight.[31]

Synthetic polyester (Spun polyester) for T-shirts in PC form was supplied by local industry. The PCPESF is belonged to PCDT (poly-1.4 cyclohexyl-dimethylene-terephthalate) group of polyester. The mechanical properties of PCPESF were: 5–7 ± 0.01 g/den tenacity, 15–30% ± 1.7 elongation, 0.40 ± 0.001% moisture regain, 1.38 specific gravity, 170°C melting temperature, and 0.07 ± 0.001 MPa elastic modulus. Physically, PCPESF has a light green color, bright luster, good reflection ability, and minimal swelling ability.

PCPETF was derived from beverage bottles. The mechanical properties of PCPETF were 1.40 g/cm^3 density, 460 ± 80 MPa tensile strength, 15–20 ± 2% strain, and 0.20 ± 0.001 GPa modulus of elasticity.

Methods

Sorting, separation, and grinding. The sorting and separation of waste was done manually at laboratory scale for pilot production. The grinding model and free-body diagrams are presented to control the size of fibers. The fabric cotton, synthetic polyester and polyethylene terephthalate sheets were cut into small pieces (no longer than 5 cm), grinded and milled using a developed prototype grinding machine at a speed of 300 revolutions per minute (rpm) for 10 min. The direct grinding disintegration machine transforms PC waste polymers into PCCF, PCPESF and PCPETF fine fibers. The length and diameter of disintegrated fibers were calculated using SEM. These steps were performed at a temperature of 21°C. The fabric material, especially cotton, was dried for 4 h at a temperature of 60°C.

Fabrication of composites materials. Initially, the PP and waste fibers (PCCF, PCPESF, and PCPETF) were mixed with a cylindrical mixer for 15 min at a speed of 80 r/min. The formulation of composite fabrication was shown in Table 1. The volume of mixing chamber was 250 cm^3. After that, the melt-mixing of waste fibers and PP is done using a plastic corder compounder. Extrusion process was utilized for compounding. The temperature, time, torque, and speed were kept at 190°C, 7 min, 60 Nm. and 80 r/min for composite formation. The chamber was filled with weighted PP and waste polymer materials using a metallic funnel tube. The melt materials (PP and waste fibers) were passed through different melting zones operating in the range of 120°C–200°C. The mixtures of PP and waste fibers were mixed for 7 min and extruded out in the form of wires. The gained product was grinded and milled into fine grains and beads. The fine grains and particles were transformed into 4 mm sheets using injection molding. The heating time, pressure and temperature were kept 7 min, 80 kg cm^{-2} and 190°C, respectively. The compressed sheets were annealed from 190°C to room temperature for 5 min.

SEM analysis. Surface morphology was investigated by SEM (Zeiss EVO® MA-15 system, Oberkochen, Germany) with LaB6 cathode in the secondary electron mode, applying an accelerating voltage of 10–15 kV at 6.5–8.5 mm working distance. Moreover, the SEM analysis of composites was done before and after mechanical testing.

Mechanical testing for commercial applications. A Universal Testing Machine (Model 5820, Instron Co.) was used to evaluate mechanical properties, namely tensile strength, breaking strength, elastic modulus, and extension at maximum load. The testing was performed according to ASTM D3039 tensile testing and standards. The specimen's size was 4 (thickness) × 25.4 (width) × 150 mm (length). The gauge length was kept at 100 mm. The crosshead speed was 50 mm/min. The force-extension and stress-strain graphs were recorded using data collection and acquisition software.

Tribological testing. The sliding testing was performed to determine COF using the ASTM G132-96 standard. Zirconia sandpaper was used as counter body. The composites were cut into 5 mm × 5 mm × 15 mm small pins. The CETR UMT-2 tribometer was introduced for sliding (Figure 1(a) and (b)). The speed, load, and sliding track distance were 0.1 m s^{-1}, 1 N and 70 mm, respectively. The composite pins covered 18 m of a linear abrasive path in 3 min. The COF tests were executed to evaluate minimum, average, and maximum values for designing considerations. The tests were performed at room temperature at a relative humidity of 60%.

The abrasive wear was (W) calculated using the following formula:

$$W = \frac{V}{L \times S} \tag{1}$$

where V is the volumetric wear loss (mm3), L is the normal load (N) applied during the test and S is the total sliding distance (m).

Prediction of COF through neural network. The data obtained from a tribo-tester for different material compositions on different forces was predicted through ANN regression. During the pre-processing step, data was cleaned to detect

Table 1. Formulation scheme of fiber reinforced (PP-PCCF and PP-PCPESF) and particulate composites (PP-PCPETF).

Fiber content (wt%)	Matrix content (wt%)	Net weight of fibers (g)	Net weight of matrix (g)
0.0	100	0.0	300
10	90	30	270
30	70	90	210
40	60	120	180

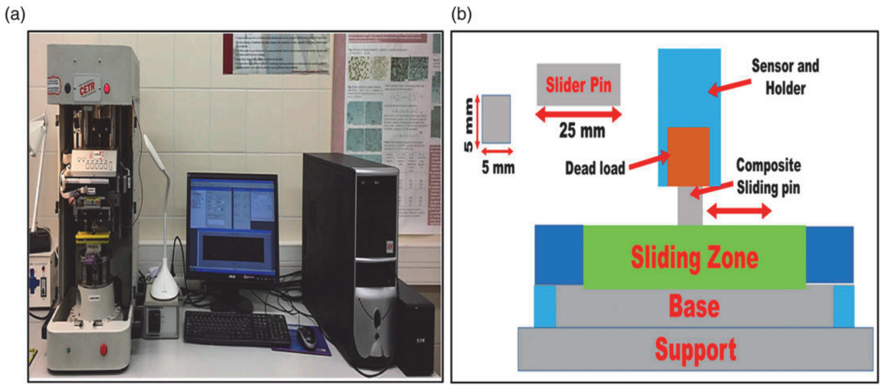

Figure 1. Abrasion testing: (a)experimental setup and (b) working principle.

and remove outliers. The removed values were filled at the nearest data points. The cleaned data of different materials composition was predicted at the forces of 0.3, 1 and 3 N. Data was divided in to 70%, 15% and 15% for training, validation, and testing. Levenberg-Marquardt optimization was employed using a back propagation training function trainlm. The network was trained and tested with several neurons ranging from 7 to 20 neurons.

Results and discussion

Grinding optimization

The rotational, parallel, and perpendicular principles of cutting include normal force during cutting. The cutting is controlled with the help of tribological friction.

The friction relies on the nature of the cutting and materials.[32] At first, the elastic deformation was produced during cutting tool edge and cotton polymer surface interaction.[33]

Generally, the simple mathematical model assists in predicting and understanding the cutting process. The cutting process can be described in terms of cutting resistance. The cutting resistance is associated with deformation and wear phenomena. The cutting tool transfers energy for creation of wear.[34] The frictional force acts as mediator for the transformation of energy into deformation. Figure 2(a) and (b) show the diagram for the cutting process.

The normal force (F_N), sliding speed (V), friction force (F_{friction}), separation force (F_s), thickness (t, mm), and separation distance (X, mm) are used to abbreviate various essential operational factors. The F_N, $F_{\text{Horizontal}}$ and $F_{\text{Perpendicular}}$ represent the three-dimensional vectors (horizontal (i), perpendicular (j), normal (k) mechanical transverse and normal forces. The traditional vector product for mechanical transverse forces can be expressed as

$$F_{Horizontal}*F_{Perpendicular} = |F_{Horizontal}|\,|F_{Perpendicular}|sin(\theta) \tag{2}$$

$$F_{Horizontal} = F_N \text{ and } F_{Perpendicular} = F_{friction} \tag{3}$$

The normal force causes the friction between the tool tip and the polymer surface. Initially, the cutting tool's hard surface compresses the soft polymer surface. The sliding produces deformation due to the shear process.

The initial area of contact for the first slide is abbreviated as

$$A_{\text{contact}} = (l_{contact} * t_{\text{blade}}) \tag{4}$$

where A_{contact}, l_{contact}, and t_{blade} parameters are apparent contact area of blade, length of blade contact, and thickness of blade cut.

The vertical area of contact can be calculated as

$$A_{\text{vertical}} = (l*w) \tag{5}$$

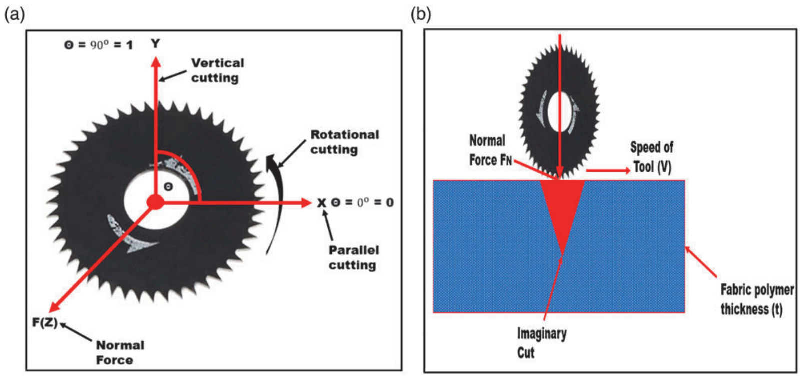

Figure 2. Cutting model: (a) free-body diagram for cutting and (b) cutting mechanism.

Where A_{vertical}, l, and w parameters are vertical area, length of cut, and width of cut.

However, the actual area of cut can be estimated as

$$A_{\text{actual}} = (l_{\text{actual}} * w_{\text{actual}}) \tag{6}$$

where A_{actual}, l_{actual}, and w_{actual} parameters are actual area, actual length of cut, and actual width of cut.

Theoretically, the friction force causes sliding due to the combined effect of shear and plowing. Therefore, mathematically:

$$F_{friction} = S + P \tag{7}$$

$$S = (\sigma_s * A_{contact}), P = (\sigma_p * A_{vertical}) \tag{8}$$

where σ_s and σ_P are shear and plowing stresses:

$$F_{friction} = (\sigma_s * A_{contact}) + (\sigma_p * A_{vertical}) \tag{9}$$

$$F_{friction} = \mu F_N \tag{10}$$

$$F_N = \sigma_P \, \text{x} \, A_{vertical} \tag{11}$$

$$\mu \, x \, \sigma_P x \, A_{vertical} = \sigma_s \text{x A contact} + \sigma_P \, \text{x} \, A_{vertical} \tag{12}$$

The wear in terms of volume loss can be calculated as

$$Q = (length\ of\ cut)\, x\, (depth\ of\ cut)\, x\, (width\ of\ cut) \tag{13}$$

Developed models and free-body diagrams have important applications in the polymer industry. Various tribological systems like cotton steel, cotton-TiAlN, cotton-TiCN, cotton-WC, cotton-Al_2O_3, and cotton-Zr_2O_3 for polymer-metallic interactions have been reported by our group.[35,36] The experimental applications are mentioned in Table 2. Outcomes of these tribological investigations play an important role in different steps of recycling, like automatic separation, grinding, processing, and finishing. However, we have considered the outcomes of COF investigations for fiber surface (Figure 3(a)–(c)) and size optimization (Table 3). Various outputs like deformation, change in COF values, abrasive wear, and grip (between polymer waste and metallic machinery parts) address different phenomena like buckling, metallic surface fatigue, corrosion, tangling during different recycling operations. In this manuscript, only the effect of surface damage and distortion due to waste-metallic interaction is considered. The results of SEM analysis and the size of fine fibers (PCCF, PCPESF, and PCPETF) are tried to evaluate in Figure 3(a)–(c) and Table 3, respectively.

SEM characterization of PC wastes

The surface characterization of PC waste was done using SEM. The matrix and fiber phases were studied before and after testing. The PCCF, PCPESF and PCPETF were found irregular and intermittent, seeFigure 4(a)–(c).

The fiber length, diameter and area were shown in Table 3. Most precisely, the fiber is composed of microfibrils. The microfibrils join the core hemicellulose through lignin. This type of fibrous structure causes fibers to absorb humidity and other impurities naturally, see Figure 3(a). This effect reduces fiber strength. Figure 3(b) belongs to PCPESF. Synthetic fibers possess damaged surface and microfibrils. These fibers show the same response as that of PC-PCCF regarding grinding, mixing, and processing. Optimum size of PCCF and PCPESF assists in transferring load from one phase of matrix to another.[37] Besides this, the variation in size of fibers contributes to elasticity, plastic deformation, high tensile strength, resistance towards fracture, flexural properties, impact strength and ductile to brittle transition. PCPETF is used to fabricate particulate composites due to the small size of ground fibers (or particles), see Figure 3(c). PCPETF produces specific composite phases (when mixed with PP) which can withstand

Table 2. Model and free-body diagram experimental applications.

Tribological system	Average COF values	Output of analysis
Cotton-steel	0.20	Provide goop grip for processing and deformation.
Cotton-TiAlN	0.37	Produce plastic deformation, wear, and local material removal
Cotton-TiCN	0.35	Produce abrasive wear and plastic deformation
Cotton-Al_2O_3	0.15	Introduce good grip and surface finishing
Cotton-WC	0.10	Produce no surface wear or deformation
Cotton-Zr_2O_3	0.08	Manifest good surface finishing

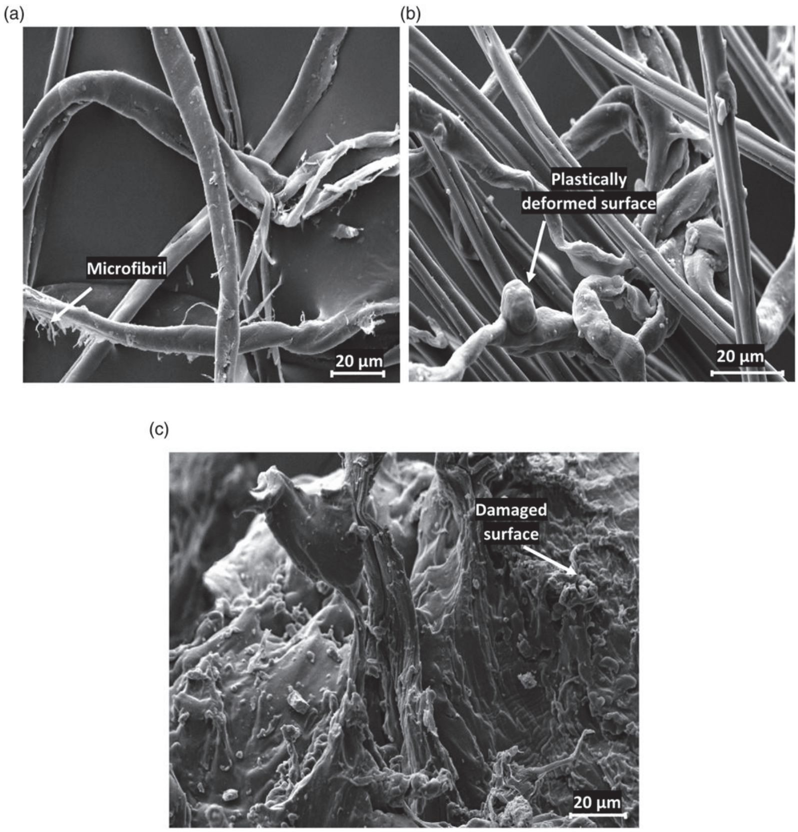

Figure 3. SEM characterization of polymer wastes fibers: (a) PCCF, (b)PCPESF and (c) PCPETF.

environmental impacts, sudden loads, and higher strengths. However, the smaller size of fibers makes composites brittle. The successive sequence of grinding made the surface of PCCF, PCPESF and PCPETF rough.[36]

Table 3 expresses very important information. The average length, diameter, and area of PCCF and PCPESF are almost the same. The longer fiber size of PCCF and PCPESF provide good matrix-fiber interfacial adhesion (see special Figure 5(b) and (e)) and plastic deformation in terms of elongation (especially composite materials PP-PCCF 10% wt. and PP-PCPESF 10% wt. in Table 4). Moreover, higher content of fibers (PCCF and PCPESF) produced defects in composite materials (Figure 5(b)–(j) and Figure 6(a)–(c)) due to poor adhesion. However, the PCPETF only forms special compounds of PP-PCPETS. The smaller size of PCPETF lowered tensile strength and

Table 3. Average length, diameter, and area of PCCF, PCPESF, and PCPETF waste materials.

	Length (mm)			Diameter (μm)			Area (μm²)		
Fiber nature	Minimum	Average	Maximum	Minimum	Average	Maximum	Minimum	Average	Maximum
PCCF	1.5 ± 0.001	3 ± 0.001	10 ± 0.001	16.87 ± 0.002	17.87 ± 0.002	20.55 ± 0.002	106 ± 0.004	250 ± 0.004	350 ± 0.004
PCPESF	2 ± 0.001	3.5 ± 0.001	9 ± 0.001	15.31 ± 0.002	17.50 ± 0.002	21.15 ± 0.002	120 ± 0.004	245.21 ± 0.004	323 ± 0.004
PCPETF	0.08 ± 0.0002	0.31 ± 0.0002	0.51 ± 0.0002	0.01 ± 0.0001	0.01 ± 0.0002	0.02 ± 0.0002	0.15 ± 0.0004	0.19 ± 0.0004	.30 ± 0.0004

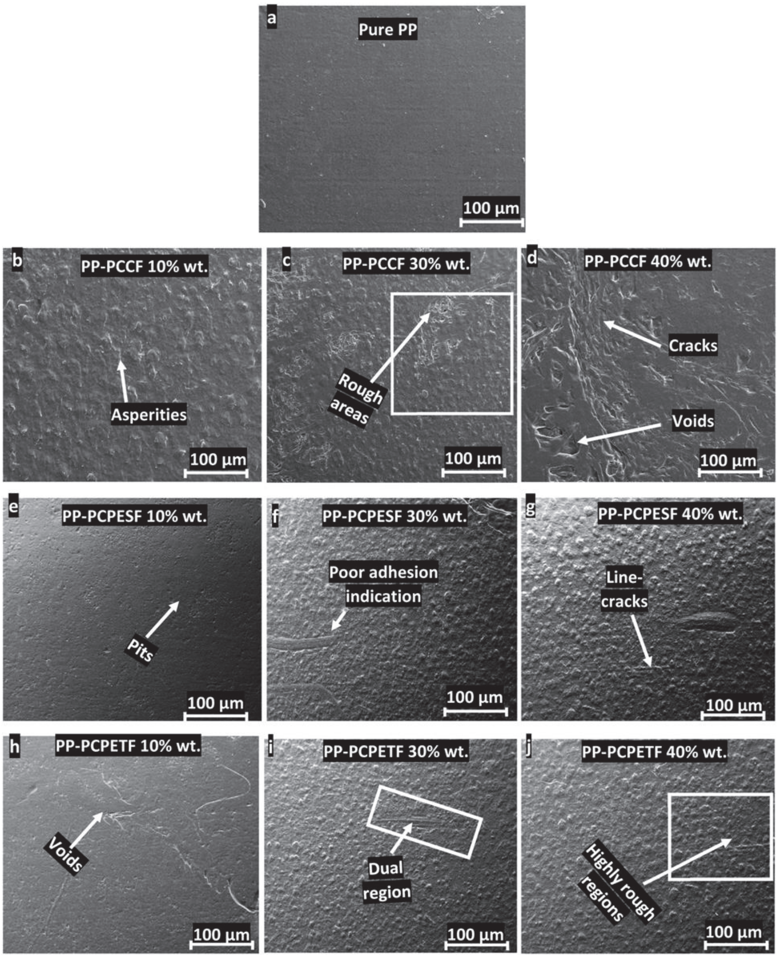

Figure 4. SEM characterization of PP and fabricated composites: (a) pure PP, (b) PP-PCCF 10% wt., (c) PP-PCCF 30% wt., (d) PP-PCCF 40% wt., (e) PP-PCPESF 10% wt., (f) PP-PCPESF 30% wt., (g) PP-PCPESF 40% wt., (h) PP-PCPETF 10% wt. (i) PP-PCPETF 30% wt., and (j) PP-PCPETF 40% wt.

plastic deformation (all PP-PCPETF composite groups of material with 10, 30, and 40 of PCPETF content in Table 4). Moreover, it also produced various types of surface defects (Figure 5(h)–(j)). According to abrasion investigation, all types of developed composite materials show good abrasive wear resistance (Figure 7(d)–(f)) against zirconia sandpaper.

Systematically, the SEM micrographs of polymer composites (before testing) were shown from Figure 4(a)–(j). Figure 4(a) represents a SEM micrograph of pure PP. Factors like the same size of PP powder particles, automatic control of temperature, pressure and compaction provide uniform surface and minimum production of defects.[38] Addition of PCCF as a reinforcement (of course impurity) engendered thermal and mechanical strains, see Figure 4(b) of PP-PCCF 10% wt. composite. These strains appeared as asperities on PP-PCCF 10% wt. composites surface. Further increase in PCCF contents causes the creation of rough surface areas. These rough areas transform the behavior of PP-PCCF 30% wt. composites from ductile to brittle. The results are depicted in Figure 4(c). Addition of PCCF is directly proportional to the strength and hardness of composites. However, higher amounts of PCCF cause the creation of cracks and voids. Therefore, the composites become permanently brittle, see Figure 4(d).

Figure 4(e)–(g) presents the SEM micrographs of PP-PCPESF composites. Mixing and homogeneity mostly rely

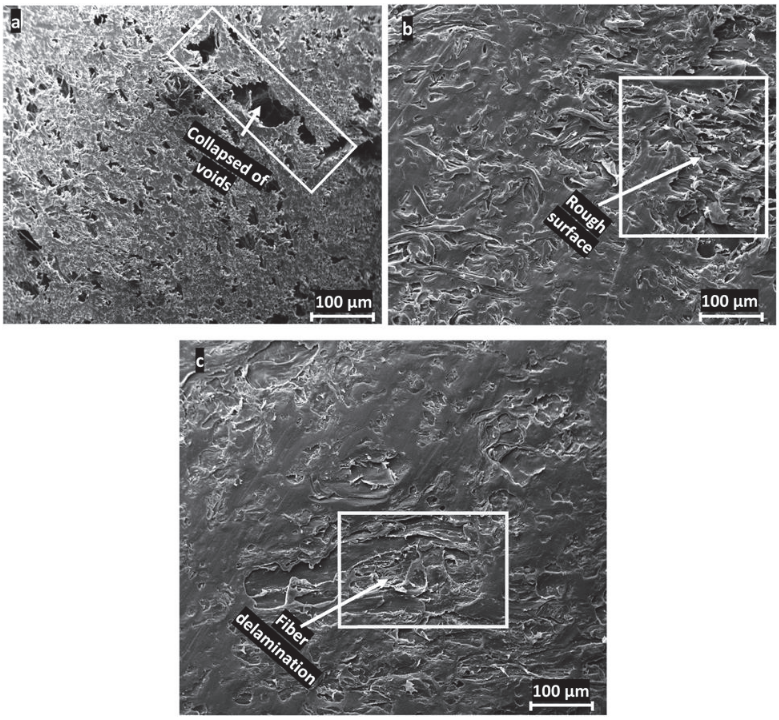

Figure 5. Cross-sectional SEM characterization of developed composites: (a) PP-PCCF 40% wt., (b) PP-PCPESF 40% wt., and (c) PP-PCPETF 40% wt.

Table 4. Tensile properties of natural and synthetic developed composites.

Composite family	Tensile strength (MPa)	Elastic modulus (MPa)	Percentage of elongation (%)
Pure PP	31 ± 5	1226 ± 90	10 ± 2
PP-PCCF 10% wt.	26 ± 5	1476 ± 80	8 ± 1.5
PP-PCCF 30% wt.	24 ± 6	2521 ± 102	4 ± 0.75
PP-PCCF 40% wt.	22 ± 5	2751 ± 150	3 ± 0.5
PP-PCPESF 10% wt.	29 ± 6	1401 ± 70	10 ± 2.5
PP-PCPESF 30% wt.	25 ± 5	1526 ± 85	5 ± 0.5
PP-PCPESF 40% wt.	23 ± 5	1591 ± 110	4 ± 0.25
PP-PCPETF 10% wt.	26 ± 5	1167 ± 85	5 ± 0.75
PP-PCPETF 30% wt.	22 ± 3	1117 ± 70	4 ± 0.50
PP-PCPETF 40% wt.	22 ± 3	790 ± 110	3 ± 0.25

on polymer material nature. Therefore, pits appear on the surface of PP-PCPESF 10% wt. composite materials. From a mechanical point of view, these micro pits act as stress concentrators, see Figure 4(e). PP-PCPESF composites 30% wt. fiber loading show lower mixing and homogeneity as compared to PP-PCCF composites materials with the same compositions.

PCPESF, pits and asperities appeared on the PP-PCPESF (30% wt.) composite's surface as an indication of poor adhesion. Line cracks appeared on the surface of PP-PCPESF composites with a 40% wt. fiber amount. Besides this, asperities were also prominent on the surface (Figure 4(g)). Such defects suggest that these composites are not suitable for structural applications.

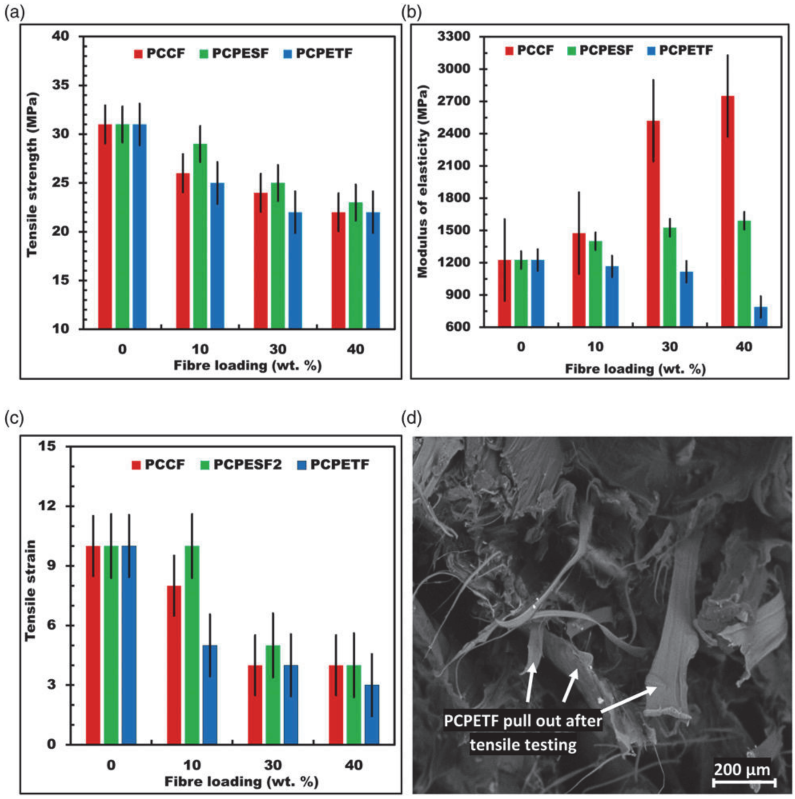

Figure 6. Comparative tensile properties demonstration of developed composite materials: (a) Tensile strength, (b) Modulus of elasticity, (c) Tensile strain, and (d) PCPESF pullout after tensile test.

PP-PCPETF particulate composites belong to Figure 4(h)–(j). These composites are designed specially to withstand impact loads. The microstructure of PP-PCPETF 10% wt. composite shows different behavior (Figure 4(h)).

Collectively, line cracks, pits, voids, and rough areas appeared on PP-PCPETF 10% wt. composites. Some smooth regions can also be seen in the shown microstructure. Surface defects are signs of purely brittle materials. PCPETF Fibers or particles do not participate in the transfer of load. However, PCPETF fibers combined with PP and made new phases. These phases resist abrasion, erosion, fatigue, impact, and point loads. Figure 4(i) corresponds to PP-PCPETF 30% wt. An increase in PP-PCPETF vanished the uniform regions. Asperities and rough areas replaced the uniform regions. In some regions, uniform and rough regions appeared together.

These are termed as dual phases. Dual phases push PP-PCPETF 30% wt. composites to show complex behavior. However, an increase in PCPETF (40% wt.) transformed composites into permanent brittleness, see Figure 4(j).

The fabricated composites were cut cross-sectionally to study the internal structure. Evaluation results are shown in Figure 5(a)–(c). Figure 5(a) shows fibrous structure, cotton fibers, matrix, holes, voids, and collapse of porosity. Figure 5(b) (PP-PCPESF 40% wt.) shows lower surface defects and more homogeneity (when compared with Figure 6(a)). However, it shows the asperities fiber pull out and micro holes. Similarly, Figure 5(c) expresses the microstructure of PP-PCPETF. Besides other defects, it shows the PCPETF delamination. These defects arise due to poor bonding, humidity removal and poor adhesion. Moreover, porosity, voids, asperities, debris, and other defects could have special roles in thermal, acoustic and construction applications.[39]

Tensile testing of developed composites

The results of tensile testing of PP-PCCF, PP-PCPESF, and PP-PCPETF of composite materials along with 0, 10, 30, and 40% fiber variations are shown in Table 4. Tensile

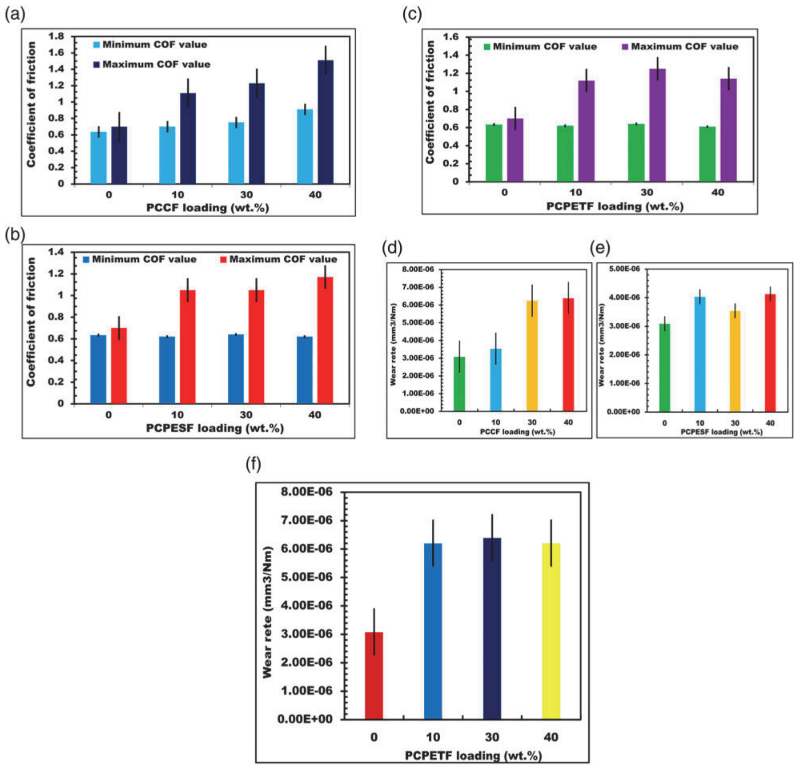

Figure 7. Representative COF and abrasive wear graphs of recycled composites: (a) COF graph of PP-PCCF composites, (b) COF graph of PP-PCPESF composites, (c) COF graph of PP-PCPETF composites, (d) abrasive wear graph of PP-PCCF composites, (e) abrasive wear graph of PP-PCPESF composites and (f) abrasive wear graph of PP-PCPETF composites.

strength is the maximum strength that a composite can withstand during dynamic or static conditions. Pure PP exhibits a maximum tensile strength of 31 MPa. The higher strength of pure PP may be due to uniform structure, see Figure 4(a). The uniform structure of pure PP appeared due to good adhesion between polymer particles and minimal creation of defects. In composite materials with 10% wt. fiber (PCCF, PCPESF, and PCPETF) loadings, PP-PCPESF 10% wt. shows a higher value of tensile strength of 29 MPa. The PP-PCCF 10% wt. and PP-PCPETF 10% wt. composite materials have a tensile strength of 26 MPa. The variation in values of tensile strength exists due to the different nature of defects.[39] For instance, Figure 4(b), (e) and (h) express asperities, pits, and voids, respectively. Therefore, the disparate creation of defects lowered the quality of composite materials. Similarly, PP-PCCF 30% wt., PP-PCPESF 30% wt., and PP-PCPETF 30% wt. composite materials have tensile values of 24 MPa, 25 MPa, and 22 MPa. Figure 4(c), (f) and (i) are related to rough areas, poor adhesion, dual regions and mentioned related values of tensile strength, respectively. At the highest level of fiber loading, the PP-PCCF 40% wt., PP-PCPESF 40% wt., and PP-PCPETF 40% wt. composite materials manifest tensile strength of 22 MPa, 23 MPa, and 23 MPa, respectively. Qualitatively, these values are related to Figure 4(d), (g) and (j). The marginal decrease in tensile strength may be due to the presence of cracks, highly rough regions, collapse of voids and higher values of fiber loadings.[40]

Fiber addition is directly proportional to an increase in the value of the modulus of elasticity. Therefore, the value of the modulus of elasticity rises gradually. Pure PP as a reference material has a value of modulus of elasticity 1226 MPa. Systematically, the values of modulus of elasticity of PP-PCCF 10% wt., PP-PCPESF 10% wt., and PP-PCPETF 10% wt. composite materials are 1476 MPa, 1401 MPa, and 1176 MPa, respectively. The marginal decrease in value of stiffness of PP-PCPETF is due to a smaller size of fiber, shape (flake), and defects, see Table 4, Figures 3(c) and 6(b). The PCPETF resists the transfer of load. Similarly, the value of modulus of elasticity of PP-PCCF 30% wt., PP-PCPESF 30% wt., and PP-PCPETF 30% wt. composite materials are 2521 MPa, 1526 MPa, and

1127 MPa. In this case, higher values of PCCF based composite materials are due to ductile to brittle transition, nature of fiber material, and degree of cross-sectional defects (Figure 5(a)). That is why PP-PCCF 40% wt. expresses the highest value of modulus of elasticity of 2751 MPa. Besides this, the values of the modulus of elasticity of PP-PCPESF 40% wt. and PP-PCPETF 40% wt. are 1591 MPa and 790 MPa. The lowest value of modulus of elasticity of PP-PCPETF 40% wt. in the composite family may be due to smaller fiber length (Figure 3(c)), delamination of fibers (Figure 5(c)) and poor adhesion between the PP and PCPETF interface.

Plastic deformation in terms of tensile strain plays vital role during design of materials for different commercial applications. The tensile strain fundamentally relies on length of fiber, nature of matrix and reinforced materials. The highest value of tensile strain of pure PP has PP-PCPESF composite materials is 10. The highest values of tensile strain of materials are due to equal particle size and higher fiber length (Figures 3(b) and 6(c), and Table 3) of PP and PCPESF reinforced. Additionally, the values of tensile strain of PP-PCCF 10% wt. and PP-PCPETF 10% wt. composite materials are 8 and 5, respectively. According to Table 4, and Figure 6(c), conventionally, increase in fiber (PCCF, PCPESF, and PCPETF) loading causes the decrease in plastic deformation (strain). Therefore, the values of tensile strain of PP-PCCF 30% wt., PP-PCPESF 30% wt., and PP-PCPETF 30% wt. composite materials are 4, 5, and 4, respectively. This phenomenon can also be correlated to microstructural defects like poor adhesion (Figure 4(f)) and rough regions. At 40% wt. of fiber loading, all composite materials become brittle due to marginal decrease in the values of tensile strain. Higher values of fiber materials constrained the transfer of tensile load. The resistance in transfer of load also pulls out the fibers from composite materials, see Figure 6(d). That is why, the lowest value of tensile strain of 3 was measured for the PP-PCCF 40% wt. and PP-PCPETF 40% wt. composite materials. The PP-PCPESF 40% wt. composite material shows better quality with tensile strain value of 4 due to better quality of PCPESF (Figure 3(b), Table 3, Figure 6(c), and Table 4, respectively). All composites (with 30% wt. PCCF, PCPESF, and PCPETF fiber loadings) exhibit complex behavior. This behavior can be termed as ductile to brittle transition. Tribological investigations are introduced to evaluate the behavior of composite materials against environmental impacts.

A one-way factorial ANOVA was conducted to compare the main effects of tensile strength, modulus of elasticity, and tensile strain. The elastic modulus and tensile strain effects were significant at a p value <0.05. According to Table 5, tensile strain and elastic modulus yield an effect size of 4.48×10^{-4} and 2%, respectively. However, tensile strength yields an effect size of 86.9% at a standard p-value of <0.05.

Abrasion testing of developed composite materials

The representative COF graphs of composites were shown in Figure 7(a)–(c). The COF varies with the nature of the fiber's material variation. According to Figure 7(a)–(c), the COF value increased from 0.65 to 1.65. The pure PP and composites with 10% cotton show minimum COF value against zirconia sandpaper. However, composites with 30% and 40% fibers show higher COF values. The highest value was recorded for PP-PCCF with 40% fiber loading. It varied from 1.50 to 1.65 (see Figure 7(a)). The reason for the COF increment is two-fold. Firstly, the increase in fiber addition enhances the degree of micro-defects, see Figures 4(a)–(j) and 5(a)–(c). During sliding, micro-defects interact with zirconia sandpaper particles. These interactions create resistance in adhesion between zirconia sandpaper and polymer composite surfaces.[41]

This resistance softens the polymer composites due to heat dissipation. Therefore, it increases the COF values. After initial adhesion and softening of the composite's surface, the hard particles overcome the binding energy and poor physical adhesion between the matrix-fiber interface. It starts with the phenomenon of slippage on composite surfaces. Slippage is transformed into regular shear due to an increase in the number of sliding cycles. Shear produced plowing and cutting. Cutting causes the composite's material removal from the surface (abrasive wear).[42] The typical graphs of abrasive wear of PP-PCCF, PP-PCPESF, and PP-PCPETF composites materials are shown in Figure 7(d)–(f). Pure PP expresses minimum abrasive wear due to good crystallinity, tensile strength, and flexibility in nature. According to Figures 4(a)–(j) and 5(a)–(c), asperities, rough areas, voids, cracks, and pits act as stress concentration sites and deformation strains. These deformation strains and stress concentration sites are enlarged with a rise in fiber loadings. Hence, the value of abrasive wear rises, see Figure 7(d)–(f). According to graphs, the minimum value of abrasive wear of PP was 3×10^{-6} mm^3/Nm

Table 5. Single-factor ANOVA test of tensile strength, elastic modulus, and tensile strain.

Source of variance	Tensile behavior	Sum of square(SS)	Degree of freedom(df)	F value	p-value
Between group	Tensile strength	330.15	9	0.498	0.869
Between group	Elastic modulus	14,203,574.67	9	3.408	0.002
Between group	Tensile strain	276.94	9	6.51	4.48×10^{-6}

(Figure 7(d)). Figure 7(f) expressed maximum value of abrasive wear of 6.5 × 10^{-6} mm^3/Nm for PP-PCPETF composites with 30% wt. fiber loading. However, all composites' materials with 30% wt. of fibers (PCCF, PCPESF, and PCPETF) loadings expressed complex behavior (especially, Figure 7(e)) due to ductile to brittle transition.

A detailed analysis of pure PP and PP-PCCF composite SEM micrographs was done after tribological testing. According to Figure 8(a) and (b), the fibers are pulled out due to interactions between pure PP, composites, and counter body zirconia. The force and speed were kept constant. However, 18-m sliding path variation and fiber fractions (0, 10%, 30%, and 40%) played a vital role. The fiber fraction variations provide voids, porosity, and asperities as a stress concentration. During an increase in the sliding path, the applied force produces strains and deformation in the region of stress concentration. The strains and deformation cause debonding and damage to composite materials. The same results were recorded for PP-PCPESF and PC-PCPETF composite materials.

The debonding and composite surface damage increases with the increase in fiber fraction. The increase of fiber fraction causes the increase of voids and porosity exponentially, seeFigure 6(a)–(c). According to Figure 7(a)–(b), the COF values of pure PP, 10% and 30% fiber fractions are very close to each other. Pure PP is used as a reference material. Additionally, the levels of porosity and voids were found similar and proved through Figure 6(a)–(c). However, the increment of fiber fraction (40%, etc.) beyond optimum level engendered huge damage and distortion. The corresponding results were Figures 5(a)–(j), 6(a)–(c), and 8(a) and (b).

A one-way factorial ANOVA was also conducted to compare the main effects of abrasive wear. The abrasive wear effects were significant at a p value of <0.05. According to Table 6, the abrasive wear yields an effect size of 98.7.

ANN regression and COF optimization

The optimization of COF was performed at three different forces, 0.3 N, 1 N, and 3 N, against fabricated composite materials using ANN regression. The fluctuations in machine values, optimization and validation are shown in Figures 9 and 10. Similarly, the regression value R and error in optimization of each individual composite material is shown in Table 7. The results were agreed with our investigations from reference[43] and of Friedrich.[24] The best correlation was found for pure PP.

However, R or correlation decreases with increase in fiber content. The results revealed that developed composites with 10% wt. of fibers (PCCF, PCPESF, and PCPETF) have a R value of 0.80. Similarly, all fabricated composites with 40% wt. of fibers expressed a regression value of 0.75. However, composites with 30% wt. of fibers have complex fluctuations in R values. The best optimization results were achieved at 0.3 N, where COF predicted, and experimental values show good fit. In 1 N

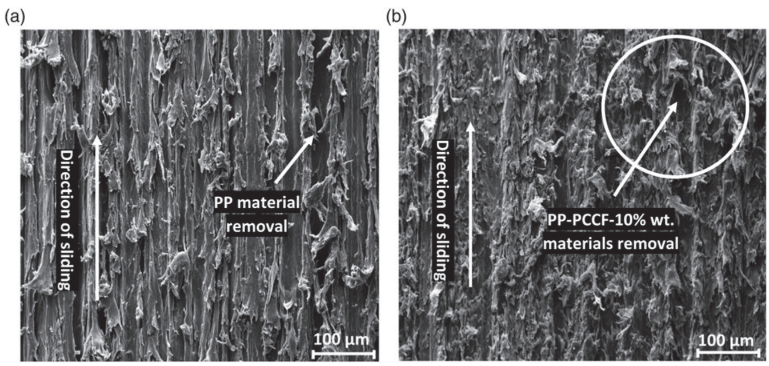

Figure 8. Representative SEM characterization of polymers after abrasion testing: (a) Pure PP after sliding test and (b) PP-PCCF Composites after sliding test.

Table 6. Single-factor ANOVA test of abrasive wear.

Source of variance	Tensile behavior	Sum of square (SS)	Degree of freedom(df)	F value	p-value
Between group	Abrasive wear	13.75	9	0.238	0.987

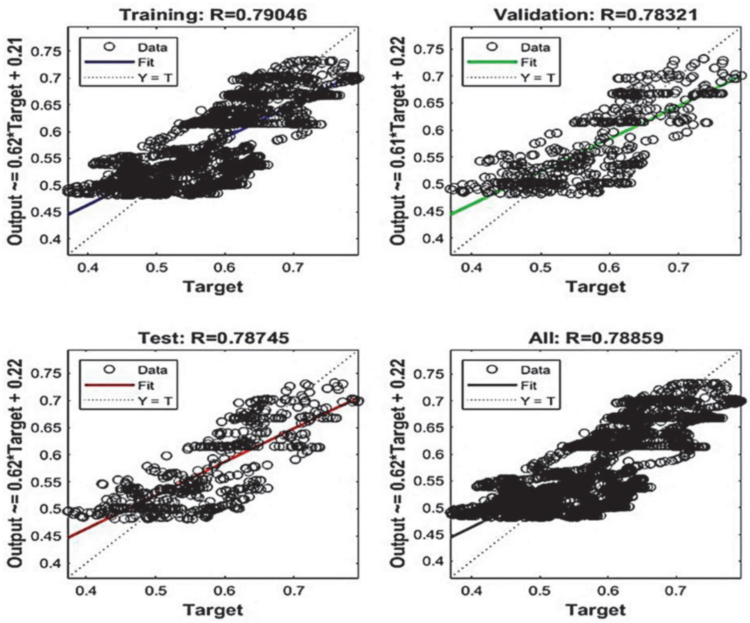

Figure 9. COF optimization using ANN.

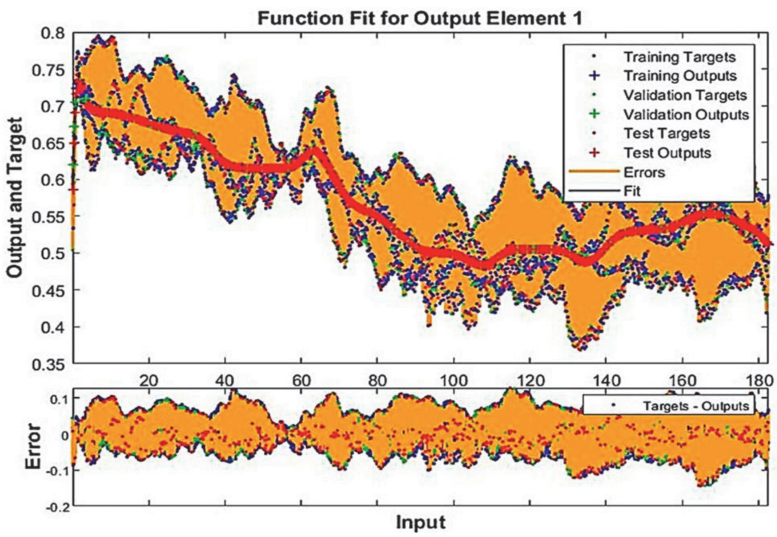

Figure 10. COF validation using ANN.

and 3 N, there was a huge difference between forecasted and actual results, which is attributed to the values. The composite's defects and ductile to brittle transition may contribute to this phenomenon.

The Rockwell surface hardness (HR15W) for fabricated composites was also mentioned in our previous publication. The Rockwell hardness values were 33 HR15W, 13 HR15W, 19 HR15W and 23 HR15W for pure PP and

Table 7. ANN regression values of each individual polymer composite material.

Composite family	Regression R value	Error of machine
Pure PP	0.80	0.05
PP-PCCF 10% wt.	0.78	0.05
PP-PCCF 30% wt.	0.79	0.05
PP-PCCF 40% wt.	0.75	0.06
PP-PCPESF 10% wt.	0.77	0.05
PP-PCPESF 30% wt.	0.78	0.05
PP-PCPESF 40% wt.	0.76	0.07
PP-PCPETF 10% wt.	0.79	0.05
PP-PCPETF 30% wt.	0.76	0.07
PP-PCPETF 40% wt.	0.77	0.07

respective fabricated composite materials. Besides PP reference material, the value of hardness increases with the increase in fiber fractions. Moreover, Rockwell hardness values of all composites indicate soft surface behavior. The rough texture, random orientation and structure of cotton fibers impart reasonable mechanical, chemical and tribological properties to recycled composite materials.[44]

Circularity and technical discussion

The current investigations have an influential role in the industrial circularity of polymer wastes. The combination of optimum grinding and extrusion before injection molding helps to produce polymer composites with suitable surface, mechanical and tribological properties.[45] The investigated family of composite materials exhibit almost the same mechanical properties as composites fabricated using virgin polymers. For instance, PP-PCCF 10% wt. and PP-PCPESF 10% wt. have manifested good plastic deformation (almost 10% in terms of extension), reasonable tensile strength (26–29 MPa), modulus of elasticity (1401–1475 MPa) and abrasive wear (3.5×10^{-6}–4.1×10^{-6} mm^3/Nm). Such types of composites could be used functionally in civil and automotive work. This is an example of successful utilization of PC cotton and synthetic polyesters. An increase in fiber addition (just after 10% wt.) makes manufactured composites brittle, hard and very stiff. Such PP-PCCF and PP-PCPES composites materials, especially with 40% wt. fibers, show reasonable resistance to impact loads and environmental impacts. Besides this, in all PCCF and PCPESF based developed composites, fibers help in transferring loads from one phase of matrix to another. The PP-PCPETF particulate composites exhibit lower strain due to the small size of PCPETF fibers. The PCPETF produces special crystalline phases with PP. These phases provide reasonable strength, toughness, and breaking strength at the cost of plastic deformation. Such types of PP-PCPETF composites can be used for commercial applications where mechanical designs are in static conditions during service life. All composites with 30% wt. fiber (PCCF, PCPESF, and PCPETF) loadings present complex behavior. These composites have moderate strength, flexibility, and tribological properties.

Overall, as a pilot production, fiber-reinforced and particulate composites were fabricated successfully using injection molding without using mixers and binders. The customer satisfaction was assured by SEM characterization, mechanical testing, and tribology investigations. Therefore, the developed methodology and physical testing can be used operationally for commercial conversion of polymer wastes into suitable products.

Conclusion

The cutting model, free-body diagram and reported PC-metallic tribological systems are utilized for optimum cutting of PCCF, PCPESF and PCPETF. The average length, diameter and area of cotton and synthetic polyester reinforced fibers were 3.5 mm, 17.50 μm, and 250 μm^2. Similarly, the same parameters for polyethylene terephthalate were 0.30 mm, 0.01 μm and 0.20 μm^2, respectively. Finally, PP-PCCF, PP-PCPESF and PP-PCPETF fiber-reinforced and particulate composites were developed for surface, mechanical and tribological applications.

Asperities, rough areas, voids, pits, line cracks, fiber delamination have appeared in recycled composites due to surface damage and microfibrils creation. Pure PP and PP-PCCF 10% wt. composites demonstrate maximum value of tensile strength (29–31 MPa) and percentage of extension (10%). However, fiber addition creates microdefects that result in decrease of tensile strength (22 MPa), fiber-matrix interfacial adhesion and strain (3.5) of PP-PCCF, PP-PCPESF, and PP-PCPETF composites with 40% wt. of fiber loadings (PCCF, PCPESF, and PCPETF). PP-PCCF fiber-reinforced composites (with 40% PCCF) show a maximum value of modulus of elasticity (2751 MPa) due to good adhesion between PP-PCCF phases. Similarly, PP-PCPETF particulate composites (with 40% PCPETF) present the lowest stiffness constant (750 MPa) due to poor adhesion and the smaller size of PCPETF.

The lowest value of COF (0.65) was recorded for Pure PP. However, PC-PCCF composites with 40% fiber loadings exhibit a maximum value of COF in the range of 1.50–1.65 due to higher hardness and brittle behavior. Additionally, PP and PP-PCCF with 40% fiber loadings express minimum (3×10^{-6} mm^3/Nm) and maximum values (6.5×10^{-6} mm^3/Nm) of abrasive wear, respectively.

All types of PP-PCCF, PP-PCPESF and PP-PCPETF composites with 30% wt. fiber amount show complex mechanical and tribological behavior due to ductile to brittle transition. Moreover, the ANN regression shows the best optimization and validation results at 0.3 N, where COF predicted, and experimental values show good fit.

Operationally, the PCCF, PCPESF and PCPETF waste can be utilized successfully for recycling and commercial applications. Moreover, the developed methodology of grinding, injection molding, tribological and mechanical testing could be introduced for pilot and industrial production of different polymer products.

Author contributions

Abrar Hussain: Conceptualization, Methodology, Composite Development, Tensile testing, abrasion testing, ANN regression, SEM analysis, review, and editing, complete experimentation, Writing – original draft, Validation. Single-factor ANOVA. Moreover, CE industrial implementation review, open system to closed system transformation, sustainability check. Vitali Podgursky.: Supervision review and editing, Validation. Dmitri Goljandin.: Cutting, grinding of cotton waste. Maksim Antonov: Tribological testing supervision.

Declaration of conflicting interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

Funding

The author(s) disclosed receipt of the following financial support for the research, authorship, and/or publication of this article: This research was supported by Estonian Ministry of Research and Education, Tallinn University of Technology under project KIK (19019).

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Publication VI

Abrar Hussain, Goljandin, D., Podgursky, V., Abbas, M. M., & Krasnou, I. (2023). Experimental mechanics analysis of recycled polypropylene-cotton composites for commercial applications. Advanced Industrial and Engineering Polymer Research, 6(3), 226-238. doi.org/10.1016/j.aiepr.2022.11.001.

Advanced Industrial and Engineering Polymer Research 6 (2023) 226–238

Contents lists available at ScienceDirect

Advanced Industrial and Engineering Polymer Research

journal homepage: http://www.keaipublishing.com/aiepr

Experimental mechanics analysis of recycled polypropylene-cotton composites for commercial applications

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ARTICLE INFO

Article history:
Received 2 September 2022
Received in revised form
28 October 2022
Accepted 1 November 2022

Keywords:
Fiber reinforced composites
Circular economy
Tensile testing
Polymer composites
Textile wastes

ABSTRACT

The sustainable processing of recycled products requires veritable testing during quality control for commercial application. In this research work, mechanical (ASTM D3039), compression (ASTM D5467) and impact (ASTM A370) are utilized to observe the usability, diversity, and suitability of the developed polypropylene-postconsumer cotton fibers (PP-PCCF) composites for industrial applications. The cotton waste was ground using a grinding machine. The ground fibers were introduced to manufacture composites from 0 to 40% fiber loading variations. The fine cotton fibers and synthesized composites were characterized by scanning electron microscope before and after mechanical testing. The fiber length, diameter and area were in the range of 2.5 mm–5.5 mm, 12.5 μm–22 μm and 200.15 μm^2–250.50 μm^2, respectively. The engineering and design values were tensile strength (31.16 MPa–22.77 MPa), breaking strength (26.69 MPa–22.77 MPa), modulus of elasticity (2223.79 MPa–2770.77 MPa), and extension (17.48–3.21). Similarly, flexural strength, modulus, energy, and fracture force are also enhanced with an increase in fiber loading. The impact energies of pure polypropylene and PP-PCCF composites (with 10, 30, and 40% PCCF contents) were 50 kJ/m^2, 48 kJ/m^2, 43 kJ/m^2, and 58 kJ/m^2. The micrographs of PP-PCCF composites prove that the density of voids is enhanced with an increase in fiber contents. The PP-PCCF composites with 0%–30% fiber loadings showed minimum defects and were observed to be suitable for structural applications. On the other hand, the PP-PCCF composites with 30%–40% fiber loading are acceptable for environmental applications.

1. Introduction

A circular economy (CE) or circularity is a system of closed production. CE provides innovative solutions for the transformation of polymer wastes into valuable products. The study of renewable and environmentally friendly natural fibers as reinforcement phase is a complex field in recycling industries. Generally, the use of synthetic and natural polymer products produces waste [1]. The categorization of polymer wastes depends on level of impurity. However, recycling and reuse of polymers lowers the physical, chemical, tribological, quality and performance of recycled products [2]. The separation of polymer wastes, determination of friction values between machinery parts and polymer waste surfaces, stiffness, abrasive wear, fatigue, composition of polymer wastes, and other mechanical properties investigations assist to decide the suitable recycling technique. Therefore, the polymer wastes are selected for primary, secondary, tertiary, quaternary recycling and incineration [3–5]. The fibers mostly come from agroforestry wastes (rice, eggshells, husk, wheat, corn) and traditional wastes (wood, cotton, kenaf, flax, sisal, banana, etc.). The incorporation of fabric wastes reduces cost and carbon dioxide emissions [6].

Physical analysis plays an important role in the prediction of mechanical properties of polymer composites for commercial applications [7,8]. The elastic and plastic deformation, modulus of elasticity, tensile strength and fracture define the extent of damage, distortion, and withstand ability to predict the service life of produced products [9,10]. The hybrid and reinforcement composites

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https://doi.org/10.1016/j.aiepr.2022.11.001
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exhibit good mechanical properties. These features increase the importance of such materials in the automotive, construction, aerospace, military and nuclear industries [11]. The fiber addition is always desired to attain high performance and quality. The integral relationship can be utilized to affect mechanical design and commercial use. The reshaping and influence produced reasonable features like ease of processing, molding, casting, fatigue resistance, suitable surface finishing and hence tribological properties improvement for variety of applications [12–18].

A fiber reinforced composites (FRC) material is comprised of matrix and fiber phases. Thermosetting and thermoplastics materials like polyvinyl chloride (PVC), polypropylene (PP), polyesters (PE) and other famous matrix materials are introduced commercially to provide optimum mechanical properties, erosion, abrasive, and environmental resistance. The fibers are generally produced from petrochemical raw materials [19–21]. The crystallinity, higher distortion temperature, design stability, high tensile strength, reasonable flame resistance, blending and filling characteristics make PP a satisfactory candidate to use as a matrix. Moreover, fibers help in the transfer of load and withstand ability. The commercial use of natural fibers like cotton, jute, hemp, kenaf, wool, silk angora etc. requires extensive research for commercial applications. The natural fiber's core is composed and linked through microfibrils cellulose, lignin, and hemicellulose. Moreover, cellulose, hemicellulose and lignin contribute 60–85%, 10–25% and 2–15% to the final fibrous structure of natural polymers [22]. The classification of polymer composites is mostly based on their quality and performance, like common use, engineering applications and strategic utilization. The spherical powder, elliptical beads, filaments, and string shaped fibers are mostly derived from plants and animals. The natural fibers can absorb impurities, humidity, and water. The fabrication with matrix also imparts reasonable abrasion resistance, good resilience, compatible density, lower electrical conductivity, excellent acoustic properties and suitable thermal barrier [23–27].

In modern processing and recycling, the strategies and paradigm of circular economy are introduced to reduce polymer waste to its minimum level [27,28]. Commercially, primary, secondary, and tertiary processing are utilized for the transformation of wastes and raw materials into valuable products. Global polymers production has grown from 30 million metric tons in 1970 to approximate 400 million metric tons per year in 2020 and is still climbing. In processing, generally die extrusion, screw extrusion, calendaring, injection molding, compression molding, rotational molding, thermoforming, melt spinning and additive manufacturing are used traditionally for manufacturing of composites from thermosetting and thermoplastic polymers [29–32]. However, polymer wastes create operational challenges, for instance, waste inventory management, cost analysis, separation of various wastes, selection of recycling process and lowering in recycled product quality. The replacement of hazardous and unrecyclable polymers with sustainable and green recycled products is needed innovation in polymer processing [33–35]. Use of natural polymer composites and processing are increasing exponentially due to low production cost and rapid availability [36].

Normally, mechanical testing like tensile, impact and bending is performed during manufacturing to check usability and customer requirements [37]. The flexure strength, flexural modulus [38], impact energy [39], yield strength [40], toughness and bending resistance assist in predicting the quality, diversity, and performance of manufactured and recycled composites products. The variations in fiber content altered the physical and chemical characteristics of hybrid composites. The bearing of load and stress can be calculated from the following traditional formulation:

$$\sigma_{failure} = \frac{F_{failure}}{A} \quad (1)$$

The parameters $\sigma_{failure}$, $F_{failure}$, and A represent the value of stress failure, fracture force and cross-sectional area [41–43]. The natural reinforced composite materials express comparable mechanical properties for different commercial applications. The durability of composite materials relies on the nature of testing techniques and selection of recycling method. The fiber's distribution, fillers, presence of crystallinity, interfacial bonding between fiber-matrix phases and the nature of polymer materials also affect the performance of recycled products [44].

The mechanical testing evaluates the ability and performance of composites in terms of elastic deformation, plasticity, and fracture behavior for commercial applications. The structural (construction, automotive, electronic, nuclear, aerospace, etc.) and environmental (impact, wear, fatigue, abrasive, erosion, corrosion, temperature, etc.) applications can be predictable through simulation and machine

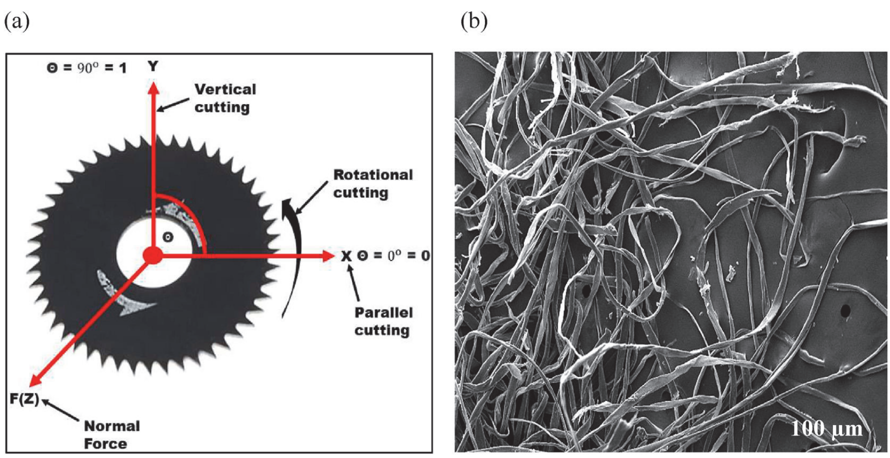

Fig. 1. Grinding mechanism: (a) Free body diagram of the cutting process along with tool disc, (b) SEM micrograph of PCCF after direct grinding.

Table 1
Fabrication scheme of PP-PCCF composites.

Composites name	PCCF content (wt.%)	PP content (wt.%))	Net weight of PP (g)	Net. Weight of PCCF (g)
R_1-R_5	0	100	500	0
C_{11}–C_{15}	10	90	450	50
C_{31}–C_{35}	30	70	350	150
C_{41}–C_{45}	40	60	300	200

Table 2
Mechanical tests specifications according to ASTM standards.

Composites Coding	Tensile test (ASTM D3039) specimen size (mm)	Compression (ASTM D5467) specimen size (mm)	Impact test (ASTM A370) specimen size (mm)
R_1- R_5	4 × 25.4 × 150	4 × 25.4 × 150	55 × 10 × 10
C_{11}– C_{15}	4 × 25.4 × 150	4 × 25.4 × 150	55 × 10 × 10
C_{31}–C_{35}	4 × 25.4 × 150	4 × 25.4 × 150	55 × 10 × 10
C_{41}– C_{45}	4 × 25.4 × 150	4 × 25.4 × 150	55 × 10 × 10

learning [45]. However, optimization of polymer hybrid composites is done using experimental testing. Additionally, the nature of fibers, coupling agents, fiber loading, and surface treatments are introduced to enhance the mechanical properties of hybrid composites. The microstructure of natural fibers (fiber core, lignin and microfibrils) causes poor wettability, thermal resistance, lower dimensional stability, and moisture absorption (40%–60%) [46–48].

In this manuscript, the mechanical properties and damage behavior of polypropylene (PP)- post-consumer cotton fibers (PCCF) reinforced composites with various fiber loadings. The tensile test (American Society for Testing and Materials (ASTM) D3039), bend test (ASTM D5467) and impact test (ASTM A370) are used to evaluate injection molding processing-mechanical properties in the relationship with industrial practices of circular economy. The free body diagram has been presented for cutting cotton polymer waste. The PP-PCCF fabricated composites of various cotton fiber fractions (0, 10%, 30%, and 40%) have been used for commercial optimization. The correspondence between PP-PCCF composites and mechanical properties has been analyzed. The surface analysis of PP and PCCF composites materials (10, 30 and 40% wt.) was done using a scanning electron microscope (SEM). Based on experimental results, the fracture models have been developed for the first time to study brittle and ductile behavior of polymer composites. Moreover, fabricated composites are also classified for commercial applications using literature review.

2. Materials and methods

2.1. Cutting of textile fibers

The pure PCCF waste was imported from the Estonian local industry. Initially, the cotton textile fabric was cut into small pieces. Fig. 1 shows the free body cutting diagram. The rotational, vertical, and parallel mechanism of cutting requires normal force to produce friction between the cotton fiber and the cutting tool [49]. The frictional contact produces shear between the cutting tool edge and the fabric surface. The mathematical formulation of a free body diagram in terms of vector product is abbreviated as

$$F_{Horizantal} \text{ x } F_{Perpendicular} = |F_{Horizantal}| \, |F_{Perpendicular}| \sin(\theta) \, k \quad (2)$$

where $F_{Horizantal}$ and $F_{Perpendicular}$ represent horizontal and vertical forces. The parameters k and θ show the normal to transverse and

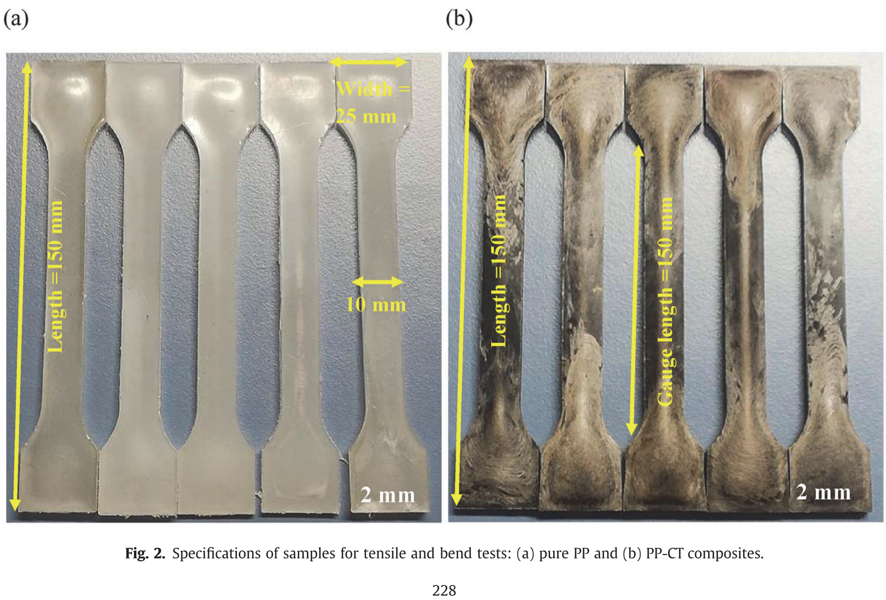

Fig. 2. Specifications of samples for tensile and bend tests: (a) pure PP and (b) PP-CT composites.

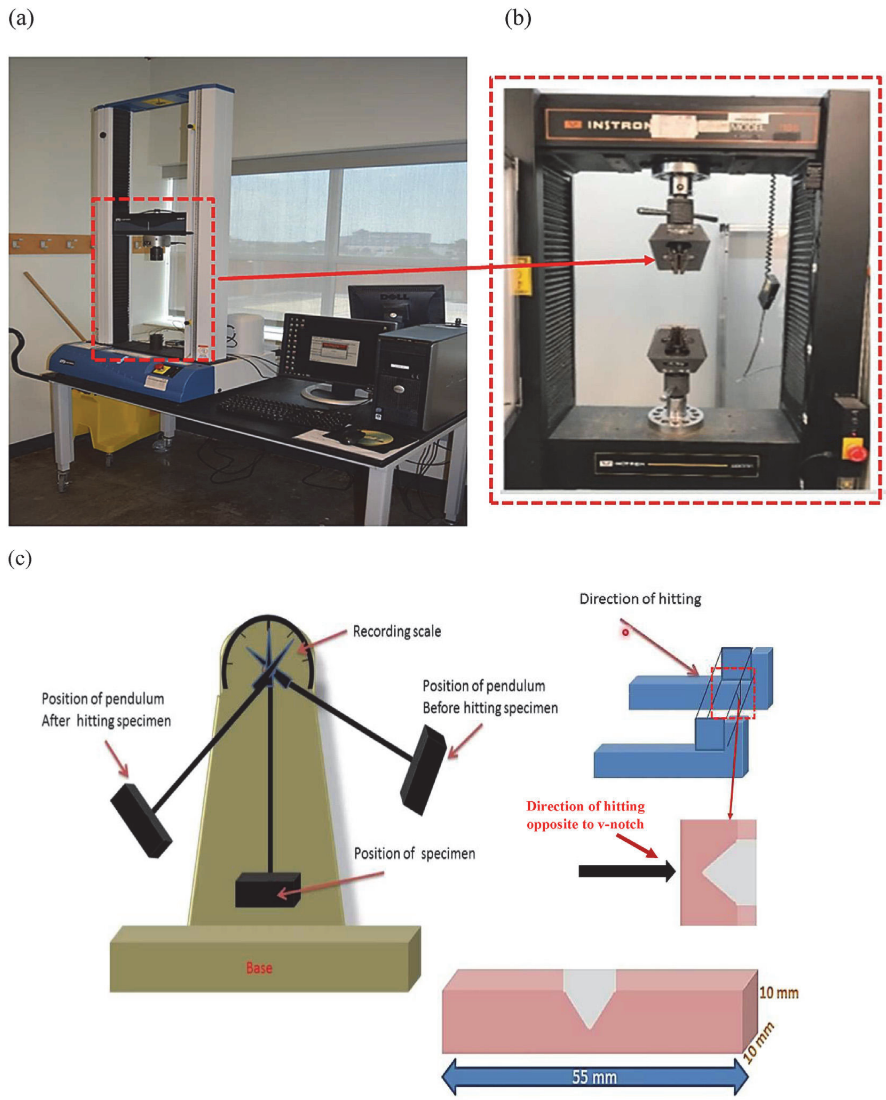

Fig. 3. Experimental setup and specifications demonstration: (a) Instron 5820 tensile testing machine, (b) measuring unit and (c) impact testing machine, principle and specimen specifications.

Table 3
Average mechanical true stress values of pure PP and PP-PCCF composites.

Specimen Label	Tensile strength in Megapascal (MPa)	Breaking strength (MPa)	Modulus of elasticity (MPa)	Extension at maximum load (mm)	Extension at maximum load (mm)
R_1–R_5	31.16	26.91	1223.79	9.77	17.48
C_{11}–C_{15}	27.05	25.69	1643.09	8.06	12.82
C_{31}–C_{35}	28.89	28.87	2590.97	4.27	4.44
C_{41}–C_{45}	24.96	24.92	2770.77	4.56	4.73

cutting angle, respectively. The friction force produced deformation (elastic and plastic) that caused the fracture of cotton waste [50,51]. However, the cutting ability relies on the nature of the cutting tool, cutting angle, and nature of cutting materials. The higher hardness, corrosion, fatigue, and wear resistance of hard tools assist in fine cutting of cotton fabrics. The higher values of these physical properties help to control the cutting resistance [17,52,53]. The cutting resistance between metallic and polymer surfaces increases

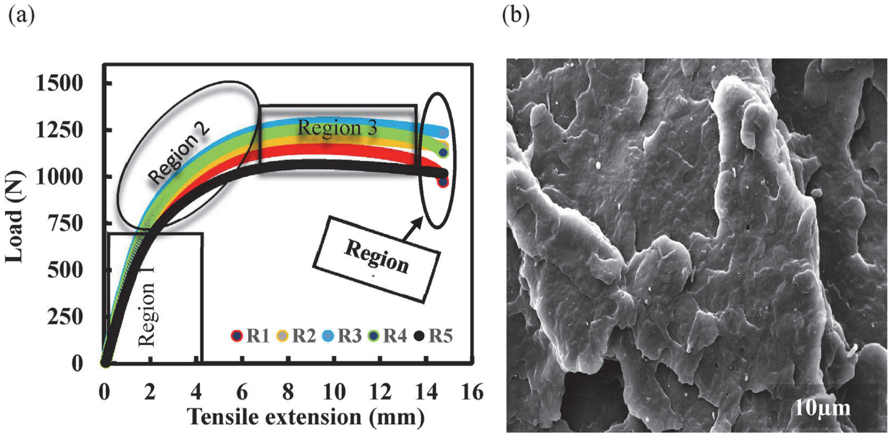

Fig. 4. The tensile properties and fracture surface characterization of pure PP polymer: (a) load-extension graph of pure PP and (b) fracture surface of pure PP.

Table 4
Average mechanical design stress values pure PP and PP-PCCF composites.

Specimen Label	Tensile strength (MPa)	Breaking strength (MPa)	Modulus of elasticity (MPa)	Extension at maximum load (mm)	Extension at maximum load (mm)
R_1–R_5	25.77	21.30	1139.79	8.98	14.61
C_{11}–C_{15}	25.39	24.88	1459.09	6.65	10.11
C_{31}–C_{35}	26.45	26.38	2467.78	3.65	3.65
C_{41}–C_{45}	22.77	22.77	2770.77	3.16	3.21

due to rough and distortion conditions of polymer wastes [54]. Similarly, the cutting ability is maximum ($\sin(\theta)$, where $\theta = 90°$) when the cutting disc is perpendicular to the cotton fabric surface. The sliding between the cutting tool and the cotton surface is minimal ($\sin(\theta)$, where $\theta = 0°$) when both are parallel to each other. However, the nature of cutting tools materials and fabric provide different frictional coefficients [54].

A direct type of grinding machine was utilized for cutting PCCF waste. The cotton fabric was transformed into fine fibers at a speed of 250–300 revolution per minute (rpm) for 10 min. The ground PCCF were observed using a scanning electron microscope (SEM).

The fabricated PP-PCCF Composites were used to investigate tensile, bending and compression properties. The plastic PP and natural cotton waste fibers were used as matrix and reinforcement materials. The PCCF waste was imported from the local Estonian polymer industry and grinded into fine fibers. The ground cotton was analyzed using a scanning electron microscope. The average PCCF diameter and length were 15 µm (µm) and 5 mm (mm), respectively. The grinded PCCF has 99% cellulose, 12,000° of polymerization, 73 average crystallinity, 37 g per 1000 m of yarns (g/tex) dry fiber strength and 47 g/tex wet fiber strength.

2.2. Composites fabrication steps

The PP-PCCF composites were produced using a compounder and injection molding machine. The cotton polymer waste was cut manually into small pieces using a cutter. In the next step, the small pieces were grounded with the help of a direct grinding machine into fine fibers. After that, the fine fibers and commercial powder were mixed for 10 min to get homogeneity of matrix and fiber phases. The mixed compound was melted in a compounder machine (extruder). The melt was passed through various 80 °C (°C), 100 °C, 150 °C and 190 °C melting zones in a chamber for 10 min. The melt was transformed into wires and grounded into fine pellets. The fine beads were then fabricated into 4 mm × 25.4 mm × 150 mm samples for mechanical testing. The cotton fiber loading was changed from 0 to 40%. The fabrication scheme of PP-PCCF composites is shown in Table 1.

2.3. Methods

The mechanical properties like elastic deformation, modulus of elasticity, yield point, tensile strength, fracture, strain, elongation, flexural stress, flexural modulus, energy, compressive load and impact energy were determined using ASTM D3039 tensile testing, ASTM D5467 bending test, ASTM D7249 compression test and ASTM A370 Charpy test, respectively. At least twenty tests were performed for each type of individual composite material. After that, on average, five samples were selected for results demonstration. The formulation and specifications are mentioned in Table .2 and Fig. 2(a) and (b). Therefore, R_1–R_5, C_{11}–C_{15}, C_{31}–C_{35}, and C_{41}–C_{45} abbreviations were used for reference material (pure PP) and PP-PCCF composites (10, 30, and 40 wt% fiber loadings), respectively. The experimental setup of mechanical testing machine, measuring unit, and impact testing machine along with specifications of samples were shown in Fig. 3(a)–(c) and Table 3, respectively [39,55–57].

According to standards, the gauge length and clamp length of PP-PCCF composite specimens for tensile and compression tests were 100 mm and 25.4 mm, respectively. The compression and tensile tests were conducted at a crosshead speed of 50 mm/s [54]. The Instron computerized data acquisition software was used to evaluate and predict various tensile and compression properties of composite materials. The respective samples were clamped into centrally oriented upper and lower jaws. The load-extension graphs were plotted automatically. Moreover, Charpy impact test with v-notch provides information about strain rate, impact energy (toughness) and

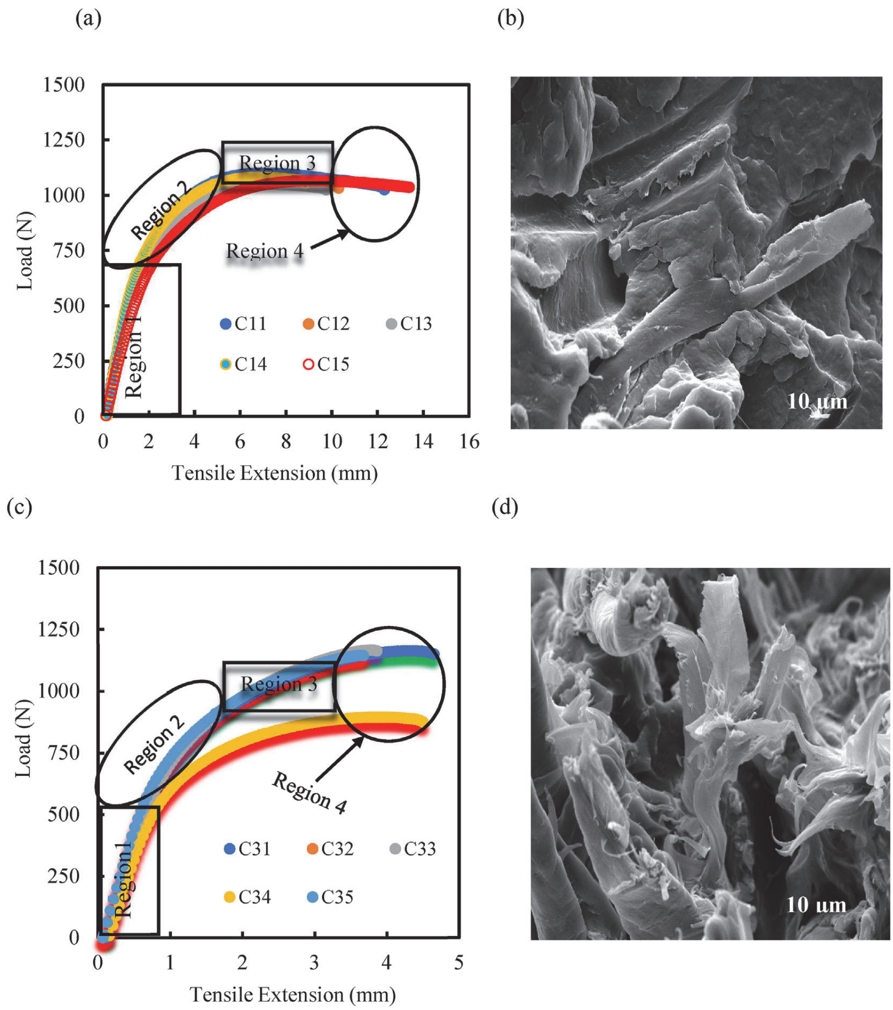

Fig. 5. Tensile Properties and fracture surface characterization of PP–PCCF composites at 10 and 30% wt. Fibre: (a) Load-displacement diagram of PP-PCCF composites with 10% fibre loading, (b) SEM micrograph of PP-PCCF composite's (10% wt. fiber loading) fractured surface, (c) load-extension graph of PP-PCCF (30% wt. fibre loading) composites and (d) SEM micrograph of PP-PCCF (30% wt. fibre loading) composite's fractured surface.

fracture for commercial applications. The quantitative and qualitative results assist in prediction of ductility, temperature transition and percentage of both brittle and ductile behavior.

3. Results and discussions

3.1. Engineering tensile testing

The load-extension graph and SEM image of the fracture surface of R_1–R_5 samples (pure PP reference material) are shown in Fig. 4(a) and (b), respectively.

In Graphs, regions 1, 2, 3, and four belong to elastic. Plastic, tensile and fracture behavior, respectively. Fig. 4 (a) proved that the displacement increased linearly with increase in applied load. The phenomenon represents the elastic limit, elastic deformation and hence the proportion limit of the pure PP material. Digitally, the elastic limit was found at a point (1.95 mm, 870 N (N)). After the elastic limit, the deformation starts to become permanent. As the applied load increases, the plastic deformation becomes permanent. The point at which the deformation becomes permanent is known as the yield point. Experimentally, these results belong to region 2. Region 2 consists of elastic deformation, dual (elastic plus plastic deformation), yield point and permanent plastic deformation. These properties exist in the range of 870 N–1300 N force and 2 mm–12.55 mm displacement. In the region of extension from 10 mm to 13 mm, the applied load touches to its peak value. The peak value at which composites can withstand maximum load is known as tensile strength of composite materials.

The results are supported by region 3. After bearing maximum load, the pure PP starts to soften. At the end, in the region of extension from 13.5 to 17.48 mm, the pure PP failed due to permanent plastic deformation and mechanical hardening. The quality of materials is

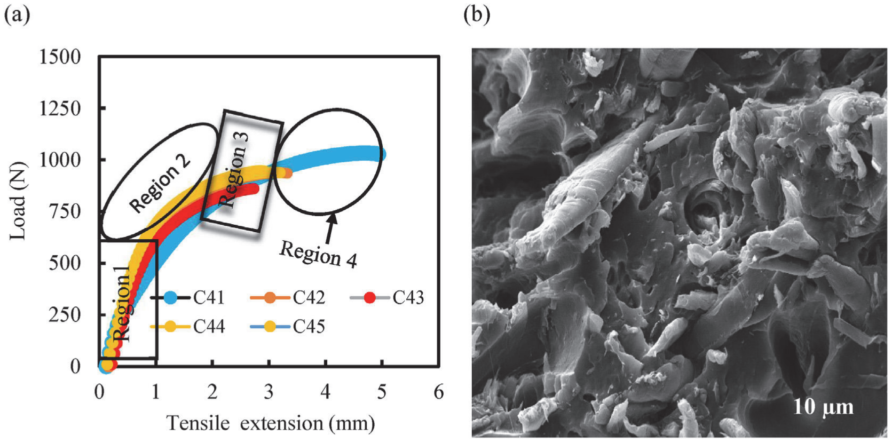

Fig. 6. Tensile properties and fracture surface characterization of PP-PCCF composites at 40% wt. Fibre loading: (a) Load-displacement diagram of PP-PCCF composites with 40% wt. Fibre loading, (b) SEM micrograph of fractured surface of PP-PCCF composites with 40% wt. Fibre loading.

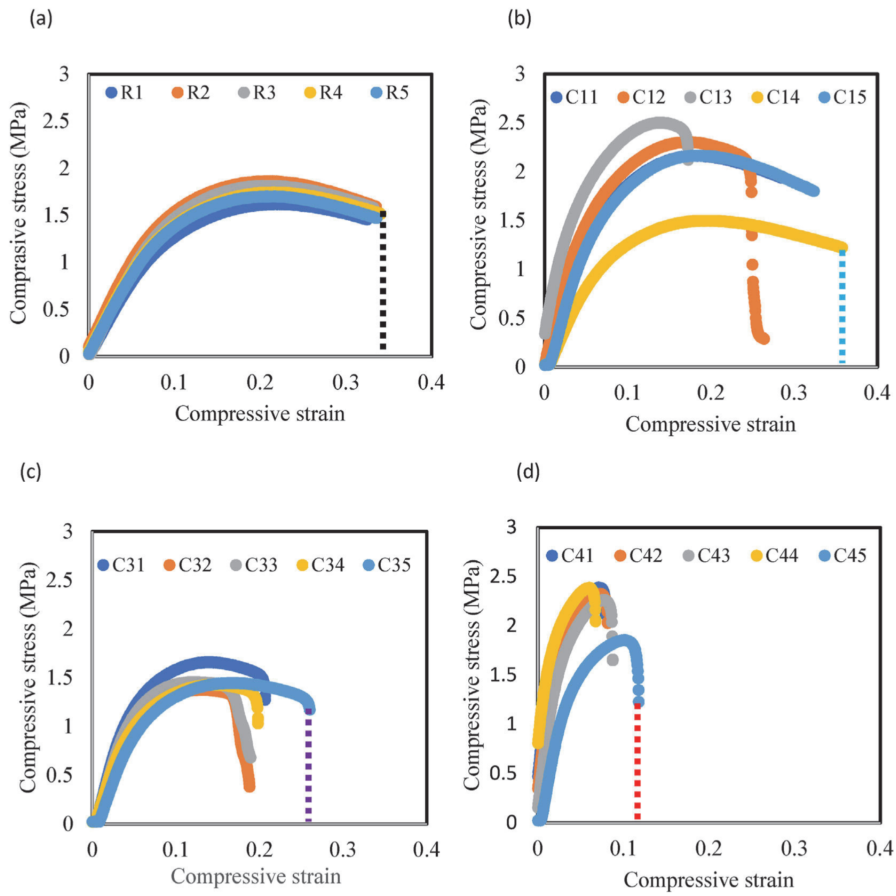

Fig. 7. Bend testing and flexural properties of PP-PCCF composites: (a) pure PP, (b) 10% PCCF loading composites, (c) 30% PCCF loading composites and (d) 40% PCCF loading composites.

Table 5
Average flexural properties of PP-PCCF composites with various fibre loadings.

Specimen Label	Flexural strength (MPa)	Energy at maximum load (J)	Flexural modulus (MPa)	Extension at maximum load (mm)	Maximum compressive load (mm)
R_1−R_5	45.70	0.76	1293.71	14.17	74.50
C_{11}−C_{15}	60.03	0.72	2750.95	12.84	100.05
C_{31}−C_{35}	40.86	0.40	2130.09	9.14	58.26
C_{41}−C_{45}	57.36	0.34	4229.75	4.54	95.60

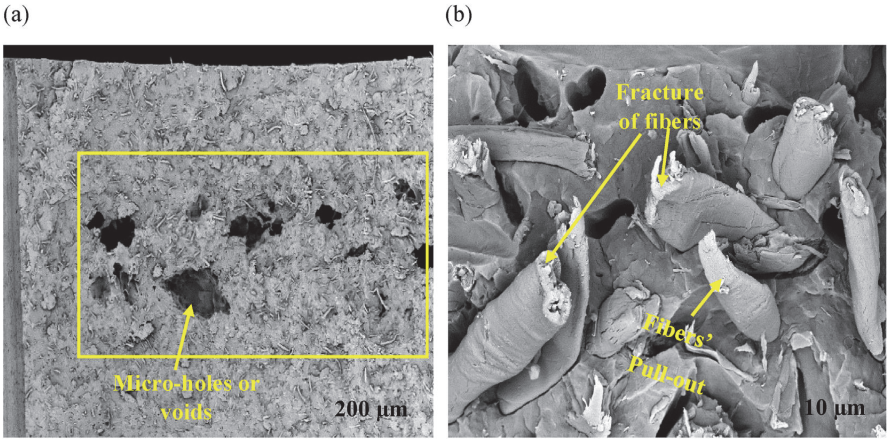

Fig. 8. SEM micrographs of the impact fractured surface of PP-PCCF composites: (a) micro-holes demonstrations (b) pull-out, impact and brittle fractured fibres.

termed as fracture. The respective possible results for engineering and design applications are shown in Tables 3 and 4.

The force-displacement curves of 10% (C_{11}−C_{15}) and 30% (C_{31}−C_{35}) PP-PCCF loading composites are depicted in Fig. 5(a) and (b), (c) and (d). However, these both hybrid composites peculiar behavior. The elastic limit for PP-PCCF composites with 10 and 30% wt. fiber loadings was observed at (1.75 mm, 775 N) and (0.70 mm, 550), respectively, see region 1 in Fig. 5(a) and (c). Similarly, according to region 2 in Fig. 5 (a) and (c), the plastic deformation, yield point and dual deformation exist in the range of 750 N−1100 N force and 1.75 mm−9.5 mm displacement. The region 3 of the same diagrams revealed the tensile force points at 1110 N and 1230 N for C_{11}− C_{15} and C_{31}−C_{35} type composite samples, respectively. After softening and lowering of tensile force, according to the mentioned diagrams, the fracture of C_{11}− C_{15} and C_{31}−C_{35} types of composites occurred at 1033 N and 1135 N.

The force-extension graph and SEM image of the C_{41}− C_{45} PP-PCCF composite simples' (40% wt. cotton fiber loading) fracture surface were shown in Fig. 6(a) and (b). The figure exhibits linear behavior within region 1 till at a point (0.22 mm, 375 N). The PP-PCCF composite simples' (40% wt. cotton fiber loading) were deformed permanently at the approximate yield point (0.75 mm, 670 N). The yield point corresponds to region 2. At a point of (2.75 mm, 950 N), the PP-PCCF composite simples' (40% wt. cotton fiber loading) bears maximum load known as tensile strength. The average tensile strength for 40% of the fiber loading composite was 22.77 MPa. Finally, the PP-PCCF composites (with 40% wt.) fractured within the range of (2.80 mm, 945 N) to (3.25 mm, 975 N) and region 4.

3.2. Bend testing and engineering aspects of damage mechanics

The flexural properties of R_1−R_5, C_{11}−C_{15}, C_{31}−C_{35}, and C_{41}−C_{45} PP-PCCF composite samples are presented in Fig. 7(a)–(d) and Table .5. The PP-PCCF composite materials (10, 30, and 40 wt% fiber loadings) show different behaviour due to matrix and fibre reinforcement materials variations. According to Table .5, the average flexural strength for R_1−R_5, C_{11}−C_{15}, C_{31}−C_{35}, and C_{41}−C_{45} was 1293.71 Megapascal (MPa), 2750.95 MPa, 2130.09 MPa and 4229.75 MPa, respectively. The flexural strength, compressive extension, compressive force, and compressive energy exhibit the same behaviour. Briefly, all mentioned flexural properties are enhanced by 10% wt. PCCF addition. However, with 20% and 30% PCCF contents, the flexural properties are reduced due to entanglement of cotton fibres. The fibre length, nature of fibre material and hydrophilicity also contribute to reduction of properties [58–61]. Interestingly, the value of flexural properties (expect energy and compressive extension) ameliorates. Similarly, Fig. 7(d) shows the minimum compressive strain due to high content of PCCF. The higher values of PCCF cause the poor adhesion between the PP-PCCF phase interface and the low level of crystalline phases present in the composite.

3.3. Impact test and behavior of synthesized composites

The impact strength of R_1−R_5, C_{11}−C_{15}, C_{31}−C_{35}, and C_{41}−C_{45} PP-PCCF composite's samples are expressed below. The value of impact energy, damage and fracture of composites can be divided into crack initiation, propagation, and termination stages. Initially, the surface and internal defects provide weak sites and stress concentration points. The cracks are produced and initiated by these weak sites due to applied load. The microcracks start to propagate when crack length exceeds critical values. The composites exhibit brittle fracture. The lack of plastic deformation prevents crack growth. The impact of energy increases gradually with the increase in fibre content. The average impact energies of R_1−R_5, C_{11}−C_{15}, C_{31}−C_{35}, and C_{41}−C_{45} are 50 kJ per meter square (kJ/m^2), 48.50 kJ/

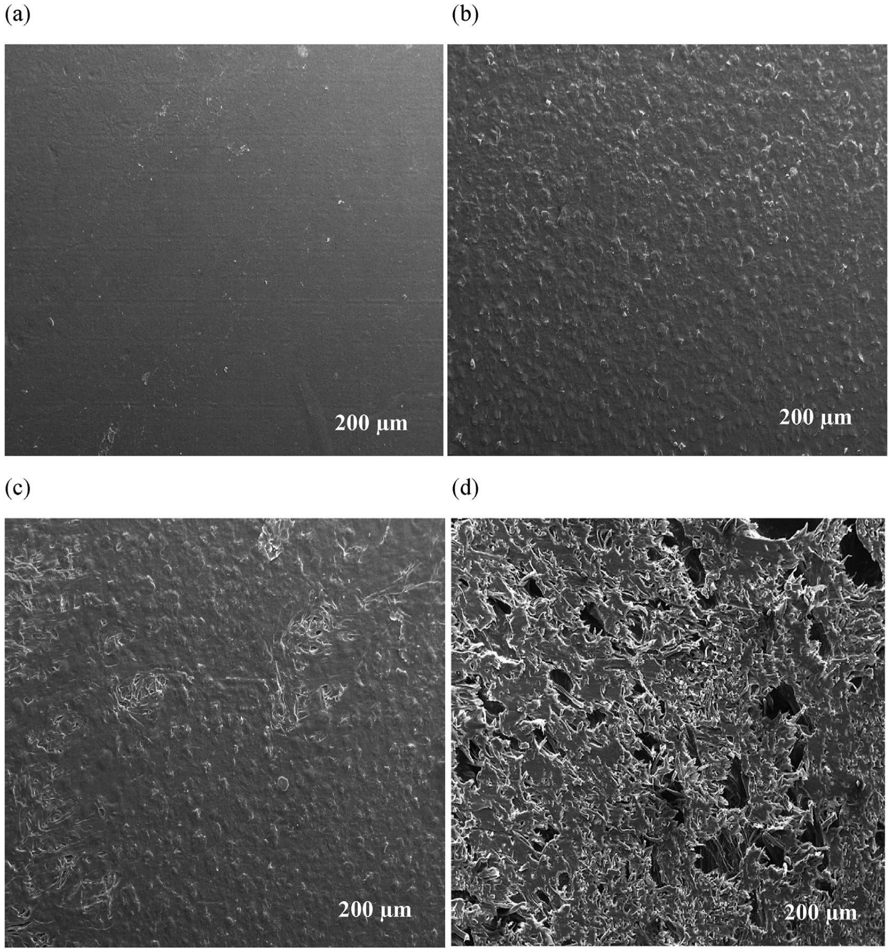

Fig. 9. The SEM characterization of PP and PP-PCCF bulk samples: (a) pure PP, (b) PP-P. CCF composites with 10% fiber loading (c) PP-PCCF Composite with 30% fiber loading (d) PP-PCCF Composite with 40% fiber loading.

m^2, 43 kJ/m^2, and 58 kJ/m^2. Traditionally, the impact strength of the PP-PCCF composite increases with an increase in fibre content.

The SEM micrographs of the PP-PCCF composite are shown in Fig. 8(a) and (b). Fig. 8(a) shows the rough surface and internal defects. The interfacial micrograph, Fig. 8(b), proved that cotton fibres are embedded properly in PP matrix. The increase in fibre fractions produces poor adhesion between cotton fibres and PP matrix.

3.4. Technical discussion

The simulations, especially of damage mechanics, assist in the prediction of mechanical properties [62]. The different mathematical formulations have been matured to explain fracture phenomena [63]. Bazant et al. demonstrated the mathematical model for evaluation of principal aspects of a failure mechanism. The composite fabrication creates various internal and external surface defects like voids, porosity, asperities, fiber pullout and micro cracks during molding and casting techniques. The extent of these defects depends on physical and chemical parameters like heating temperature, pressure, nature of matrix materials, percentage of reinforcement fibers, composition of fiber contents, length of fibers and wettability in the matrix phase. These interstitial and macro defects provide stress concentration sites for micro-crack initiation. The initiation of microcracks started from the weakest part of the mechanical design. The surface finishing during manufacturing plays a critical role in determining the extent of crack initiation. The excellent surface finishing increases the service life of a fabricated product [64–68]. The fracture mechanism of polymer composites is directly related to deformation produced due to mechanical stresses, chemical treatments, and surface damage. The deformation can be described physically in terms of strain or extension.

We have considered the SEM surface of R_1 - R_5, C_{11}–C_{15}, C_{31}–C_{35}, and C_{41}–C_{45} PP-PCCF composites to demonstrate the phenomenon of damage mechanism. The pure PP (R_1 - R_5) fractured plastically, see Figs. 4(a), (b), 7(a) and 9(a). The polypropylene shows good mixing and maximum crystallinity (40%). The good mixing and higher percentage of crystallinity provide suitable surface finishing. The surface emerged as smooth after synthesis. Pure PP is used as a

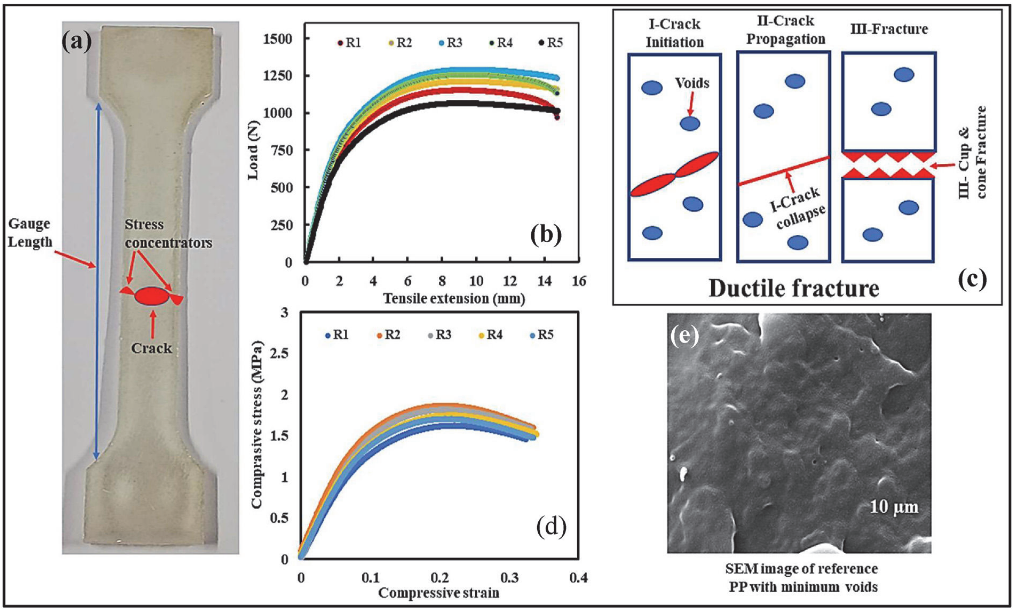

Fig. 10. Material behaviour analysis of pure PP: (a) sample for analysis, (b) stress-strain curve of reference material, (c) ductile fracture mechanism of hybrid composites, (d) compression analysis and (e) SEM image of fracture PP material.

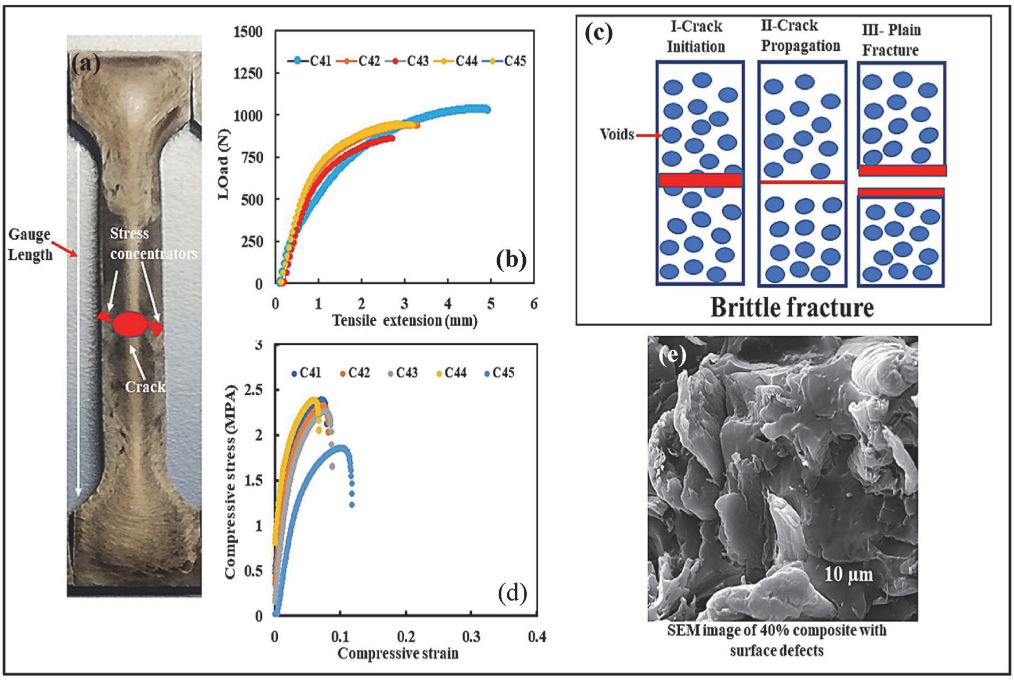

Fig. 11. Material behaviour analysis of C_{41}–C_{45} type of PP-PCCF composite: (a) sample for analysis, (b) stress-strain curve of C_{41}–C_{45} type of PP-PCCF composite, (c) brittle fracture mechanism of C_{41}–C_{45} type of PP-PCCF composite, (d) compression analysis and (e) SEM image of fracture of C_{41}–C_{45} type of PP-PCCF composite.

reference material. Fig. 9(b) shows the SEM micrograph of the $C_{11}-C_{15}$ PP-PCCF composite. The $C_{11}-C_{15}$ PP-PCCF composite are deformed and fractured plastically, see Figs. 5(a), (b), 7(b), and 9(b). The $C_{11}-C_{15}$ composite's samples show almost the same (just like pure PP) mechanical and surface properties. The interfacial, adhesive and adherents' defects start to produce. These defects arise due to the nature of natural fibres (micro-fibrils, fibrous core, lignin), the nature of plastic matrix materials and surface conditions (damage, distortion, rough, abrasive, fatigue, etc.). During processing, poor temperature control, unequal distribution of applied pressure, and poor bonding give rise to voids, asperities, holes and microcracks. Previous research of other natural fibre loadings shows the same behaviour as in $C_{11}-C_{15}$ and $C_{31}-C_{35}$ PP-PCCF types of composites [1–6]. The plastic deformation in terms of extension (see Figs. 4(a), 5(a), (c), and 6(a)) and strain rate (see Fig. 7(a)–7(d)) changes abruptly. The extension decreases from 15 mm (pure PP) to 2.5 mm ($C_{41}-C_{45}$ type of PP-PCCF composite). The compression test also expressed the same behaviour where strain contracted from 3.7 (pure PP) to 0.75 ($C_{41}-C_{45}$ type of PP-PCCF composite). Finally, the density of PP-PCCF composites defects has become huge, above 40% fiber loading. The asperities, voids, micro holes, micro-cracks, and other defects provide interstitial and substitutional stress concentration sites for crack initiation. The phenomenon is expressed in Fig. 10(a)–(e) (see Fig. 11).

Circular economy in terms of materials analysis (PCCF, PP), cost evaluations (almost free of cost), recycling technique selection (traditional injection and compression molding with technical alterations), and physical testing (mechanical, surface, tribological, chemical, etc.) provides engineering and industrial environment for sustainable production. The quality and performance directly depend on recycling. The quality associated with various attributes, like polymer materials nature, type of processing technique, level of impurity, sorting, separation of waste, polymer-machinery parts interactions, and finishing techniques. The optimization of these operational parameters leads towards sustainability. The $C_{11}-C_{15}$ and $C_{31}-C_{35}$ types of PP-PCCF composites exhibit large plastic deformation, survival under extreme loads with sudden failure, slow propagation of cracks, and high energy absorption. However, the $C_{41}-C_{45}$ types of PP-PCCF composites show little elastic deformation, low energy absorption and rapid crack propagation.

The collection of polymer wastes from textile, electronic, nuclear, construction, automotive, and other industries costs almost zero price. The sorting and separation of wastes preliminary decides the final recycling technique for processing. The conventional injection molding gains sustainability [69–74] due to pre-testing (surface, mechanical, tribological, chemical, physical, etc.) of polymer waste [15–17,48,50,52,53]. Therefore, the ductile composites ($C_{11}-C_{15}$ and $C_{31}-C_{35}$ types of PP-PCCF composites) can be used operationally for structural applications like automotive, construction, aerospace, and electronics. However, $C_{41}-C_{45}$ types of PP-PCCF composites can be used for environmental applications like building decoration, medical, and manufacturing industries.

4. Conclusions

The free model for cutting of post-consumer cotton fiber (PCCF) has been presented. The ground fine PCCF is used to fabricate polypropylene (PP)-PCCF reinforced composites. The PP-PCCF composites have 0, 10, 30 and 40 % wt. variation of PCCF and the following conclusions are drawn:

- In tensile testing, besides PP, the PP-PCCF composite with 10 % wt. PCCF shows good tensile properties with minimal surface defects. The tensile strength, extension and breaking strength decreases with an increase in PCCF loading. However, the modulus of elasticity is improved with an increase in PCCF contents.
- According to bend testing, flexural strength, strain, energy at maximum load and compressive extension are lowered with increase in fiber content. Additionally, the PP-PCCF composite with 40 % wt. PCCF expresses a maximum flexural constant of 4229.75 MPa.
- The average impact energies of R_1-R_5, $C_{11}-C_{15}$, $C_{31}-C_{35}$, and $C_{41}-C_{45}$ are 50 kJ/m^2, 48.50 kJ/m^2, 43 kJ/m^2, and 58 kJ/m^2, respectively.
- The PP-PCCF composite with 30 % wt. PCCF presents complex behavior (regarding tensile, bending and impact testing) due to the transition from ductile to brittle behavior.
- The increase in PCCF causes the creation of voids, micro-cracks, poor adhesion between the PP-PCCF interface and surface asperities.
- The ductile and brittle fracture models suggest the use of PP-PCCF composites with 10 and 30% wt. PCCF can be used for structural applications like automotive, construction, aerospace, and electronics. However, PP-PCCF composites with 40% wt. can be utilized for environmental applications like building decoration, medical, and manufacturing industries.

Data availability statement

The original contributions presented in the study are included in the article and further inquiries can be directed to the corresponding author.

Authorship contribution statement

Author Contributions: Abrar Hussain.: Conceptualization, Methodology, Composite Development, Tensile Testing, Bend Testing, Impact Testing, review, and editing, complete experimentation, Writing – original draft, Validation. Moreover, CE industrial implementation review, open system to closed system transformation, sustainability check. **Dmitri Goljandin.:** Cutting, grinding of cotton waste. **Vitali Podgursky:** Supervision and validation. **Muhammad Mujtaba Abbas.:** Review and Editing**.**

Funding

This study has received funding from Tallinn University of Technology.

Declaration of conflicting interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

Acknowledgements

This research was supported by Estonian Ministry of Research and Education and Tallinn University of Technology under project KIK 19019.

Abbreviations:

PP	Polypropylene
mm	Millimeters
μm	Micrometre
PCCF	Post-Consumer Cotton Fibres
CE	Circular Economy

PVC	Polyvinyl Chloride
$\sigma_{failure}$	Stress at Failure
A	Cross-section Area
SEM	Scanning Electron Microscope
$F_{Perpendicular}$	Perpendicular Cutting Force
θ	Transverse Cutting Angle
g/tex	Grams Per 1000 Meters of Yarns
s	Second
ASTM	American Society for Testing and Materials
%	Sign of percentage
MPa	Mega Pascal
kJ/m^2	Kilo Joule Per Meter Square
FRC	Fiber Reinforced Composites
PE	Polyesters
$F_{failure}$	Fracture Force at Failure
wt	Weight
$F_{Horizontal}$	Horizontal Cutting Force
k	Normal Cutting Angle
rpm	Revolution Per Minute
$^{\circ}C$	Degree Celsius
N	Newton

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KIK19019	Tekstiilijäätmete purustamistehnoloogia ja uudsete materjalide arendamine tekstiilijäätmete väärindamiseks ning ringmajanduse toetamiseks

ISSN 2585-6901 (PDF)
ISBN 978-9916-80-078-2 (PDF)