TALLINNA TEHNIKAÜLIKOOL
TALLINN UNIVERSITY OF TECHNOLOGY

Department of Materials and Environmental Technology

"Effect of some organic and inorganic filler on physical properties of LDPE composites"

"Mõningate orgaaniliste ja anorgaaniliste täiteainete mõju LDPE füüsikalistele omadustele"

MASTER THESIS

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TALLINNA TEHNIKAÜLIKOOL
TALLINN UNIVERSITY OF TECHNOLOGY

Materjali- ja keskkonnatehnoloogia instituu

“Mõningate orgaaniliste ja anorgaaniliste täiteainete mõju LDPE füüsikalistele omadustele”

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Hereby I declare, that I have written this thesis independently.

No academic degree has been applied for based on this material.

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Abstract

In the present study, composites of low density polyethylene (LDPE) has been prepared with some organic filler (polyester fiber) and inorganic fillers (glass fiber, oil shale ash, limestone ash, mineral wool fiber) are provided in the form of waste as they are creating huge land fillings and polluting nature. The later aim is to observe the effect of organic and inorganic fillers on the mechanical properties of LDPE, in order to improve the flexural properties and tensile properties of LDPE. The further work is to increase heat conductivity and cooling rate of LDPE by use of filler like mineral wool fiber, oilshale ash, and limestone ash, in order to increase the processing speed of LDPE composite for large scale production.

Kokkuvõte

Käesolevas magistritöös valmistati madaltiheda polüetüleeni komposiite mõningate orgaaniliste ja anorgaaniliste täiteainetega nagu klaaskiud, põlevkivi tuhk, paekivi tolm, mineraalsed ja polüesterkiud. Nimetatud täiteained esinevad jäätmetena, mis kuhjuvad prügilatesse ja saastavad loodust. Töö eesmärgiks on selgitada nimetatud orgaaniliste ja anorgaaniliste täiteainete mõju madaltiheda polüetüleeni mehaanilistele omadustele ja parandada selle materjali tõmbe- ning paindeomadusi. Samuti uuriti võimalusi madaltiheda polüetüleeni soojusjuhtivuse ja jahtumiskiiruse suurendamiseks kasutades mineraalseid kiude, põlevkivituhka ja paekivitolmu. Materjali suurem jahtumiskiirus võimaldab suurendada masstootmisestöötlusprotsesside kiirust.

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List of abbreviations and acronyms

BPO	Benzoyl Peroxide	
$CaCO_3$	Calcium carbonate	
C&D	Construction and demolition	
Fe_2O_3	Ferric oxide	
GF	Glass Fiber	
HDPE	High Density Poly Ethylene	
LDPE	LowDensity Polyethylene	
MA	Maleic Anhydride	$C_4H_2O_3$
MAPP	Maleic Anhydride grafted Polypropylene	
MF	Mineral wool Fiber	
PEF	Poly Ester Fiber	
PEgMA	PolyEthylene grafted Maleic Anhydride	
PEK	Polyether Ketone	
PET	Poly Ethylene Terepthalate	
PP	Poly Propylene	
PS	Poly Styrene	
PVC	Poly Vinyl Chloride	
S_iO_2	Silicondioxide	
UTM	Universal Testing Machine	
VTMS	Vinyl TriMethoxy Silane	$H_2C{=}CHSi(OCH_3)_3$
WPC	Wood Plastic Composite	

Introduction

Recycled thermoplastics are some of the major components of global municipal waste and it can be used as raw material for recycled composites because of huge volume and low cost. High density polyethylene (HDPE), low density polyethylene (LDPE), polypropylene (PP), Polyethylene terephthalate (PET), polystyrene (PS) and polyvinyl chloride (PVC) are the primary constituents of plastics in recycled plastics. If the recycled plastics are considered as new materials in plastic composite production, it is necessary primarily to understand well the elemental and fundamental structure of these materials. By knowing the properties of recycled plastics, the processes for manufacturing final product can be well controlled and then the relationship between the properties of recycled plastics and their mechanical aspects can be better understood as well as those of the resulted composite products. At the end of the first life cycle of plastic products, or after being re-used several times, plastics degradation is a problem that frequently occurs when a polymer is submitted to a process or service. Degradation processes are generally quite complex; often more than one type of degradation is operational, e.g. thermo-oxidative degradation, thermo-mechanical degradation, etc. Degradation usually manifests itself as discoloration, loss of mechanical properties. Although in some cases the properties of recycled plastic is similar to virgin plastic, mostly the properties of waste and recycled plastics are very different from virgin plastics[1]. Textile wastes According to the most recently released Figures from the Environmental Protection Agency (EPA), in 2010 Americans discarded 13.1 million tons of textiles. Only 15% of which was reclaimed for recycling, while more than 11 million tons of textiles were dumped in landfills across the country. Polyester fibers (PEF) are organic in nature and it has been used widely in drinking beverage manufacturing. In this experiment one used organic fillers such as polyester fiber. And inorganic fillers such as glass fiber (GF) and mineral wool fiber (MF), oilshale ash and limestone ash to know its effect on the mechanical and cooling properties of LDPE. The main objective is to know the effect of fillers on the tensile and flexural properties of LDPE, and to know the effect of compatibilizer on the mechanical properties, to increase the flexural properties of LDPE by the incorporation of filler. And to analysis the effect of fillers like oilshale ash, limestone ash and mineral fiber to improve the heat conductivity and cooling rate of LDPE in order to increase the processing time.

Chapter 1.Literature overview

1.1. Composites

The development and use of composites begin in 1940'swith use of "Glass Fiber" (GF) as filler and later it went through different generations like "High Performance Composites" (carbon fiber composites) and "nanocomposites". Composites are basically mixture of raw polymer and reinforcement material (Figure 1) which affects the mechanical and physical properties of composite [1]. Use of plastic composites has been increased in automobile fields and aerospace applications because of its less weight compare to metals. The high performance composites are usually made of carbon fiber, aramid fiber, coupling agent will use to increase the bond between polymer and reinforcing filler, the selection of coupling agent is depend on surface chemistry of filler. Usually composites are ductile or tough material with low density [3].

Products manufactured from waste and recycling plastic has attracted more and more attention in the past decade because of the ecological and environmental requirements. Post-consumer waste consists of a wide variety of polymer types. The largest fraction of waste is mainly composed of polyolefins, such as polyethylene (PE) and polypropylene(PP) (60–70%) and the remaining include polystyrene (PS) (10–15%), polyvinyl chloride (PVC) (15%), and polyethylene terepthalate (PET) (5%) [3].

The main problems in post-consumer recycling are due to the degradation undergone during lifetime and processing steps. The properties–reprocessing relationships are an important tool for determining not only the properties of recycled polymers, but also strategies to apply for obtaining recycled polymers with good mechanical properties.

Typically, polyolefin are reinforced with fibers or fillers. The incorporation of mineral fillers into thermoplastics has been widely practical in industry to extend them and to enhance certain properties. Fillers often increase the performance of polymeric product. The addition of fillers to polymers is a fast and cheap method to modify the properties of the base materials.

There are a large range of polyolefin composites in market. During the last few decades, these have been of interest to industry and academia, especially in the areas of automotive, aerospace, electronic systems, medical products, civil construction, chemical industries, and other consumer applications. This is because of their superior properties such as high strength to weight ratio, good electrical insulation, ability to transfer load, and easy and inexpensive processing. In polymer composites, the matrix phase is the primary phase, which is more ductile phase and it holds the reinforcement that is the secondary phase. Reinforcements are usually stronger than the polymer matrix that improves the mechanical properties of the polyolefin composite [2].

Strictly speaking, polymers can be considered composite materials, since functional fillers are often incorporated in order to provide specific properties. While commodity resins such as PVC, PS, PE, and PP are often sold as pure resins, price escalations and possible uncertainty in petroleum feedstock, as well as increased performance criteria, have established a widespread market for functional fillers. Filled polymer composites extend the available volume of resins, improve many of the physical properties and are generally cheaper. In certain instances, they facilitate faster production cycles and better dimensional stability.

Different fillers are introduced into polymer matrices to decrease manufacturing cost and to develop mechanical properties. In some cases, the filler also acts as low cost diluents to the formulation. As a result, there are literally hundreds of grades of polyolefin composites commercially available today. Common reinforcing fillers for polyolefin include GF, wood flour, talc and calcium carbonate [4] .

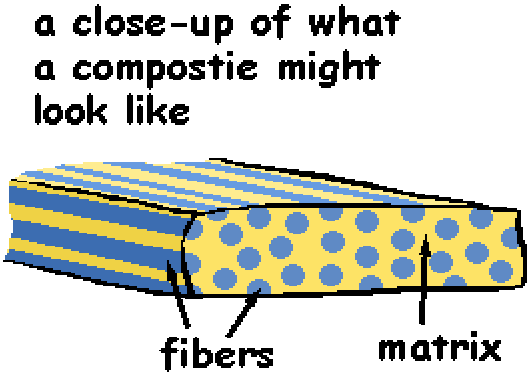

Figure 1. Composites in general form

Advantages of composites

- High strength to weight ratio (low density high tensile strength) or high specific strength ratio
- High tensile strength at elevated temperatures
- Corrosion resistance
- Dimensional changes due to temperature changes can be much less.
- Impact loads or vibration – composites can be specially formulated with high toughness and high damping to reduce these load inputs.
- High toughness
- High creep resistance

Disadvantages of composites

- Material costs
- Operating temperature can be an issue for polymeric matrix
- Fabrication/ manufacturing difficulties
- Inspection and testing typically more complex

1.2. Thermoplastics

Plastics have become the most common engineering materials over the past decade. In the past 5 years, the production of plastics on a volume basis has exceeded steel production. Due to their light weight, easy processability, and corrosion resistance, plastics are widely used for automobile parts, aerospace components, and consumer goods. Plastics can be purchased in the form of sheets, rods, bars, powders, pellets, and granules. With the help of a manufacturing process, plastics can be formed into near-net-shape or net-shape parts. They can provide high surface finish and therefore eliminate several machining operations. This feature provides the production of low-cost parts[5]. Plastics are not used for high-temperature applications because of their poor thermal stability. In general, the operating temperature for plastics is less than 100 °C. Some plastics can take service temperature in the range of 100 to 200 °C without a significant decrease in the performance. Plastics have lower melting temperatures than metals and therefore they are easy to process[3].

Low Density Polyethylene (LDPE) is the most common packaging material used for packaging a wide range of products. The efficient disposal of these plastic materials is a herculean task as they are not easily degradable and pose detrimental effects on the environment. Approximately 35% of plastics produced in the developed countries are consumed for packaging. Today the plastic consumption for food packaging in India itself is about 308,000 tones, which forms 8-10% of all types of packaging materials used in food packaging. LDPE is more popularly used [5]. LDPE is a soft and tough material which exhibits high elongation properties.

When a composite is subjected to an applied load, the matrix deforms and transfers the external load uniformly to the fibers. The matrix also provides resistance to crack propagation and damage tolerance owing to plastic flow at the crack tips. Their function is also to protect the surface of fibers from adverse environmental effects and abrasion, especially during composite processing. Plastic matrices can generally be classified into two major types: thermoplastics and thermosets. The selection criteria of the matrices depend solely on the composite end use requirements. For example, if chemical resistance together with elevated temperature resistance is needed for a composite material, then thermoset matrices are preferred. If a composite material with high damage tolerance, remoldability and recyclability is needed, then thermoplastics are preferred[18].

LDPE is a first grade of PE thermoplastic and made of monomer ethylene (Figure 2) by free radical polymerization. PE is most widely used plastic in industries and daily appliances, 50-55% will be PE from that 30% will be LDPE. The polymer has softening temperature range from 80 ^{0}C to 95 ^{0}C, melting point at the range of 110 ^{0}C -180 ^{0}C and it is easy to process at melting point region[18]. Crystalline regions provide rigidity at high temperatures, but the amorphous regions provide flexibility and high impact strength. The PEs are classified according to their densities which is the result of different degrees of crystallinity. LDPE is having crystallinity of 40-50%, density 0.915-0.935 g cm-3.

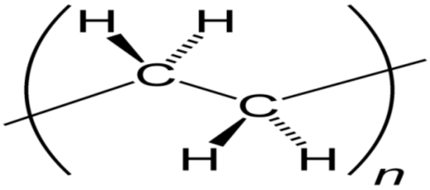

Figure 2. Chemical structure of PE

Advantages of LDPE

- High impact strength at low temperatures
- Low cost
- Can be used in all conventional methods

Disadvantages of LDPE

- Low strength
- High thermal expansion

Applications of LDPE

- Packaging films, chemical resistant lids, containers, pipes.

Mechanical and thermal properties of LDPE composites

- Tensile strength and flexural strength both increased significantly with increasing the filler (glass fiber, carbon fiber or talc) depends on concentration of filler content Compared to that of neat LDPE.
- The filler addition to LDPE composites will decrease the elongation at break point.
- The right proportion of filler with selective additives can increase the stiffness and toughness of composite by creating better bond between matrix and filler.
- Basically LDPE is a low heat conductivity and low electric conductivity. Thermal conductivity can be increased by the addition of fillers like glass fiber, carbon fiber etc.,
- The addition of fillers can also improves the dimensional stability by increasing cooling rate of composite compare to neat LDPE.

1.3. Fillers

1.3.1. Glass Fiber filled Composites

Glass fiber (GF)-reinforced composites are considerably shows good mechanical properties. Various high-performance composite materials are available today with fillers like carbon fiber, aramid fiber and basalt fiber. The aim of GF is to increase mechanical properties matrix material with uniform distribution of filler particles[6]. GF are also a easily available and less price material compared to carbon fiber, aramid, and Kevlar fibers. GF shows excellent mechanical properties with thermoset resins. The industrial use of GF is broad compare to other fillers, because of its strength to weight ratio and easy process ability, in the other side addition of GF will avoid defects like shrinkage and warpage.

Glass Fiber is the oldest and most familiar, high-performance fiber. GFs are made from fine fibers of glass strands which is light in weight and strong. The continuous strands may be converted into various other forms suitable for open-mold and other thermoset applications, such as rovings, woven rovings, fabrics, and mats. They may also be cut to specific lengths to produce chopped strands, or milled to finer sizes for a variety of thermoplastic and thermoset applications. Although the strength and stiffness properties are somewhat lesser than carbon fiber, it will use because of its availability and lower cost. Even it is easy to compound and recyclability is more with uncolored compositions[20]. GF's are now dominant due to their low cost and comparatively better mechanical properties. GF's are produced when thin strands of silica-based or other formulations of glass are extruded into many fibers with small diameters appropriate for textile processing[22]. This has good insulation properties and can maintain these properties up to 815 ^{0}C. E-GFs (Figure 3) are often preferred as reinforcement in a polymer matrix due to their higher strain to failure, better impact resistance, good fatigue life and good corrosion resistance in most common environments. End uses for regular fiber glass are mats, insulation, reinforcement, sound absorption, heat-resistant fabrics, corrosion-resistant fabrics, and high-strength fabrics. Corrugated fiber-glass panels are also widely used for outdoor canopy or greenhouse construction

Figure 3. E-grade GF

The fibers can be further characterized by their physical and chemical properties, which are governed primarily by the composition of the glass. There are several glass fiber types, with different chemical compositions for different applications. They include:
A-glass; the most common type of glass for use in windows, bottles, etc., but not often used in composites due to its poor moisture resistance.
C-glass; high chemical resistance glass used for applications requiring corrosion resistance.
D-glass; glass with improved dielectric strength and lower density.
E-glass; a multi-purpose borosilicate type and the most commonly used glass for fiber reinforcement.
S-glass; a magnesia-alumina-silicate composition with an extra high strength-to weight ratio, more expensive than E-glass and used primarily for military and aerospace applications [10]**.**

1.3.2. Polyester Fiber composites

Polyesters are one of the most classes of polymer in use today. In their simplest form, polyesters are produced by the polycondensation of glycol with a difunctional carboxylic acid. Hundreds of polyester exists due to myriad and combinations of dialcohols and diacids. During the last decades, there has been a tremendous growth in the use of composite materials in various fields of application, ranging from sporting goods to structural materials for the automotive and aerospace industries [11] . Most polyesters are used in the manufacture of PET bottles and cushioning obectives which leads to high recycling rate of polyester. Poly (ethylene terephthalate) (PET) is a semi-crystalline thermoplastic polyester widely used in the manufacture of apparel fibers, disposable soft-drink bottles, photographic films, etc. PEFs are the most used synthetic textile fibers[21] . According to the most recently released Figures 70% of textile fibers used in industries are PEFs, 60% of fibers and 30% of bottles are made from virgin PE terephthalate (PET). The recycled PEFs used in industries are from waste PET bottles, and using recycled polyester will prevent land filling of PET bottles [17]. Use of polyester fibers (PEF) in composites as filler is very rare, because of melting point lies at range of 249 to 280^0 C [7]. A process for producing PEF comprising the steps of: conducting an esterification reaction of a dicarboxylic acid composition comprising at least two kinds of dicarboxylic acid compounds with glycol; conducting a polycondensation reaction of the oligomer produced by the esterification; and melt spinning and drawing the polyester chip [14]. Wherein the PEF has tensile tenacity of 7.5 g/denier or more, and breaking elongation of 13% or more and [14] .

Figure 4. Polyester Fiber (PEF)

Characteristics of Polyester

- Strong
- Abrasion resistant
- Quick drying
- Chemical resistant
- Low shrinkage factor

1.3.3. Limestone ash and Oil shale Composites

Petroleum is a nonrenewable natural resource that is currently being rapidly consumed and as a result world petroleum reserves are being depleted at an unacceptably high rate. Thus, the development of alternate fuel sources as a substitute for petroleum is a vital necessity. One of the most promising new sources is liquid fuel from oil shale kerogen. This fuel is termed “shale oil”. In Australia, the United States of America, France, China, Russia, and Brazil, oil shale have been the source of products similar to those obtained from petroleum for many years. Shale oil technology and research have a long history and have experienced a series of fluctuations in research effort in development and achievement with time since shale oil was first used either as a fuel or lubricant[9]. Oil shale was defined by Gavin as “compact laminated rock of sedimentary origin, yielding over 33 % of ash and containing organic matter that yields oil when distilled, but not appreciably when extracted with the ordinary solvents for petroleum". Speight defines shale as "the oil produced from an oil shale on heating". As with many other fossil fuels, oil shales are complex mixtures of organic and inorganic materials and vary widely in their composition and properties.

Oil shale ash is a by-product generated by the combustion of oil shale that is collected by different means to control air pollution. It is mainly used as a replacement of Portland cement[10]. It is estimated by a report of C&EN in December 2009 that 7% of global carbon dioxide and greenhouse gases are emitted by cement production which can be reduced by the use of fly ash. Oil shale ash is a finely grained, black powdery particulate type material. It is mainly composed of more than 70% unburned carbon substances. As these materials are often associated with CO2 point source emissions and tend to be chemically more unstable than geologically derived minerals[11], they require a lower degree of pre-treatment and less energy-intensive operating conditions to enhance carbonation yields. Furthermore, after stabilization by accelerated carbonation, the leaching behavior of alkaline waste materials such as municipal solid waste incinerator, air pollution control residues and ashes from oil shale power plants is improved. This allows the usage of stabilized waste materials in civil engineering applications or for safer final disposal to landfill.

Oil shale is organic compound in the form of rock which contains kerogen (organic chemical compound). In Estonia 85% of electricity production is done by using 70% of mined oil shale, 27% for shale oil and 3% for thermal energy, cement production. Oil shale produces about 70% ordinary waste. A solid residue of oil shale in the form “oil shale ash and spent shale” is used for the production of cement. The mining and processing of about one billion tonnes of oil shale in Estonia has created about 360-370 million tonnes of solid waste. Combustion ashes are the largest component (200 million tonnes)[25]

Fly ash has also been used in the development of functionally graded polymer composites to obtain gradient in density, hardness and electrical properties, and it was earlier used in various polymers, such as polyetheretherketone (PEK) , polypropylene (PP) and high density polyethylene (HDPE). The fine fly ash particles improve dielectric properties, rigidity and heat deflection temperature of polymers. Moreover the use of ultrafine particles <10 µm (Micron), significantly affects the properties of composites due to the large specific surface of particles. The fly ash is a by-product obtained by combustion of coal in thermal power plants.

It is driven away from the boiler by the gases and extracted out from them by mechanical collectors or electrostatic precipitators or a combination of both. The incorporating fly ash particles in polymers can improve the shortcomings associated with polymers such as high shrinkage, low stiffness, poor dimensional stability, and low flame resistance, etc. The chemical stability of the blends was also reported improved on adding fly ash in polypropylene/polycarbonate blend. A few commercial polymer-based products contain fly ash; for example: floor tiles, sinks, automobile body, furniture, textile bobbins, flame resistant electronic products etc.,[26] [27].

Limestone is a sedimentary rock consists of calcium carbonate ($CaCO_3$) in the form of calcite and aragonite minerals. Estonia is using limestone for construction purpose and in manufacturing of cement. As with most materials, thermal conductivity is found to be a function of density, while also being dependent on whether the aggregate source is siliceous or limestone.

1.3.4. Mineral wool Fiber composites

Mineral wool fiber (MF) has been manufactured for a good 100 years. MFs are manufactured by melting and centrifuging suitable mineral raw materials, such as stone, glass, ceramics, slag or the like. MF slabs or panels conventionally contain a binder in addition to the MFs [12].

The construction and demolition (C&D) industry has been identified as a major source of waste, varying between 13% and 40% of the total solid waste generated, depending on the country. Only fragmented information is available about the recycling rates of C&D waste. It has been estimated that about 46% of C&D waste generated in the European countries is recycled [12]. In the US, the recycling rate is estimated to be 20–30%. Environmental regulations and laws concerning the recycling of C&D waste have been implemented in many countries, and the European Union has set an binding legislation, according to which 70% of non-hazardous C&D waste has to be prepared for re-use, recycled or recovered by 2020.

Increasing the rate of recycling C&D waste has multiple benefits. A direct effect of increased re-use would be a reduced amount of waste being disposed to legal and illegal landfill sites. The shortage of land for waste disposal and the rising landfilling costs increase the attractiveness of re-using materials instead of disposing them to landfills [13]. There are environment benefits from landfilled C&D waste decrease. Natural resources are conserved when C&D waste materials are used to replace virgin raw materials.

The utilization of wool waste cold make major role in improving recycling percentage, and it can be done by using it as filler with polymer composite materials in order to change mechanical properties and reduce weight ratio. These inorganic fillers have shown potential in improving the mechanical, fire retardant and thermal properties of wood plastic composite (WPC). Inorganic fillers are also cheaper than polymers, and therefore the raw material costs of WPC can decrease when polymers are replaced with inorganic fillers.

MF treated in the temperature range of 20°C to 600°C has greater strength than E-GF thread. Mechanical and thermal properties of continuous MFs depend not only on the method of production, but also significantly on the chemistry and nature of the fiber surface.

The chemical composition of mineral wool can vary depending on whether it is glass wool or rock wool. The main component in both rock and glass wool is Silicon dioxide (SiO_2). Glass wool has a slightly higher SiO_2 content, while rock wool contains more ferric oxide (Fe_2O_3), giving it a darker color and higher heat resistance [12]. The chemical compositions of mineral wool are rather close to that of GFs which are used as filler in composites. Pure SiO_2, the main component in mineral wool, is also used as filler in composites.

Minerals fibers are made of GF, rock and slag wool, cellulose, and natural fibers to rigid foam boards (Figure 5) to sleek foils. MF acts as heat resistant.

Continuous MFs are produced without further correction of composition by additives from selected kinds of eruptive rocks such as basalt, diabase, porphyrite, andesite, gabbro, etc. They differ in the composition from short MFs and glass based on them has different thermal-viscous constants, low tendency towards crystallization that steadily occurred towards the end of fibirising process (high-speed spinning heads somewhat like the process used to produce cotton candy) region greater than 50°C, etc. The reason for this is the difference in the chemical and mineralogical composition, especially in iron oxide content. It puts continuous MFs in between short MFs and continuous GFs in terms of fiber forming properties. Moreover, MF properties depend on thermal and on chemical and phase composition of the initial mineral [24].

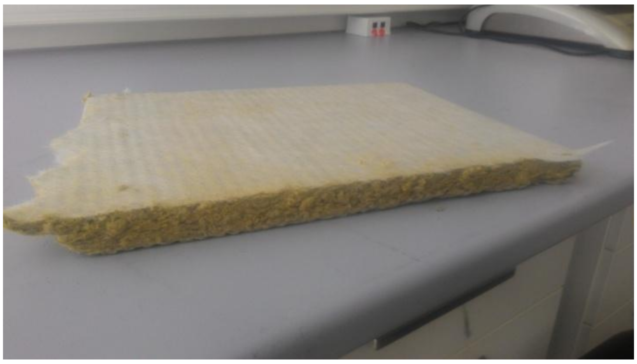

Figure 5. Mineral Fiber board

1.4. Compatibilizers for Polyolefin Composite

Compatibilizer is defined as organic or inorganic compound used to create bond between two dissimilar materials. The selection of coupling agent is based on matrix and raw polymer. Because, surface properties are different for both raw polymer and filler material. Sometimes need surface activators to activate the surface properties of filler.

1.4.1. Maleic Anhydride

Maleic Anhydride (MA) is a organic compound with a structure of $C_4H_2O_3$ as shown in Figure 6, and will be in the form of colorless or white solid with acidic odor having melting point at 210 ^{0}C [28]. The functional polymers product line includes modified ethylene acrylate carbon monoxide terpolymers, ethylene vinyl acetates (EVAs), PEs, metallocenePEs, ethylene propylene rubbers and polypropylene. MA covalently linked with main polymer and filler with modifies the functional properties of composite [28].

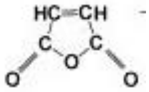

Figure 6. Structure of Maleic Anhydride

The MA groups are grafted to polymers like polypropylene to form MA grafted polypropylene (MAPP). MAPP has two functional domains, i.e anhydride carbonyl groups that interact with cellulose hydroxyl groups, and the hydrophobic group which interacts with the matrix (Figure 7). MAPP then forms a sort of a bridge of crosslinks between the polymer matrix and the natural fibre interface. polyolefins also seem to improve the interfacial adhesion between the filler and the polymer matrix due to functional groups on the compatibilizing polymer’s backbone. These functional groups will form hydrogen bonds with cellulose hydroxyl groups [18] **[**29**]** .

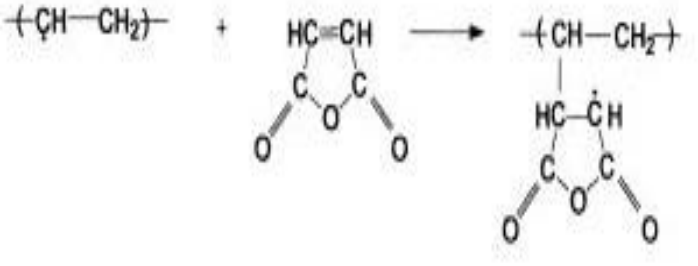

Figure 7. MA grafting reaction with LDPE

1.4.2. Silane coupling agent

Vinyl silanes is an inorganic chemical, may also refer generically to vinyl substituted silanes, such as vinyltrimethoxysilane(VTMS) with a structure of $H_2C{=}CHSi(OCH_3)_3$ as shown in Figure 8. or vinyltriethoxysilane.

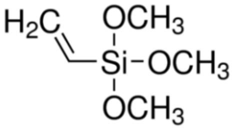

Figure 8. Structure of VTMS

The coupling mechanism of organo silanes depends on a stable link between the organo functional group (Y), and hydrolysable group (X) in the compounds of structure X_3SiRY. The organo functional groups are chosen for reactivity or compatibility with polymer, while the hydrolysable groups are merely intermediates in the formation of silanol groups for bonding to mineral surface[30].

Silane coupling agents have a large number of functional groups which can be tailored as a function of the matrix to be used, and ensures, at least, a good compatibility between the reinforcing element and the polymer matrix or even covalent bonds between them [31].

Alkoxy groups associated with silane coupling agents prior to utilization can be hydrolyzed off thereby liberating the corresponding alcohols in the presence of water and generating reactive silanol groups. The hydrolysis of trimethoxysilane producing methanol and the number of methoxy groups will determine the amount of water used to fully hydrolyze them and influence the adhesion between silanes and filler. Di- and tri-methoxy silanes produce stronger adhesion [32].

The grafting of silane compatibilizer onto a thermoplastic is by using inhibitor such as benzoyl peroxide (BPO), at elevated temperatures bezoyls peroxide will decomposes, generating oxy radicals. The oxy radicals not only have the potential to abstract hydrogen from the backbone of thermoplastic molecule or filler material (Figure 9), but can also add to vinyl double bonds of vinyl silanes, producing vinyl radicals. The vinyl free electron may either combine with each other (homoploymerization) or attack each other molecules in a similar fashion to propagate the free radical reaction. Accordingly the radical reaction would ultimately result in grafting of vinyl silane onto thermoplastic matrices has two options: one approach is graft vinyl silanes onto matrices with resulting copolymer being used a coupling agent to bond fiber material and matrices; the other way is to treat the fiber with vinyl silane solution and graft reaction appears in the thermal compounding process of vinyl silane treated fibers and matrices [32].

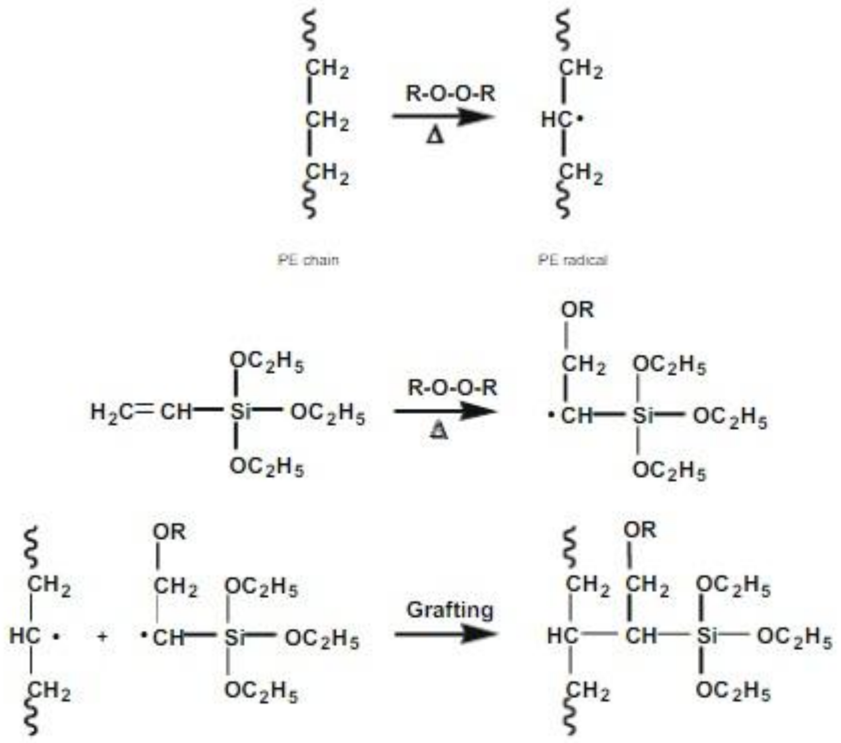

Figure 9. This shows the radical grafting of VTMS onto PE matrix [28].

1.5. Aims and Objectives

1. To find out the effect of organic and inorganic fillers on the mechanical properties of LDPE composites and to find suitable compatibilizer for the fillers.
2. To increase the Flexural strength of LDPE composite with the help on organic filler (PEF) and inorganic filler wastes such as, MF, oil shale ash, limestone ash.
3. To bring down the cooling speed of LDPE composite with fillers like MF, oil shale ash and limestone ash.

Chapter 2. Experimental Work

2.1. Materials

2.1.1. Low Density Polyethylene

Low density polyethylene powder was the matrix polymer used in this work, which was easy for mixing with fillers. LDPE has advantages of excellent moisture, chemical, and electrical resistance. Its principal disadvantage is poor mechanical strength, unless it has a little help from reinforcement. LDPE has high viscosity melt flow and having density of 0.98 $\frac{g}{cm^3}$.

2.1.2. Organic Filler

2.1.2.1. Polyester Fiber

Polyester fiber used was in the form of waste material (Figure. 4) and it was difficult to compound with LDPE powder, one brought down PEF into particle size of 2 mm by using cutting mill equipped with mesh of 2 mm diameter. PEF have properties like strong fiber strength, resistant to stretching and shrinking, resistant to most chemicals, quick drying and resilient when wet or dry. PEF has specific weigh t of 1.22 to 1.38 g/cm^3, moisture content varies from 0.2% to 0.5% and fiber melts at a temperature range of 249 ^{0}C to 288 ^{0}C. On the other hand PEF was also used with 5% of VTMS and MA compatibilizers. The process of mixing PEF with MA was easy compare to VTMS, because VTMS solution was in liquid form and hot air woven was used to evaporate water out of VTMS solution+ PEF.

2.1.3. Inorganic Fillers

2.1.3.1. Glass Fiber

E-28 glass fiber was the grade of GF used for this work and it was in chopped strand form as shown in Figure. 3. One used 10% GF in compounding ratio, which was easy to mix with LDPE powder during compounding process. E-grade GF is useful because of their high ratio of surface area to weight. However, the increased surface area makes them much more susceptible to chemical attack, glass fiber make good thermal insulation.

2.1.3.2. Mineral Fiber

Mineral fiber used in this work was from insulation boards, which contains wool and glass chopped strands in it (Figure. 5), was brought down to the form of small fibers having particle size of 2 mm, which was prepared by using cutting mill and one used 10% of it composite preparation. MF has properties like fire resistance, high pressure bearing, high elastic material and maximum working temperature of 240 ^{0}C.

2.1.3.3. Fly Ash

Oil shale ash and limestone ash were the fly ash used ,was in the form of fine powder of particle size less than 50 µm. Oil shale ashes used at 10% of ratio, mixed easily with LDPE powder. Processability during compounding done at 175 ^{0}C because of filler particle size and high viscosity, and screw speed maintained in between 40 to 50 rpm depends on melt flow.

2.1.4. Compatibilizers

2.1.4.1. Maleic Anhydride

In this work maleic anhydride used a coupling agent in the trade name of Fusabond MB226D (MA graft) by the company called DuPont, this coupling agent was in the form of pellets and one brought down into powder particles of 2 mm by using cutting mill, and 5% of MA was good with the combination of PE and glass fiber. So, one used 5% of MA coupling agent with polyester filler also.

2.1.4.2. Vinyltrimethoxy Silane

In this work vinyltrimethoxy silane was used as a coupling agent from the company called "Sigmaaldrich", which was in liquid form. For PEF wet out process, one prepared a VTMS solution of 5% concentration by diluting it with water.

Table 1. List of raw materials used in experiment

Matrix polymer	**Fillers 10%**		**Compatibilizer 5%**
	organic	**Inorganic**	
Low Density Polyethylene (LDPE)	Polyester Fiber (PEF)	Glass Fiber (GF)	Maleic Anhydride (MA)
		Mineral Fiber (MF)	
		Oil shale Ash	Vinyltrimethoxy Silane (VTMS)
		Limestone Ash	

Table 2. List of composite materials prepared in this work

List of composite materials
LDPE + 10% GF
LDPE + 10% GF + 5% MA
LDPE + 10% PEF
LDPE + 10% PEFF + 5% MA
LDPE + 10% PEFF + 5% VTMS
LDPE + 10% MF
LDPE + 10% Oil shale Ash
LDPE + 10% Limestone shale Ash

2.2. Manufacturing Methods

The process begins with bring down filler material in to proper particle size which can be mix with LDPE powder and should be feedable through the compounding machine hopper. The mixture of plastic base material (LDPE), fillers and coupling agents are mechanically mixed in a drum which placed on two roll motor. Later the mixture is transferred into a twin screw compounder and they final output of composite material in the form of pellets were subjected to injection molding machine to get the testing samples (tensile test, flexural test and Cooling rate analysis).

2.2.1. Cutting Mill

Cutting mills are suitable for the grinding of soft, medium-hard, elastic, fibrous, and heterogeneous mixes of products. The SM 100 is the budget-priced basic model among the RETSCH cutting mills. With its strong 1.5 kW drive and 1,500 rpm rotor speed the mill is particularly suitable for routine applications. Cleaning is made particularly easy. In combination with the wide choice of bottom sieves, hoppers and collecting vessels, the mill can be easily adapted to varying application requirements. The SM 100 mounted to base frame [33].

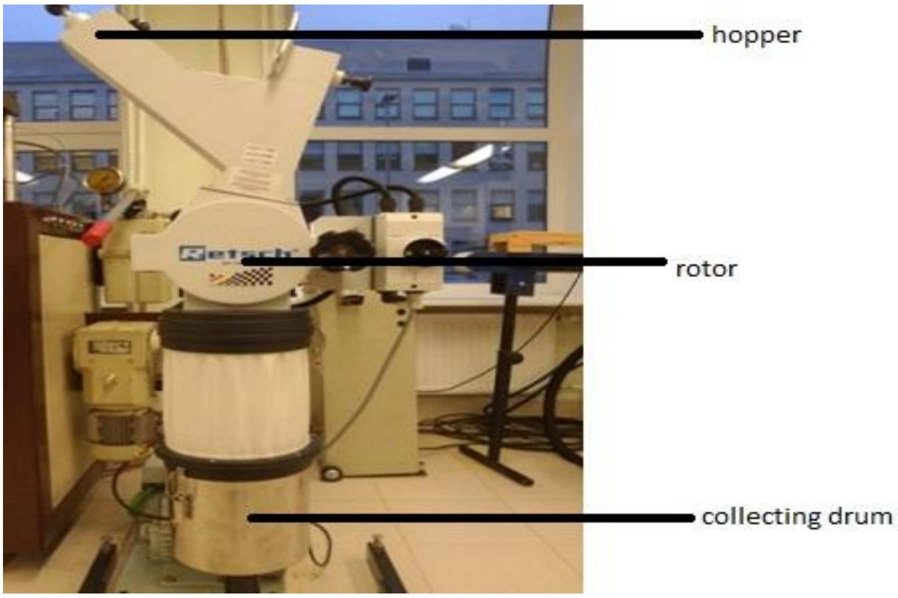

Figure 10. Retsch SM100 - Cutting mill

As the provided reinforcing materials were in big shape and thickness, our primary task was to bring down the filler material into smaller particle size which can mix thoroughly with LDPE powder. “Retsch SM100” (Figure 10) was the cutting mill used to bring down particle size of filler to 2 mm through mesh.

Advantages

- Powered enough to break particles
- Easy operation and particle size varies with different sieve sizes (0.25mm to 20mm)
- Wide range of accessories including various hoppers, collection systems, rotors and sieves
- Highest safety standards

Size reduction of reinforcing material in cutting mill SM 100 takes place when the sample comes in contact between the rotor blades, and sieve mesh with a sieve size of 2mm was used. The dwelling time of the sample in the chamber is short; as soon as it is small enough to pass through the openings of the bottom sieve it is discharged and collected in the receptacle. The rotor speed of 1.500 min^{-1}ensures gentle and rapid size reduction [33].

2.2.2. Twin Screw compounder

For this work, one used twin screw compounder (Figure 11) to prepare composite material by feeding the mixture of raw materials into compounder slowly(to avoid suck-back). Before that one has to adjust the parameters (processing temperature at three different zones maintained between 165 to 185 ^{0}C, screw speed should between 40 to 50 rpm) required depends on characteristics of LDPE (melting point= 180 ^{0}C, viscos flow) [34]. The addition of filler and compatibilizer will make changes in the rheological characteristics of LDPE (GF will makes melt flow low viscos compared to addition of fly ash fillers). The added material mixture will melt and mix homogeneously with the help of counter rotating screw inside barrel, and extrude the polymer composite strand through circular die. Later the strands pass through cooling channel/water bath to harden polymer strands, before it pelletize by pelletizer [35] [36].

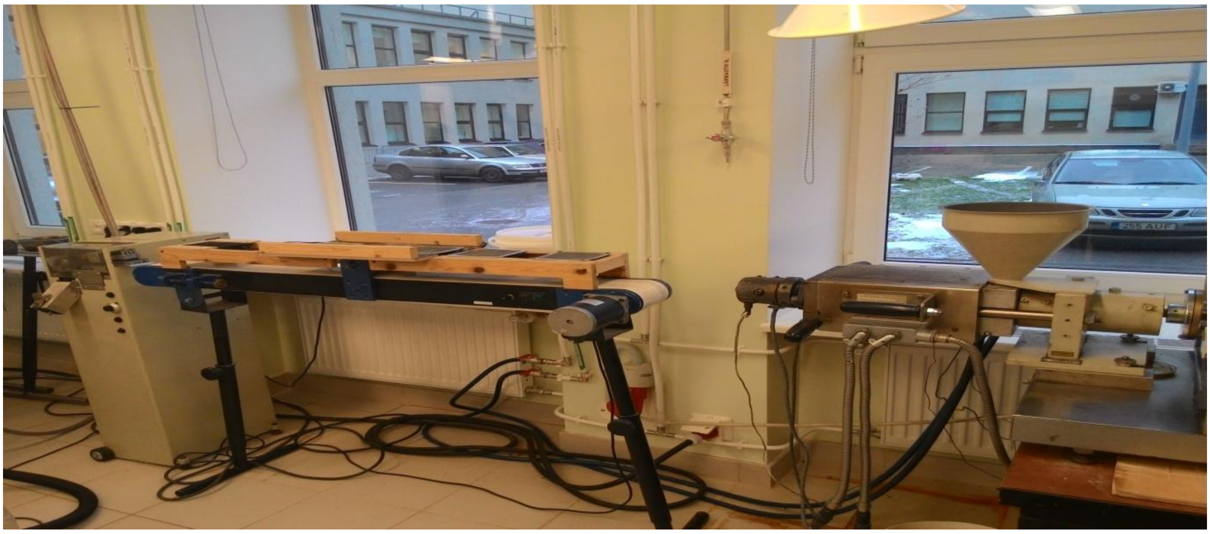

Figure 11. Twin screw compounder

2.2.3. Injection Molding Machine

In this work two different injection molding machines were used. “Battenfeld BA230E” was the one used for preparing Flexural and cooling rate test samples according to ISO 178 standard. Figure 12 Shows the injection molding machine” Battenfeld BA230E” one used in our lab and it is having a clamping force of 230 KN [38]. The parameters used in this equipment operation were (processing temperature between 165 to 185 ^{0}C, screw speed between at 60 rpm), total cycle time of 50 seconds.

Figure 12. Injection Molding Machine (Battenfeld BA230E)

Figure 13. Babyplast 610P injection molding machine

Babyplast 610P (Figure 13) injection molding machine used for producing small tensile dumbbell shaped samples according to ISO 527-2. The cycle time for this process was 35 seconds, because of small sample size[39].

2.3. Testing Methods

2.3.1. Tensile Strength Test

Tensile test, in a broad sense, is a measurement of the ability of a material to withstand forces that tend to pull it apart and to determine to what extent the material stretches before breaking and the test was done according to ISO 527-2. Tensile modulus, an indication of the relative stiffness of a material, can be determined from a stress–strain diagram. Instron 5866 was the model of Universal Testing Machine (UTM) (Figure 15) used for this test, equipped with a load cell of 10 KN which measure the load required to perform test till the failure of specimen. The specimen prepared by babyplast 610P injection molding was type 1BB under ISO 527-2 standard for tensile test, and the geometry of specimen has shown in Figure 14. Which has total length (L_3) of 30 mm, width on grip section (b_2) was 4mm initial distance between specimen grips (L) of 24mm, gauge length (L_0) of 18 mm, thickness (h) of 2 mm, and width on narrow section (b_1) was 2 mm. Testing speed for this 1BB type of tensile specimen will be 1 mm/min or 2 mm/min. one used speed of 5mm/min in this test, because it may be difficult to measure modulus on short specimens at high speed. Results obtained from small specimen are not comparable with result obtained from type 1 specimen [41].

The dumbbell shaped specimen was clamped between two fixtures, lower one is rigid/stationary fixture and upper one is movable fixture, and set the speed that upper fixture should move vertically in-order to stretch the specimen until the load drops. The test specimen is extended along its major longitudinal axis at constant Speed until the specimen fractures or until the stress (load) or the strain (elongation) reaches some predetermined value. During this procedure the load sustained by the specimen and the elongation are measured[40].

Where, L = initial distance between grips = 24 mm
L_0 = gauge length = 18 mm
h = thickness = 2 mm
L_3 = total length = 30 mm
b_2 = width on grip section = 4 mm
b_1 = width on narrow section = 2 mm

Record the forte and the corresponding values of the increase of the gauge length and of the distance between grips during the test. lt is preferable to use an automatic recording System which yields complete stress/strain curves for this Operation. Determine all relevant Stresses and strains defined in from the stress/strain curve or using other suitable means[42]

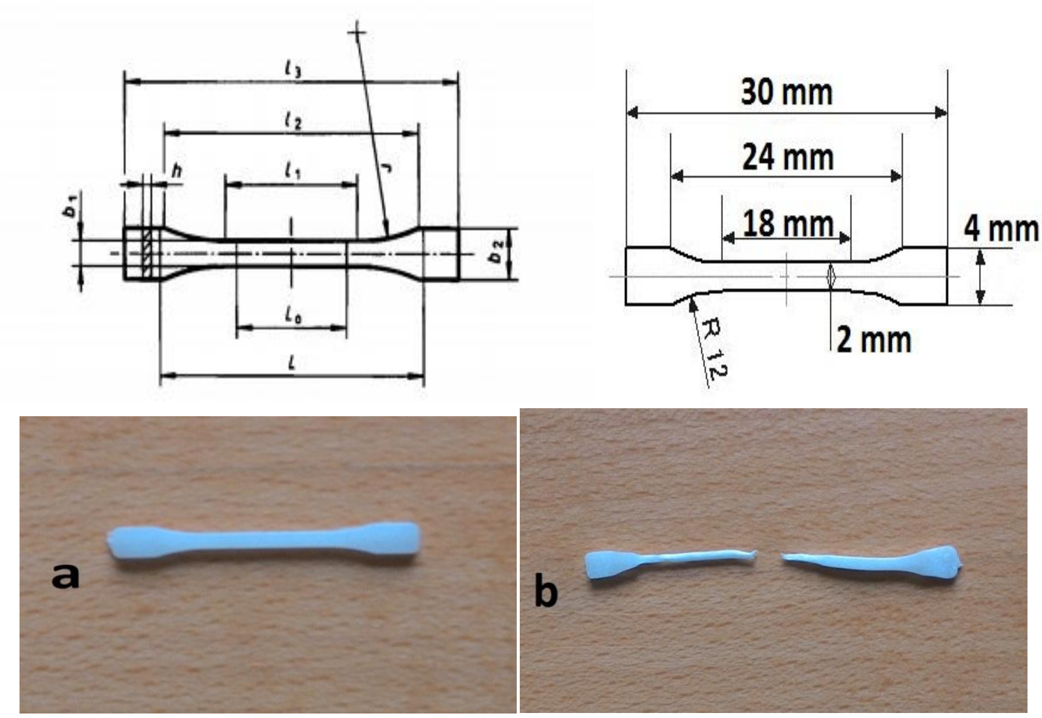

Figure 14. Geometrical representation of tensile test specimen. a: before testing, b: after testing (after failure)

.

Figure 15. Universal Testing Machine (UTM) (Instron 5866)

Calculations:

Stress calculation

$$\mathrm{\sigma = \frac{F}{A}}$$

Where,

$\mathrm{\sigma = tensile\ stress\ value\ (MPa)}$

$\mathrm{F = Force\ or\ Load\ (N)}$

$\mathrm{A = Cross - sectional\ area\ of\ the\ specimen\ (mm^2)}$ [41]

Strain calculation

$$\mathrm{\varepsilon = \frac{\Delta L}{L}}$$

$$\mathrm{\varepsilon_t(\%) = 100\ X \frac{\Delta L}{L}}$$

Where,

$\mathrm{\varepsilon = strain\ value\ expressed\ as\ dimensionless\ ratio\ or\ in\ percentage(\%).}$

$\mathrm{\varepsilon_t}$ = Nominal tensile strain, expressed as a dimensionless ratio or percentage (%).

L= Initial distance between grips, (mm).

$\mathrm{\Delta L = increase\ of\ distance\ between\ grips, (mm).}$ [41]

Modulus calculation

$$\mathrm{E_t\ = \frac{\sigma_2 - \sigma_2}{\varepsilon_2 - \varepsilon_1}}$$

Where,

$\mathrm{E_t\ = young's\ modulus\ of\ elasticity\ (MPa)}$

$\mathrm{\sigma_2 = stress\ (MPa), measured\ at\ the\ strain\ value\ \varepsilon 1 = 0.0005}$

$\mathrm{\sigma_2 = stress\ (MPa), measured\ at\ strain\ value\ \varepsilon 2 = 0.0025}$ [41]

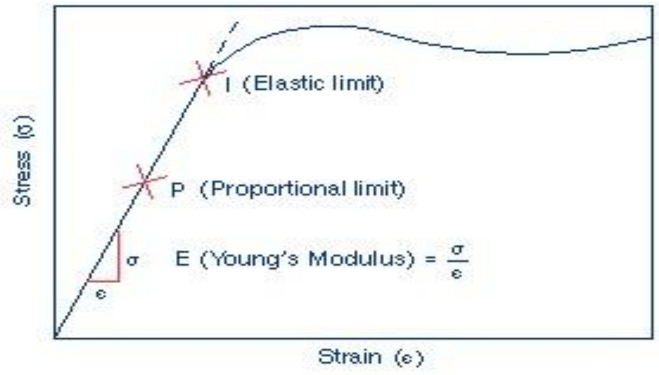

Figure 16. Graphical representation of stress/strain curve to find young's modulus

Stress-Strain Diagrams

Stress-strain diagrams were made from each tensile test. The stress-strain diagram from the tensile test is composed of measurements from the extensometer, and strain gages. A typical stress –strain can be seen from Figure 16.The samples exhibited linear-elastic stress strain behavior. This behavior is consistent until failure.

2.3.2. Three-point bending testing

Three- point bending test will be carried out with Instron 5866 according to ISO 178. For bending strength testing thickness (h=4 mm), and width (10 mm) of sample at middle section was measured. Test speed is set to 20 mm/min[43]. Length of span between supports (L) is set to 60 mm (see Fig. 17). Tests are performed and flexural stress is calculated using the following equation:

$$\sigma_f = \frac{3FL}{2bh^2}$$

Where,

$\sigma_f =$ Flexural-stress parameter in question (MPa), F - applied force (N), L - span (mm), b - width of the specimen (mm), h - thickness of the specimen (mm).

Flexural strain is calculated using the following equation:

$$\varepsilon_f = \frac{600sh}{L^2}$$

Where,

ε_f = Flexural strain parameter in question (%), s - deflection (mm), h - thickness of the test specimen (mm), L - span (mm).

Flexural modulus: To determine the flexural modulus, calculate the deflections S_1 and S_2 corresponding to the given values of the flexural strain ε_{f1} = 0,000 5 and ε_{f2} = 0,002 5 using the following equation:

$$S_i = \frac{\varepsilon_{fi} L^2}{6h}$$

$$(i = 1; 2)$$

Where,

S_i = one of the deflections (mm), ε_{fi} = corresponding flexural strain, whose values ε_{f1} and ε_{f2} are given above, L - span (mm), h - thickness of the specimen (mm) [40] [42]

Calculate the flexural modulus, Ef, expressed in megapascals, using the following equation:

$$E_f = \frac{\sigma_{f2} - \sigma_{f1}}{\varepsilon_{f2} - \varepsilon_{f1}}$$

Where,

Where, E_f - flexural modulus parameter in question (MPa), σ_{f1} - flexural stress measured at deflection S_1 (MPa), σ_{f2} - flexural stress measured at deflection S_2 (MPa).

All equations referring to flexural properties hold exactly for linear stress/strain behavior only, thus, for most plastics, they are accurate at small deflections only. The equations given may, however, be used for comparison purposes[34].With computer- aided equipment, the determination of the modulus, E_f, using two distinct stress/strain points may be replaced by a linear-regression procedure applied to the part of the curve between these two points.

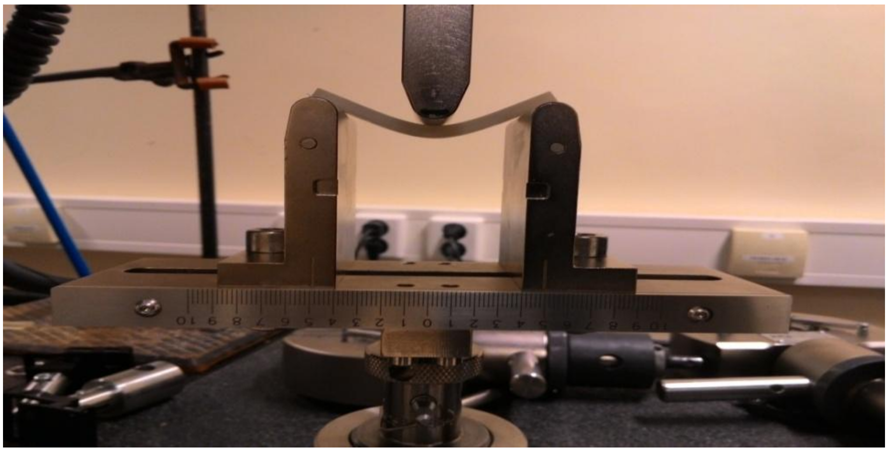

Figure 17. Three Point Load Bending Machine

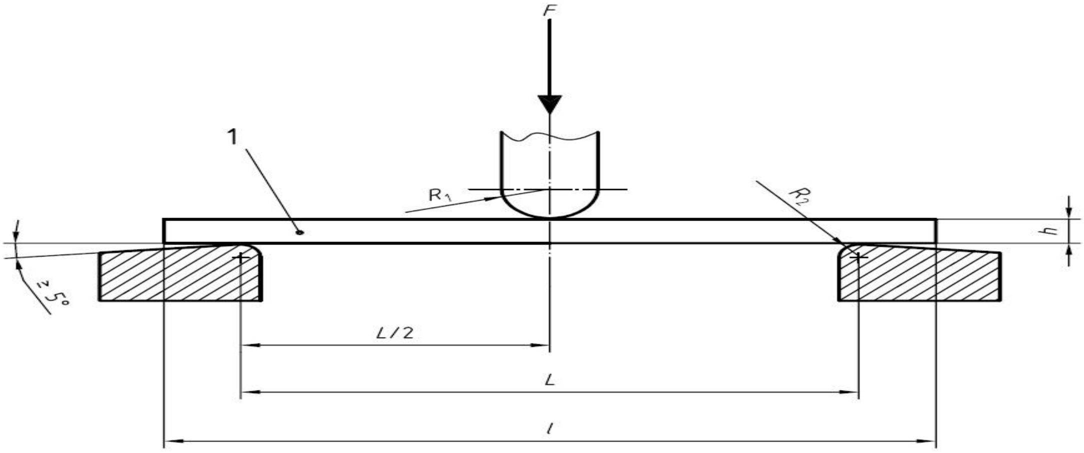

Figure 18. Position of test specimen at start of test [43]

Where,

1= Test specimen, F= Applied force, R_1=Radius of loading edge, R_2 = Radius of supports,

Thickness of specimen (h) =4 mm, total length of specimen (l) =65 mm, Length of span between supports (L) =60 mm, width= 10 mm, thickness (h) = 4mm

2.3.3. Cooling Rate Analysis

This method is similar to Differential scanning calorimeter where the sample will subject to gradual increase in temperature per minute, and later the cooling process will takes place in order to find the crystallization temperature of polymer. LDPE is a tough material with low heat conductivity, electrical conductivity, and this was the reason for using it in insulation applications of wire. When it comes to filler addition on LDPE, the fiber orientation will affect the thermal conductivity (example: the rectangular sample has more thermal stability than complicate designed sample). The factors like shrinkage and resultant stress depend on control of the cooling rate through the entire part. This can be achieved by reducing the overall cooling rate or interrupting the cooling so that the outer surface of the extruded part cools slower and allows heat from the other side to diffuse to the cooled surface. Since it’s usually necessary for extruded parts to quickly freeze the outer dimensions to conform to a certain shape, the initial cooling usually needs to be adequate to set the shape. With continued rapid cooling, there is the potential for developing internal stress in the part [40].

More recently, new ways have been investigated to enable study of polymer crystallization and. I developed a new system for analyzing the cooling speed of polymers. In this system used EL-USB data logger (Figure 19) which measures and stores over 32,000 temperature readings from K, J or T type thermocouple. The thermocouple is attached via the thermocouple socket at the base of the unit. The user can easily set up the logger and view downloaded data by plugging the module into a PC's USB port and using the supplied software. Data can then be graphed and exported to other applications. The thermocouple has been sandwich between two flexural testing specimens (PE + fillers) and place the specimen unit (which holds the thermocouple) inside the hot air oven maintained at a temperature of 190°C, where the other end of thermocouple should leave outside of oven which will connect to data logger. Leave the specimen unit inside the oven for 25-30 min for equal softening throughout the specimen area and to attain 180 to 190 ^{0}C. Set the data logger to type of thermocouple used (K-type) and speed of data record (per second), after the installation. Connect the data logger another end of thermocouple. Take out the specimen unit after 25-30 min and immediately press the data record button on device to record the time required to cool down to 40 ^{0}C from 190 ^{0}C and all changes in cooling period from melt temperature to cooling temperature [44].

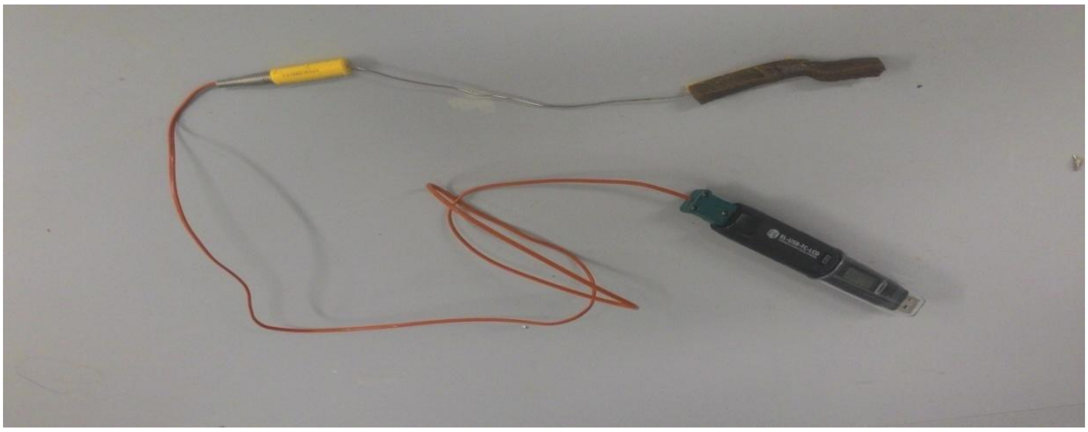

Figure 19. EL-USB logger for cooling rate analysis

Chapter 3. Results and discussion

3.1. Tensile Strength

Tensile strength is the maximum tensile stress sustained by specimen express in Newton unit. Tensile test was done by using UTM machine (Instron 5866) according to ISO 527-2 standard for specimen type 1BB which was in dumbbell shape. Test was done by using load cell of 10 KN, at the speed of 5 mm/minute.

Specimen dimension

Length between grips = 24mm, width on narrow section =2mm, thickness=2mm

Cross-sectional area of tensile specimen= width x thickness= 2 mm x 2 mm= 4 mm^2

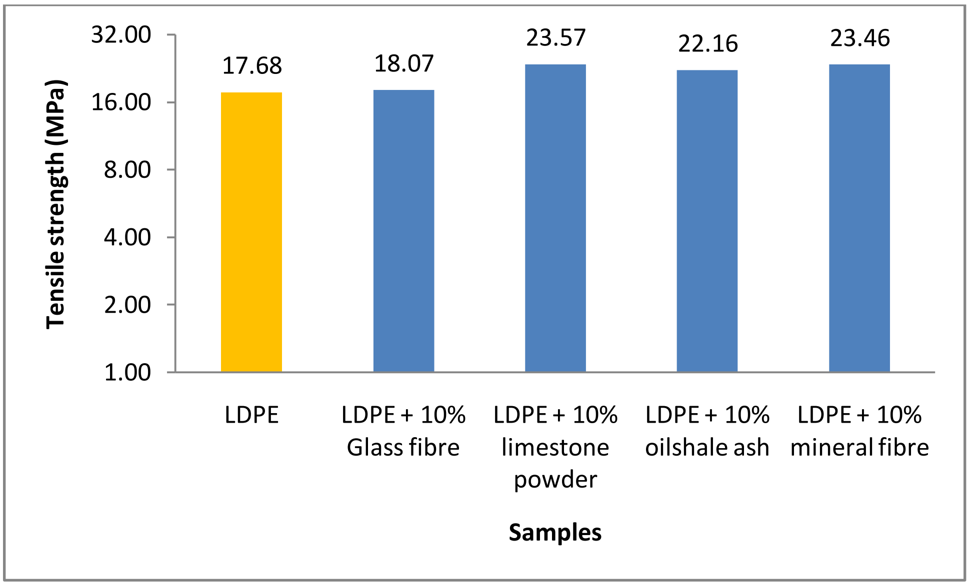

Figure 20. Effect of inorganic filler on tensile strength of LDPE

The above graph (Figure 20) shows that the effect of inorganic fillers on the tensile properties of LDPE. Fly ash fillers, MF performs as better filler material in terms of increasing tensile strength compare to tensile strength results of GF composition, and raw LDPE. This type of change in tensile strength result is due to particle size of fly ash fillers was less than 50µm, MF was 2 mm compare to the chopped strand form of glass fiber which will effect in filler stress orientation in samples.

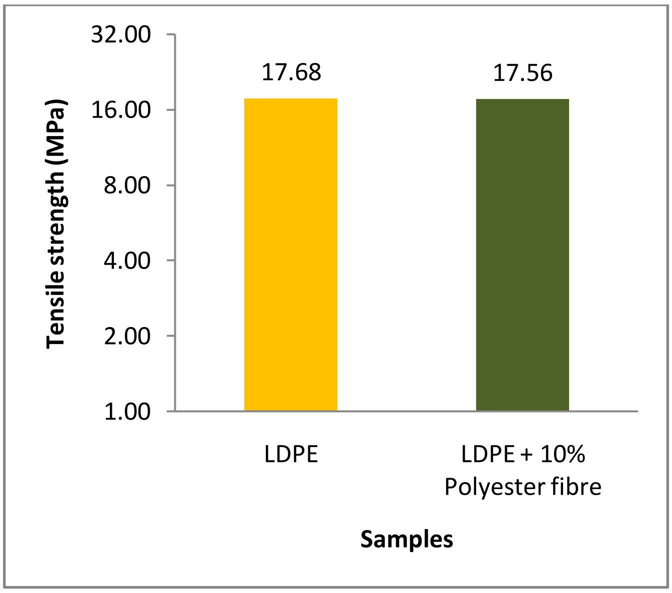

Figure 21. Effect of organic filler on tensile strength of LDPE

The effect of organic fillers on tensile properties of LDPE consists of 10% PEF, which gives same tensile results as 10% GF composition and raw LDPE material as shown in Figure 21. The particle size of PEF was 2 mm and it failed to produce similar result as MF which had same particle size.

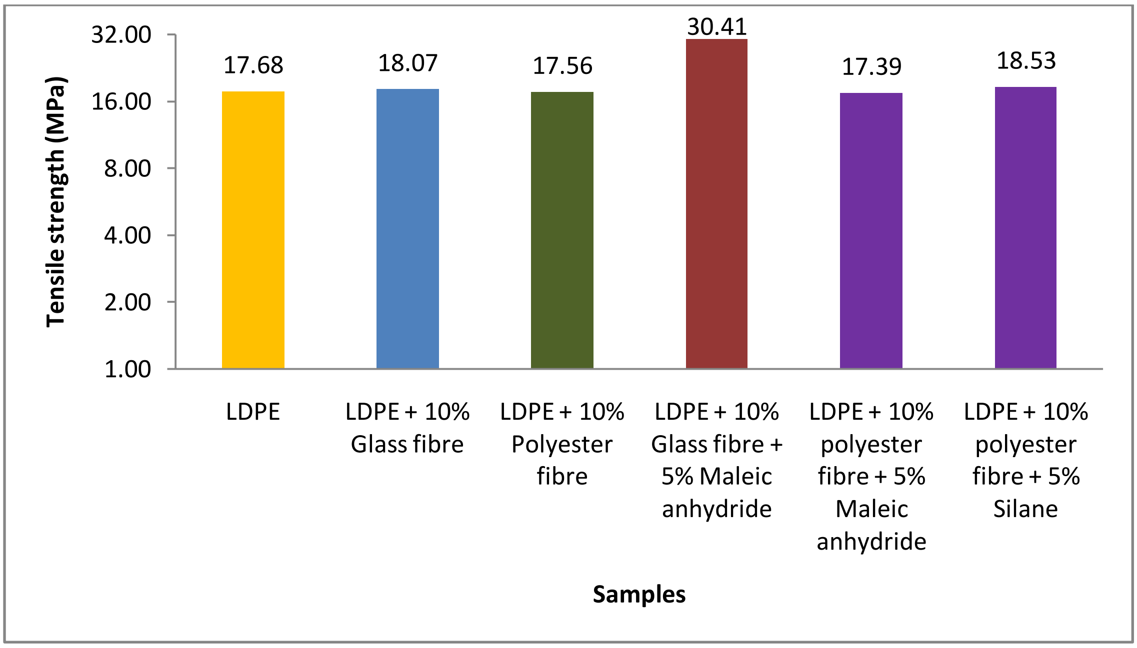

Figure 22. Effect of compatibilizer on tensile strength of LDPE

In this work tensile strength of LDPE + 10% GF+ 5% gMA has increased steadily to 30.41 MPa, compared to tensile strength of LDPE + 10% GF which has 18.07 MPa. Because, anhydride carbonyl groups that interact with PE matrix and create strong bond with GF. But, LDPE + 10% PEF gives a tensile strength result of 17.39 MPa (Figure 23), which was similar to raw LDPE. Due to the absence of surface active agents for PEF to make better bond with the use MA. But, the use of VTMS does effect in increasing the tensile strength of LDPE composite with PEF to 18.53 MPa from 17.56 MPa of LDPE + 10% PEF, and 17.68 MPa of LDPE .

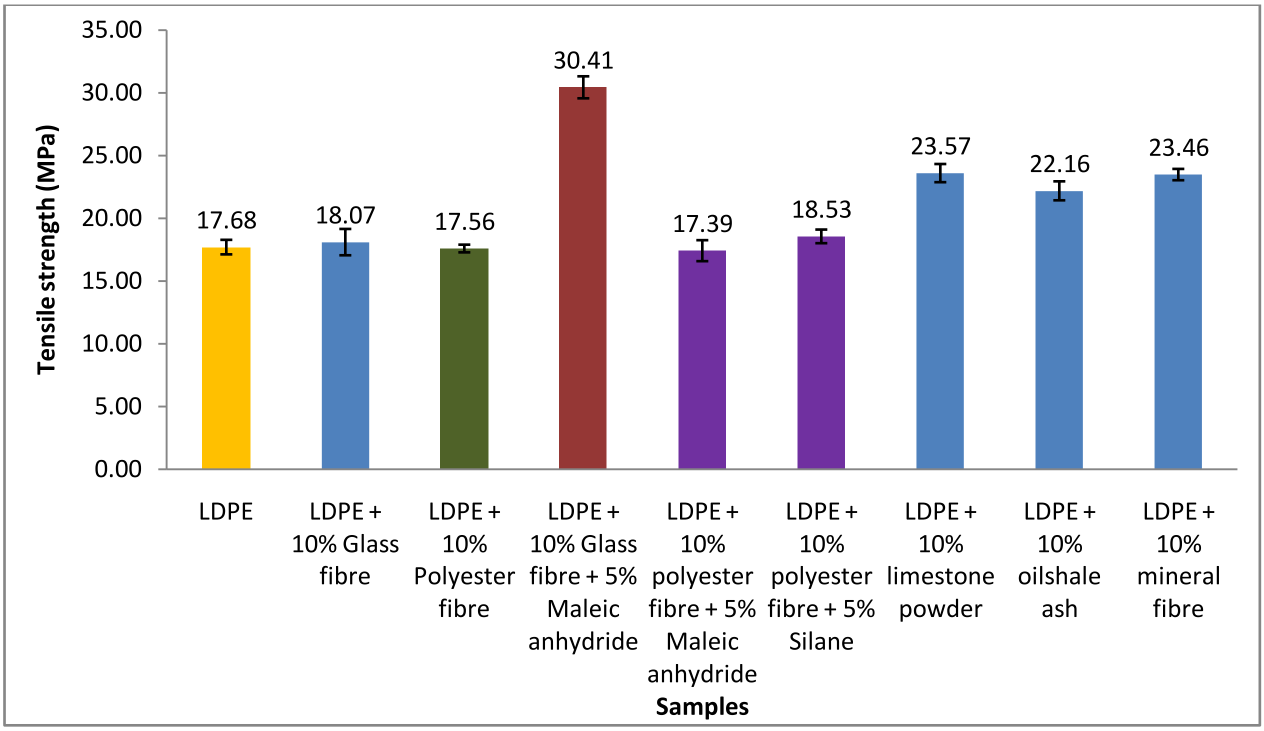

Figure 23. Effect of fillers and compatibilizer on the tensile strength of LDPE.

Tensile tests on LDPE and its composites with different filers at the ratio of 10%, and in later by the addition of coupling agents (gMA and VTMS) at the ratio of 5% were performed to obtain the modulus of elasticity, the stress strain behavior, and the ultimate tensile strength. The procedures for completion of the tensile tests are given in chapter testing methods and the results from the collected data are represented in Figure 23. A total of 8 different compositions of LDPE and its tensile specimens were tested for this phase of the testing program.

Modulus

Loads for the tensile tests were taken by the Instron 5866 UTM, and read directly by the computer. Stress-strain diagrams were made from the data collected from the all of the instrumentation. The modulus data was collected from all of the instrumentation, from the slope of these stress-strain diagrams. Due to some improper filling in specimen the results of strain varies little.

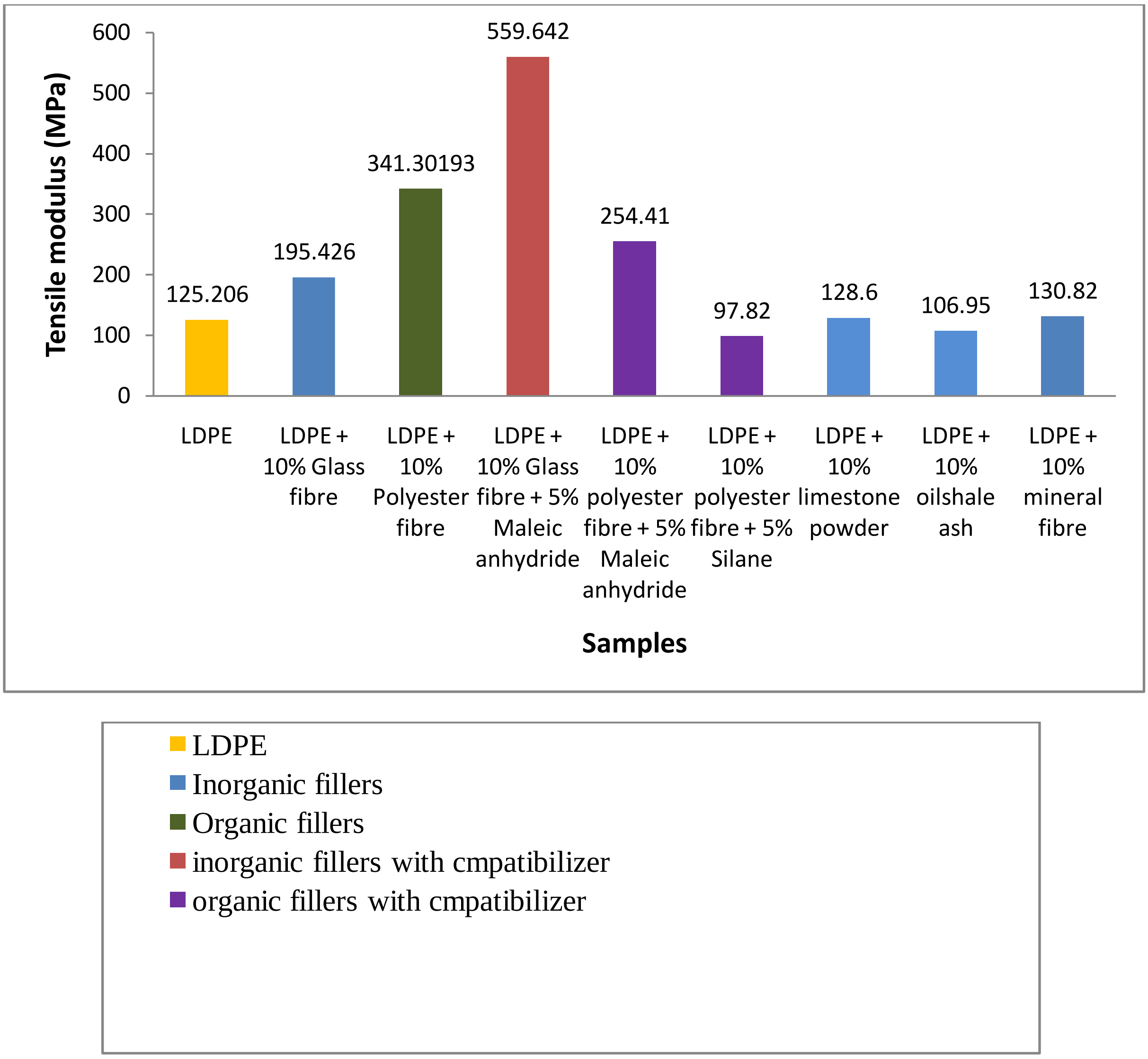

Figure 24. Effect of fillers and compatibilizer on the tensile modulus of LDPE

Upon comparison of different samples, it can be seen that LDPE + 10% GF + 5% gMA has highest average modulus of 559.64 MPa (because of high tensile strength and less elastic) with an increase of 364.22 MPa by comparision to LDPE + 10% GF which has 195.42 MPa . LDPE + 10% PEF + 5% VTMS had the lowest average modulus at 97.42 MPa with the comparison of LDPE + 10% PEF had 341.30 MPa. LDPE + 10% PEF + 5% VTMS composition has provide different lower average data compare to LDPE + 10% PEF composition average data, because of non cooperation of silane coupling agent with PEF leads to lower result. The compositions like MF and limestone ash produce almost a similar modulus such as 130.82 and 128.60 MPa (Figure 24). Though oil shale ash failed to produce a significant modulus near to limestone ash. But, capable of produce high modulus than only GF composition.

3.2. Flexural strength Test

Flexural strength is the measurement of maximum flexural stress sustained by the specimen during bending test, and test was done according to ISO 178 standard at speed of 20 mm/minute.

Specimen dimension

Length=60mm, width=10mm, thickness=4mm

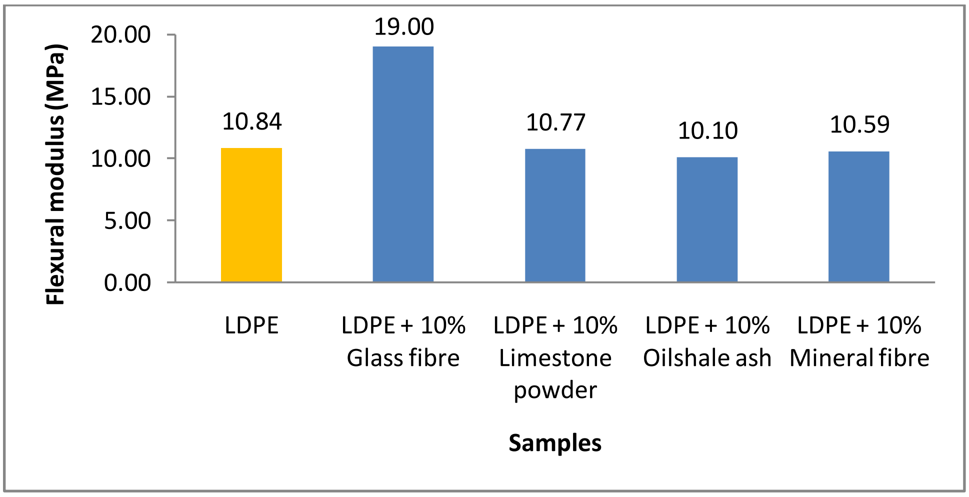

Figure 25. Effect of inorganic filler on flexural properties of LDPE

This bar graph (Figure 25) shows the effect of inorganic fillers on flexural strength of LDPE. Glass fiber performs well compatibility with LDPE than MF, which also has some glass fiber content in it. But, the GF alone was able to produce almost twice the flexural Strength value of mineral fiber and fly ash with LDPE composite. So, in this category of inorganic fillers glass fiber gave good flexural property with LDPE. So, powder or ash form of filler failed to bring any changes in flexural properties of LDPE compare to fiber form of GF, and hence fiber provide better bonding with LDPE material.

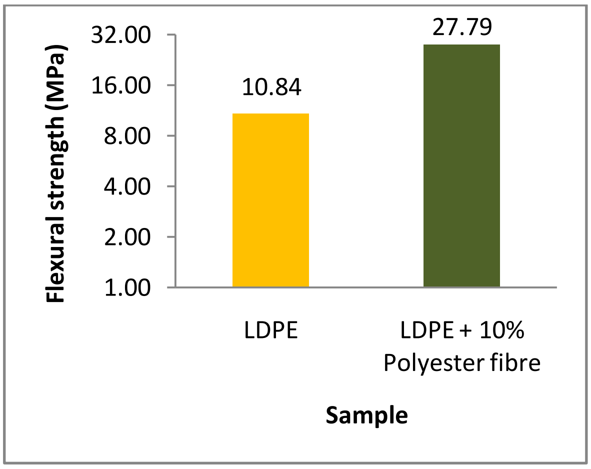

Figure 26. Effect of organic filler on flexural strength of LDPE

The above bar graph in Figure 26 shows that, in the group of organic fillers polyester fibers provide good flexural strength result than Inorganic filler like GF, MF and fly ash. The strength result increased to 27.79 MPa from its composition without PEF filler of result 10.84 MPa.

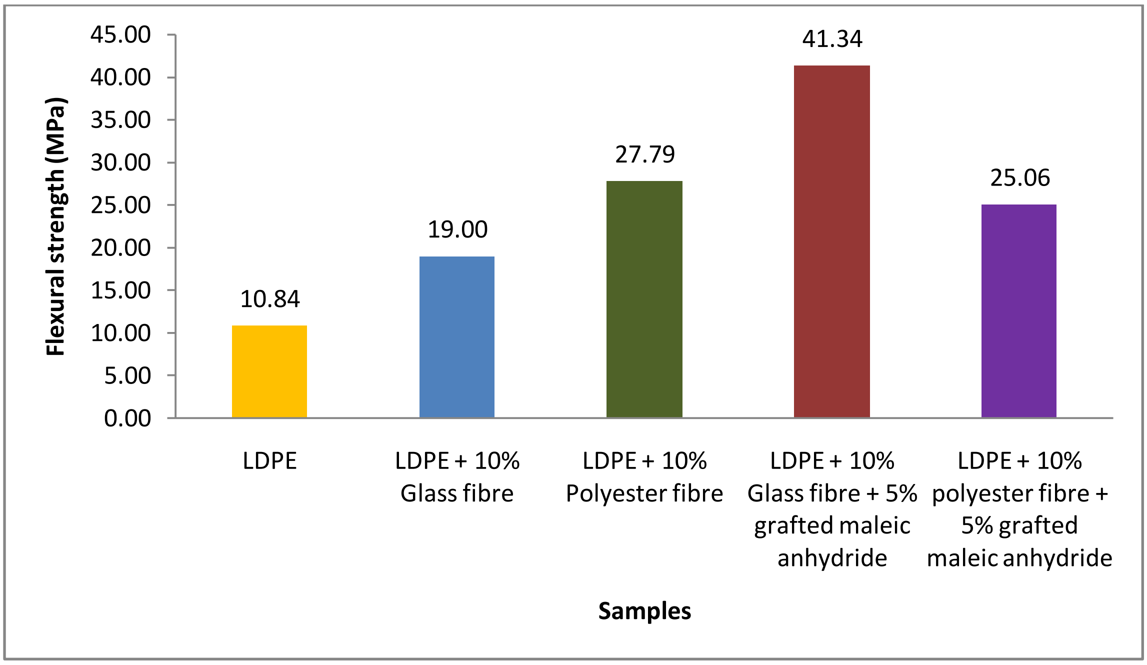

Figure 27. Effect of compatibilizer on flexural strength of LDPE

gMA used as compatibilizer with GF and PEF composites, as the result compatibility of gMA was good with GF than PEF. When it comes to development of result before and after addition of compatibilizer, the composition of GF with comatibilizer gave twice the increase in flexural strength than the composition without compatibilzer. Because, anhydride carbonyl groups that interact with the backbone of LDPE matrix. But in case of PEF composition, the flexural strength got decreased slightly than the composition without addition of compatibilizer, and the reason is due to gMA used was mentioned for GF composites and properties would have been increased by the use of surface active agent .

The flexural strength of LDPE and LDPE with different filler compositions is shown in fig. 27, and It is observed that the modulus of LDPE + 10% GF + 5% MA had increased steadily to 41.34 MPa compared to strength of LDPE + 10% GF had 19 MPa . But, LDPE + 10% PEF+ 5% gMA has a slight decrease in flexural strength of 25.06 MPa compared to its same composition without an extra 5% of gMA shows modulus of 27.79 MPa, due to loss of surface active agents and inhibitor in PEF to make better bond with the use MA. This increase may be explained by a better fibers-matrix interaction under the compressive stresses during bending, developed in the transverse section of the flexural specimens for whatever the surface condition of the fibers. Flexural modulus of LDPE + 10% PEF has more modulus data compared to 10% GF composition, this shows the addition of MA alters fiber bonding with LDPE respective to Flexural test. The other filler compositions show almost same modulus value.

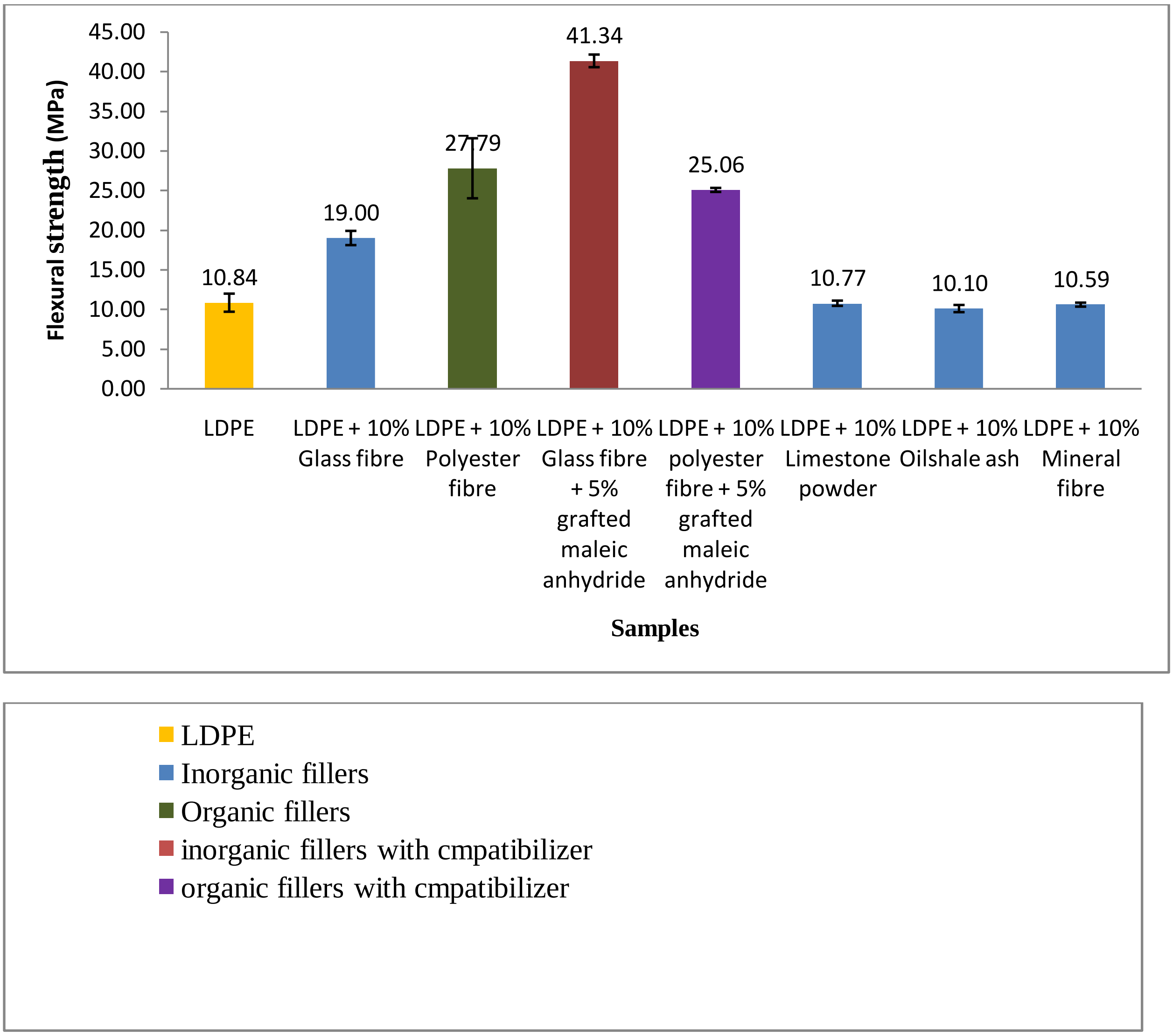

Figure 28. Effect of fillers and compatibilizer on the flexural strength of LDPE composite

3.3. Cooling Speed analysis

Cooling speed was done to know the thermal conductivity of LDPE and its composites with inorganic mineral like fly ash and MF.

From the bar graph (Figure 29) it is easy to identify the cooling speed, which was calculated between 190 ^{0}C to 40 ^{0}C. The cooling time varies with different filler compositions, and from the Figure 29, one can observe the cooling time required for limestone ash compound is much higher than LDPE, MF and oil shale ash compounds. In particular filler compositions the cooling time decreases as the filler content increases. The main objective was to find the effect of mineral wool fiber on the cooling properties of LDPE and its composites and it was decreased by 3 min for mineral wool fiber, and oil shale ash shows better results than mineral fiber by decreasing the cooling time by 5 min compare to LDPE.

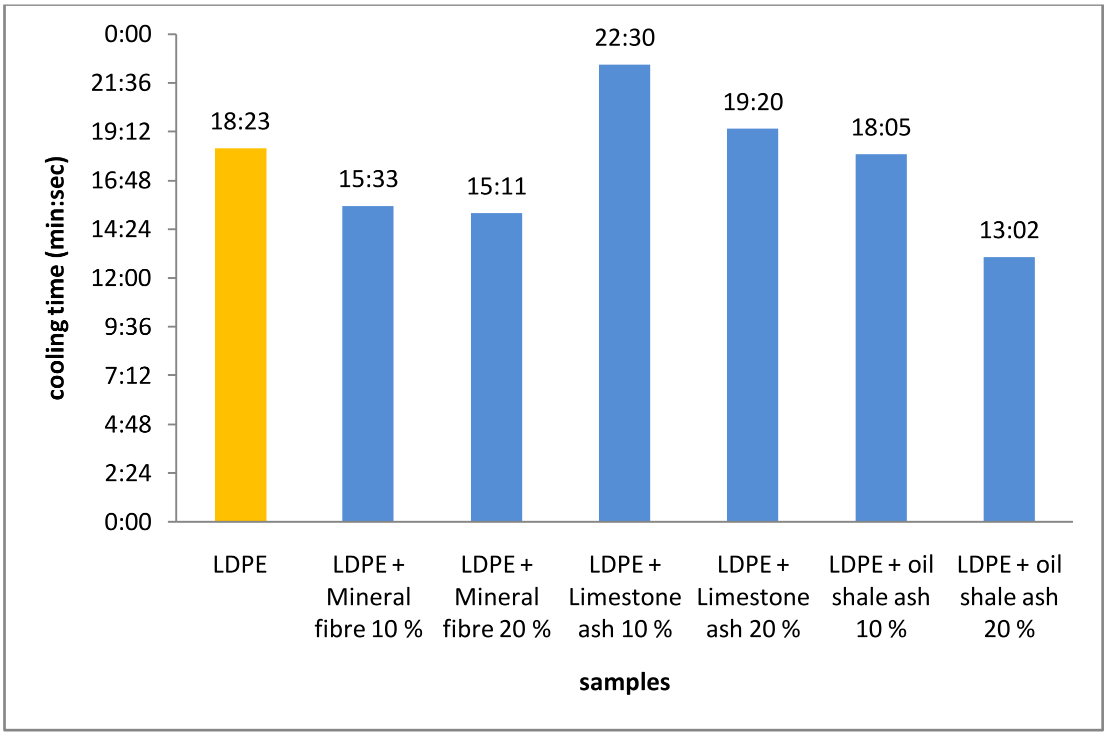

Figure 29. Effect of filler on the cooling speed of LDPE

Conclusion

In this study, fillers like MF and PEF were quite difficult to compound with LDPE powder, though its particle size had bring down to 2 mm by using “Retsch SM100” - Cutting mill.

compounding filler with MA compatibilizer was easy compare to VTMS, due to the physical form of MA was in solid particles form and VTMS was in liquid form was diluted with water to reduce concentration. And to remove the water out of PEF in fiber wet process with VTMS, the composition was subjected to heat in dry air Owen. Mineral wastes like oil shale ash, limestone ash were in fine powder form and it was very easy to mix and compound with LDPE.

Tensile strength of LDPE significantly increases with the addition of fly ash and MF, compared to GF and PEF. The addition of 5% MA to LDPE + GF 10% increased the tensile strength way better than PEF. The addition of MA gave higher tensile result than fly ashes and MF.

Addition of VTMS compatibilizer with PEF didn’t show any changes in default properties of LDPE + 10% PEF composition. Addition of mineral fillers like oil shale ash, limestone ash and mineral wool fiber slightly increases the modulus and tensile strength of LDPE.

From all the above discussion that GF with MA compatibilizer makes a better increase in tensile properties of LDPE. Fly ash and MF alone gave better results than GF and PEF.

Flexural strength results show the similar characteristics as in tensile. But, here PEF without any compatibilizer gives better bending properties than 10% GF. But, the addition of MA to both PEF and GF compositions shows increasing properties in GF than PEF. The tensile strength of MF, oil shale ash remains same with raw LDPE.

Cooling speed analysis gives a overview on the time required for cooling different compounds and it can be improved with the external cooling supply. In 10% filler compositions MF provides a better cooling time than all other compositions, but in oil shale ash as the filler concentration increases cooling rate has increased. So, both MF and oil shale can be used as filler which increases thermal conductivity by increasing the cooling rate from 18:23 to 15:11 (min:sec) for MF, and from 18:23 to 13:02 (min:sec) for oil shale ash.

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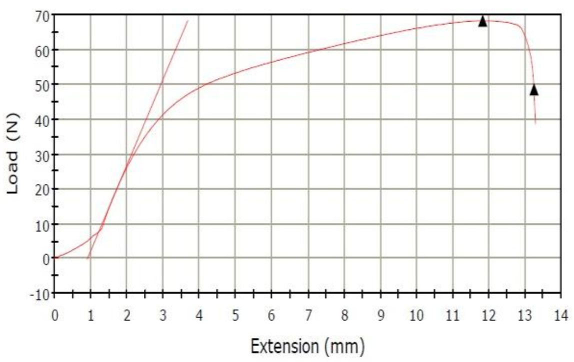
Appendix

Tensile Test

LDPE

Tensile properties of LDPE

Specimen No.	Maximum Load (N)	Extension at maximum load (mm)	Load at break (N)	Extension at break (mm)	Youngs modulus (MPa)
1	68.28	11.8	48.58	13.2	129.25
2	74.42	12.6	55.24	14.2	124.86
3	69.69	12.1	6.4	20.4	115.19
4	69.95	11.8	65.69	12.7	134.20
5	71.16	12.4	60.35	13.6	122.53
Avg	**70.7**			**14.82**	**125.206**



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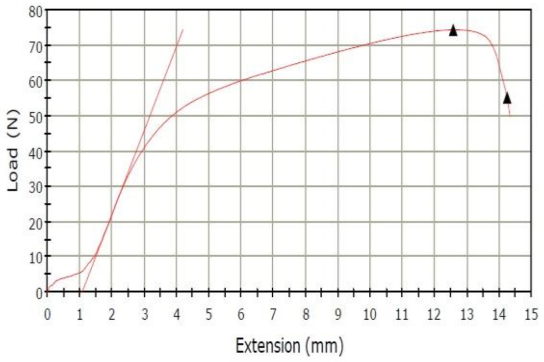

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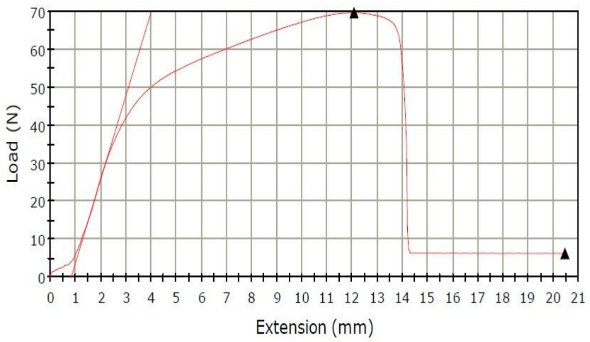

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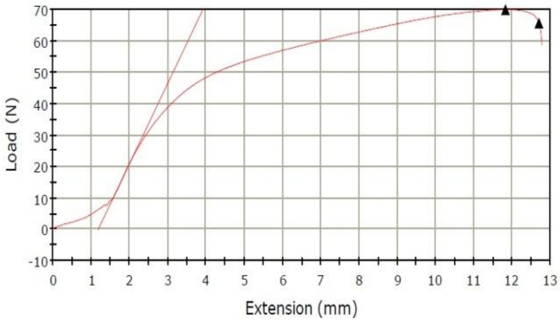

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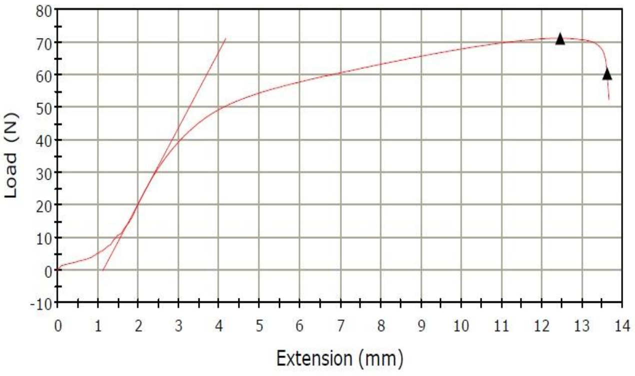


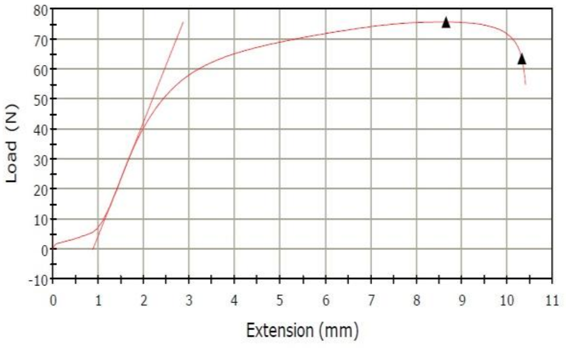
Sample 5

Tensile strength of LDPE samples

LDPE + 10% GF

Tensile properties of LDPE + 10% GF

Specimen No.	Maximum Load (N)	Extension at maximum load (mm)	Load at break (N)	Extension at break (mm)	Youngs modulus (MPa)
1	75.61	8.6	63.53	10.3	199.40
2	66.56	8.1	5.06	14	199.86
3	76.18	8.8	60.26	10.6	177.63
4	73.83	8.3	7.21	13.9	208.31
5	69.29	7.7	5.92	12.1	191.93
Avg	**72.294**			**12.18**	**195.426**



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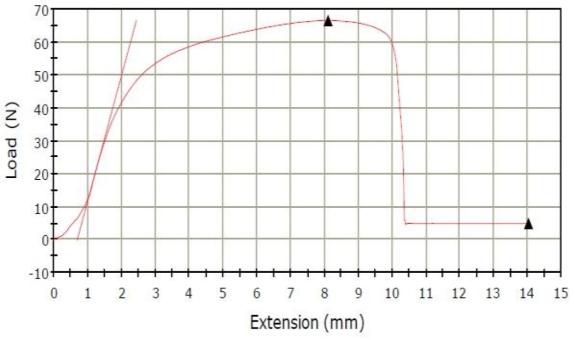

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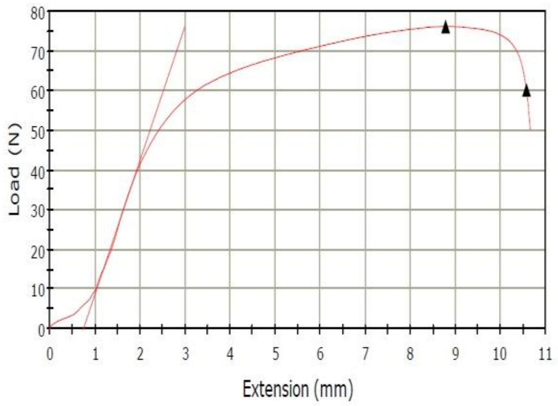

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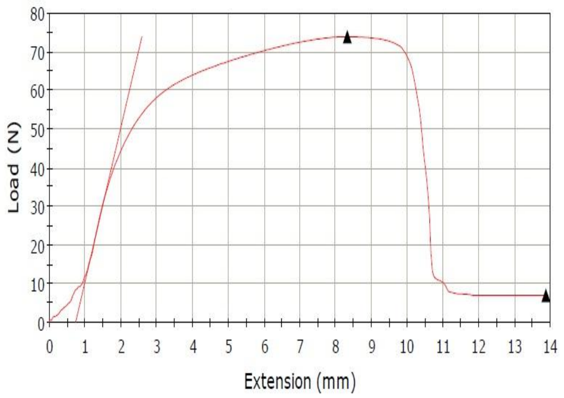

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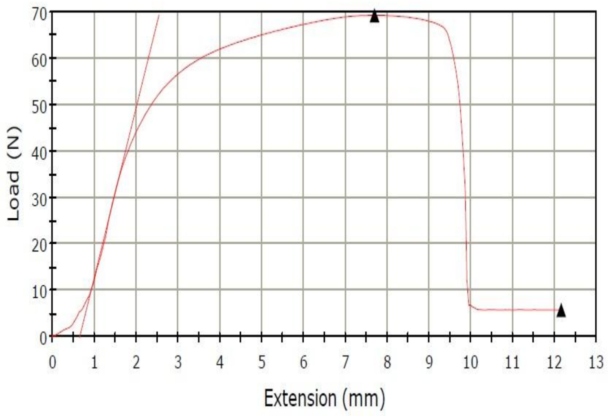


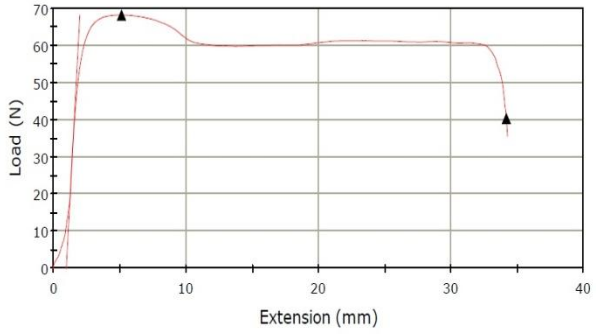
Sample 5

Effect of GF on tensile strength of LDPE samples

LDPE + 10% PEF

Tensile properties of LDPE + 10% PEF

Specimen No.	Maximum Load (N)	Extension at maximum load (mm)	Load at break (N)	Extension at break (mm)	Youngs modulus (MPa)
1	68.25	5.1	40.44	34.1	348.20
2	70.07	4.4	50.44	29.9	336.90
3	70.51	4.5	51.7	27.6	386.99
4	71.40	4.6	50.29	36.1	308.36
5	71.03	4.8	46.68	30.6	326.06
Avg	**70.252**			**31.66**	**341.30193**



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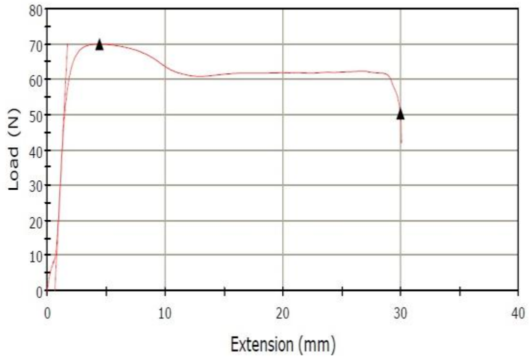

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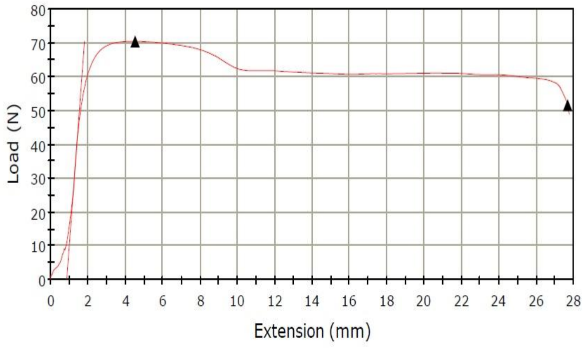

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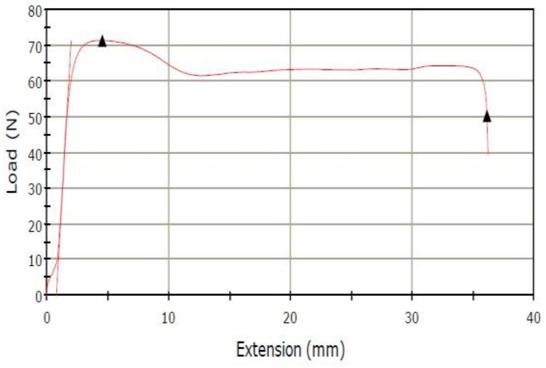

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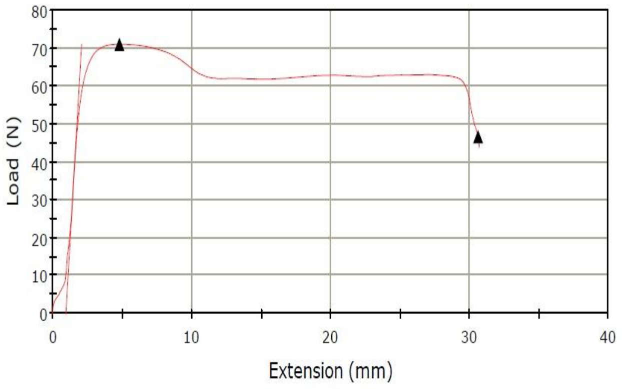


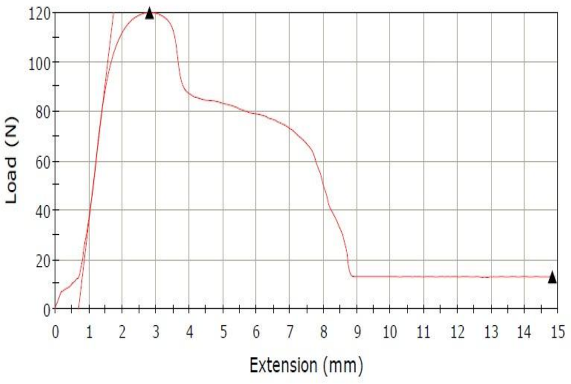
Sample 5

Effect of PEF filler on tensile strength of LDPE samples

LDPE + 10% GF + 5% grafted MA

Tensile properties of LDPE + 10% GF + 5% grafted MA

Specimen No.	Maximum Load (N)	Extension at maximum load (mm)	Load at break (N)	Extension at break (mm)	Youngs modulus (MPa)
1	119.78	2.8	13.20	14.8	603.20
2	120.89	3.6	16.23	11.9	513.90
3	118.15	2.5	13.05	9.7	771.67
4	127.37	3.7	23.72	10.5	463.51
5	121.97	4.0	22.12	11.4	445.93
Avg	**121.632**			**11.66**	**559.642**



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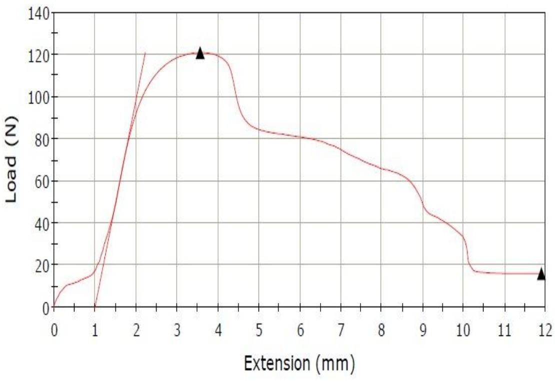

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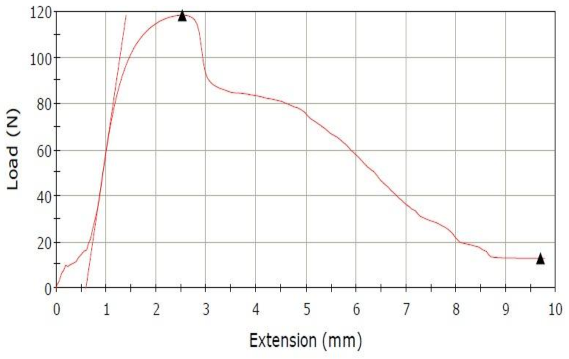

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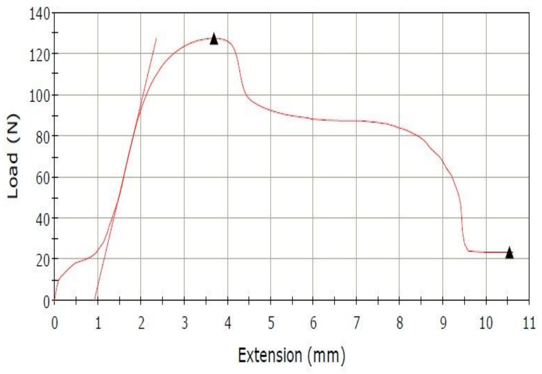

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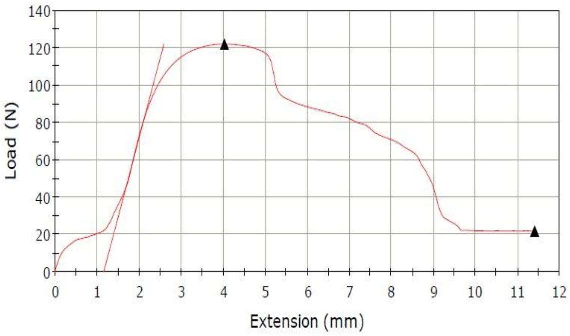


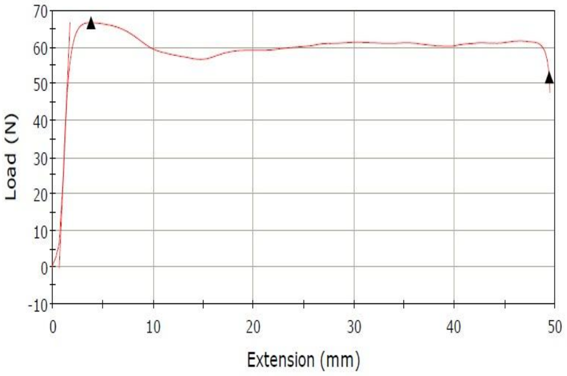
Sample 5

Effect of GF on tensile strength of LDPE samples with grafted MA as coupling agent

1.5. LDPE + 10% PEF + 5% grafted MA

Tensile properties of LDPE + 10% PEF + 5% grafted MA

Specimen No.	Maximum Load (N)	Extension at maximum load (mm)	Load at break (N)	Extension at break (mm)	Youngs modulus (MPa)
1	66.6	3.9	51.76	49.4	330.38
2	69.52	5.3	52.39	40	193.69
3	68.47	5.0	47.82	56	249.00
4	67.97	5.1	53.01	45.1	259.7
5	75.24	5.4	61.72	44.3	239.28
Avg	**69.56**			**46.96**	**254.41**



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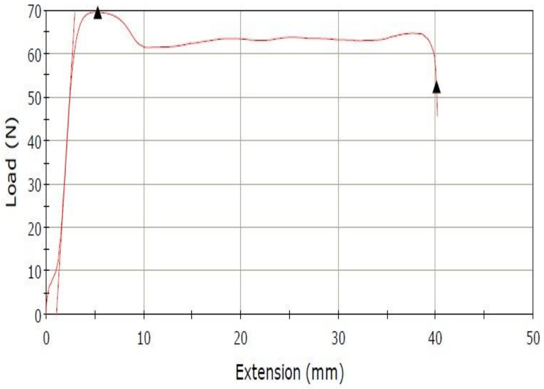

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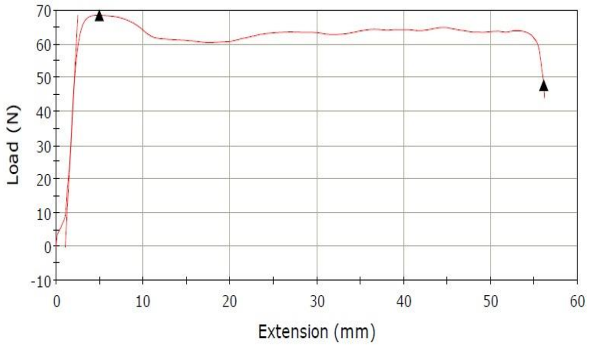

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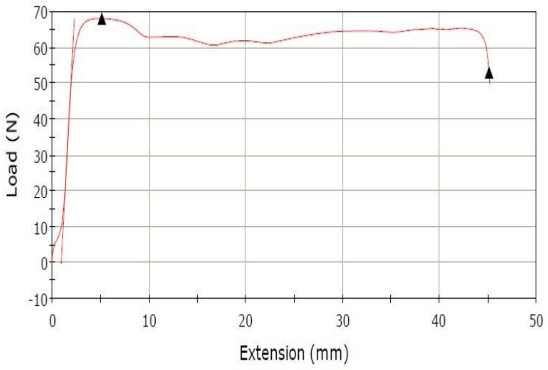

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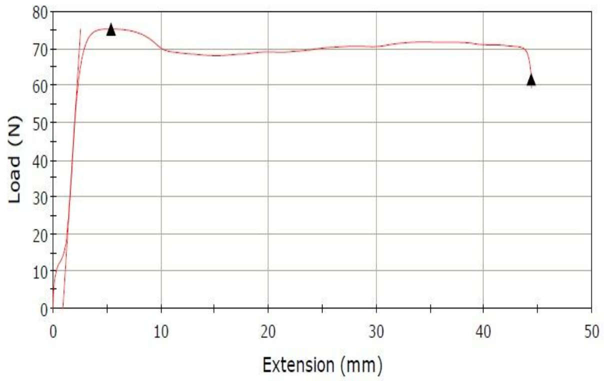


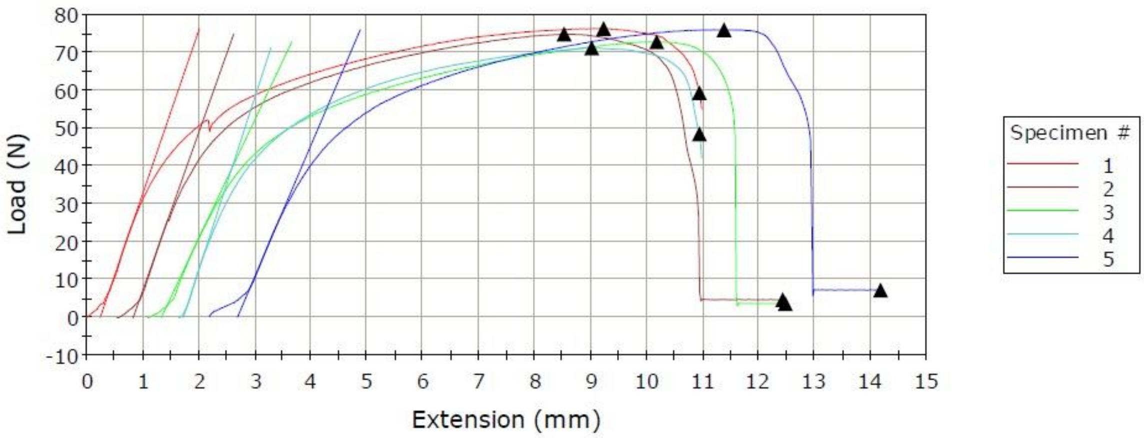
Sample 5

Effect of PEF filler on tensile strength of LDPE samples with grafted MA as coupling agent

LDPE+ 10% PEF+ 5% VTMS

Tensile properties of LDPE + 10% PEF + 5% VTMS

Specimen No.	Maximum Load (N)	Extension at maximum load (mm)	Load at break (N)	Extension at break (mm)	Youngs modulus (MPa)
1	76.19	9.2	59.32	10.9	107.33
2	74.73	9	4.79	11.9	103.99
3	72.72	9.1	3.71	11.4	77.89
4	71.09	7.4	48.4	9.3	113.9
5	75.89	9.2	7.29	12	86
Avg	**74.124**			**11.1**	**97.822**

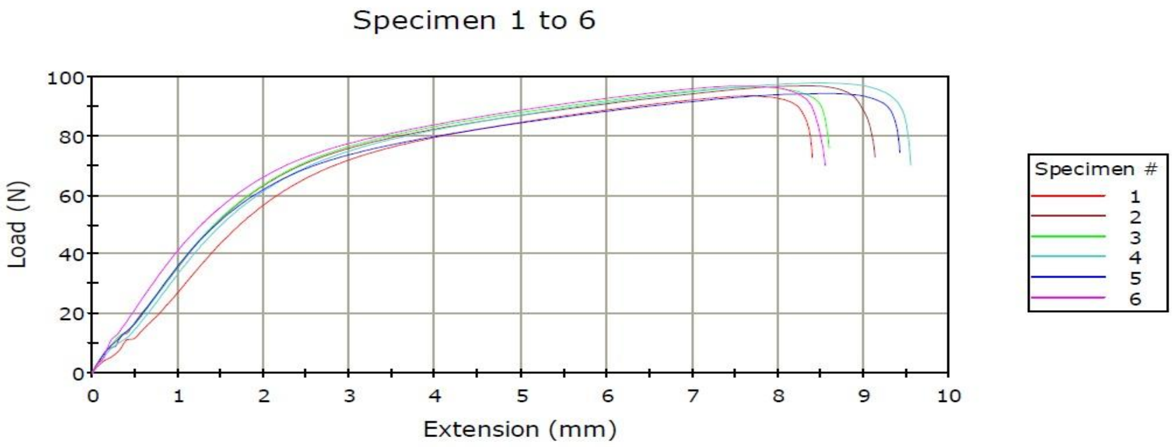


Effect of PEF filler on tensile strength of LDPE samples with grafted VTMS as coupling agent

LDPE + 10% limestone powder

Tensile properties of LDPE + 10% limestone ash

Specimen No.	Maximum Load (N)	Extension at maximum load (mm)	Load at break (N)	Extension at break (mm)	Youngs modulus (MPa)
1	89.56	7.72	85.94	8.31	107.00
2	94.12	8.65	84.52	9.05	131.00
3	96.32	7.85	88.66	8.51	131.50
4	97.19	8.72	87.05	9.46	127.10
5	92.38	8.96	86.78	9.33	130.60
6	96.14	7.98	83.51	8.46	144.60
Avg	**94.29**			**8.85**	**128.63**

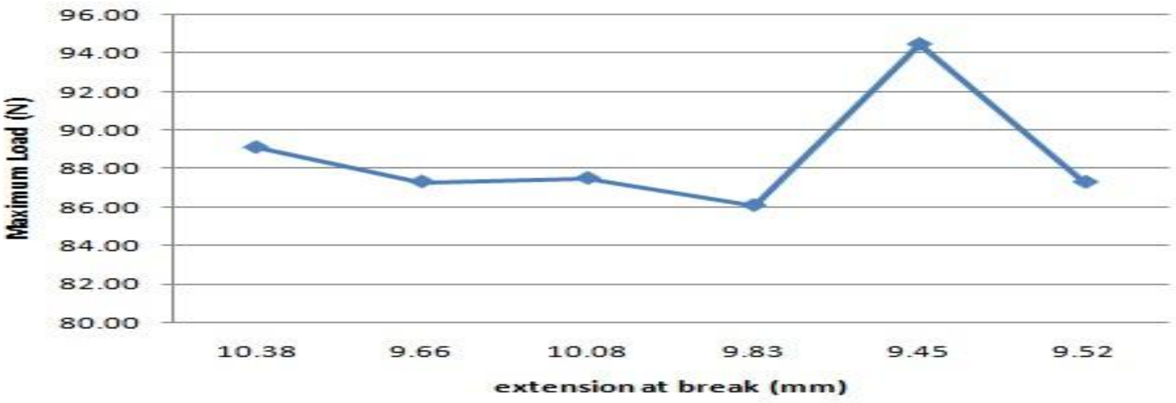


Effect of limestone ash on tensile properties of LDPE samples

LDPE + 10% oilshale ash

Tensile properties of LDPE + 10% oil shale ash

Specimen No.	Maximum Load (N)	Extension at maximum load (mm)	Load at break (N)	Extension at break (mm)	Youngs modulus (MPa)
1	89.12	9.30	71.44	10.38	103.20
2	87.33	9.45	73.27	9.66	106.20
3	87.51	9.64	77.88	10.08	104.80
4	86.10	9.62	81.74	9.83	97.50
5	94.49	9.20	86.99	9.45	119.90
6	87.31	9.32	73.26	9.52	110.10
Avg	**88.64**			**9.82**	**106.95**

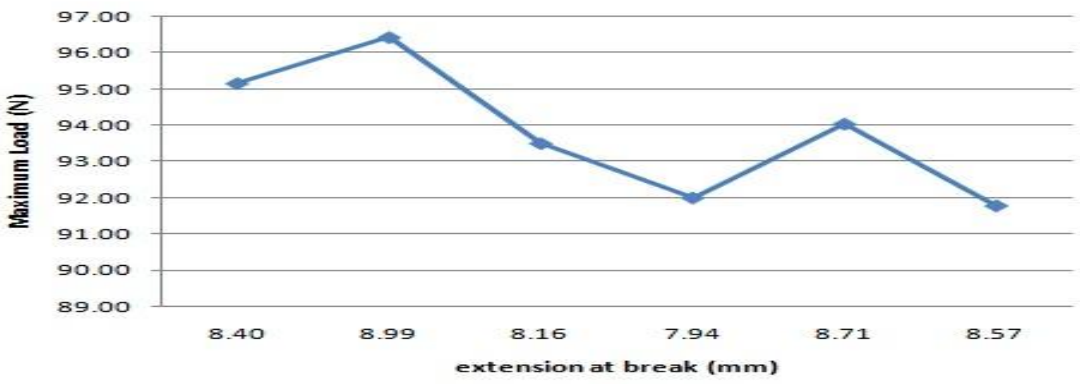


Effect of oilshale ash on tensile properties of LDPE samples

LDPE + 10% MF

Tensile properties of LDPE + 10% MF

Specimen No.	Maximum Load (N)	Extension at maximum load (mm)	Load at break (N)	Extension at break (mm)	Youngs modulus (MPa)
1	95.16	8.02	81.88	8.40	128.00
2	96.43	8.32	79.93	8.99	133.40
3	93.50	7.78	77.21	8.16	133.40
4	92.00	7.60	85.27	7.94	133.80
5	94.04	8.51	73.88	8.71	124.30
6	91.78	9.32	84.04	8.57	132.00
Avg	**93.82**			**8.46**	**130.82**



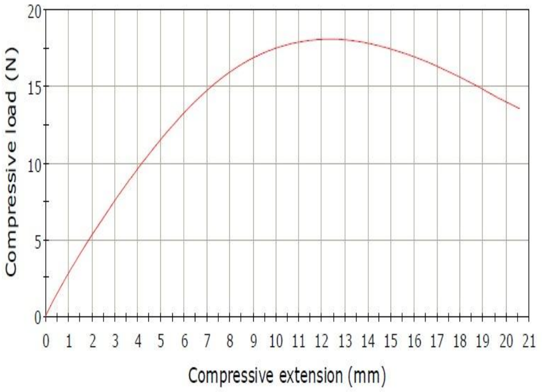
Effect of MF on tensile properties of LDPE samples

Flexural test

LDPE

Flexural strength values of LDPE samples

LDPE		
Specimen No.	**Maximum Compressive load(N)**	**Flexural Modulus (MPa)**
1	18.09	251.75
2	16.26	234.78
3	20.88	294.02
4	20.80	310.82
5	20.31	320.54
	19.268	**282.382**



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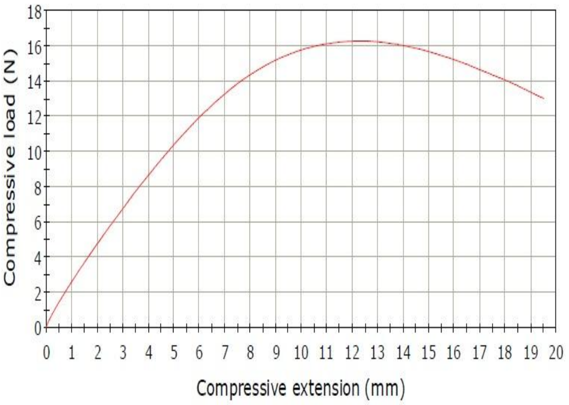

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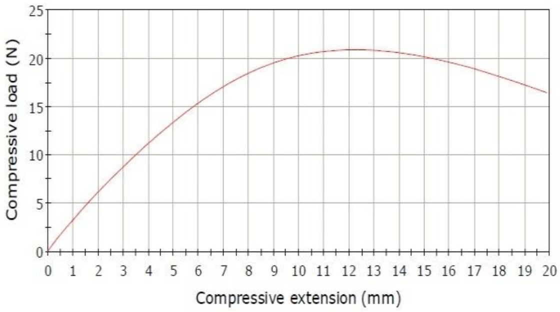

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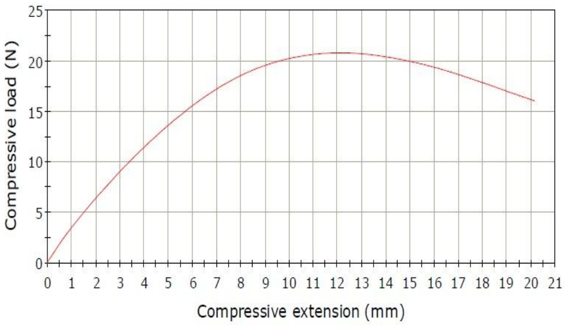

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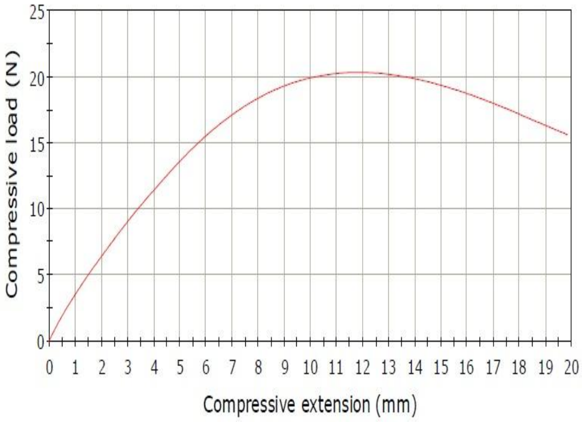


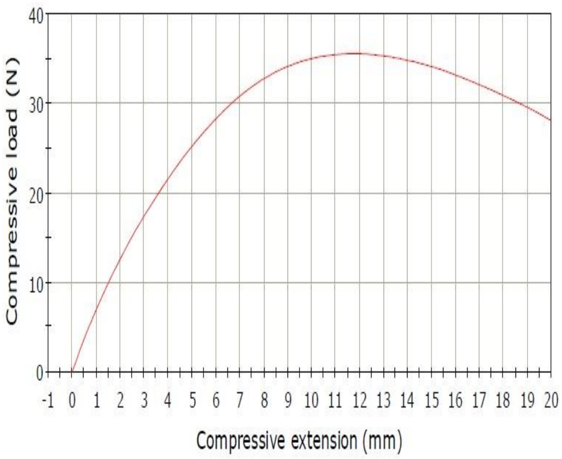
Sample 5

Flexural properties of LDPE samples

LDPE + 10% GF

Flexural strength values of LDPE + 10% GF samples

LDPE + 10% GF		
Specimen No.	**Maximum Compressive load(N)**	**Flexural Modulus (MPa)**
1	35.55	630.32
2	33.23	598.91
3	31.90	588.48
4	35.32	631.67
5	32.87	614.91
	33.774	**612.858**



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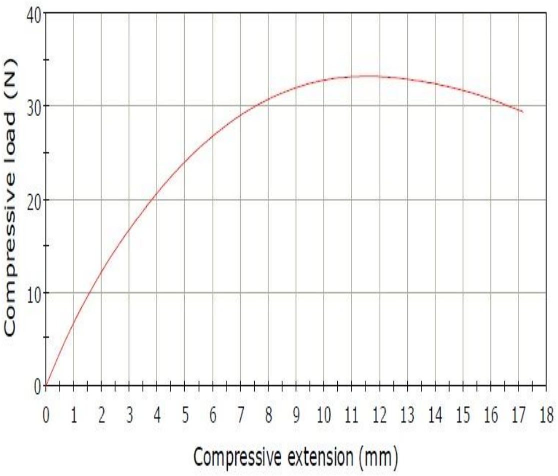

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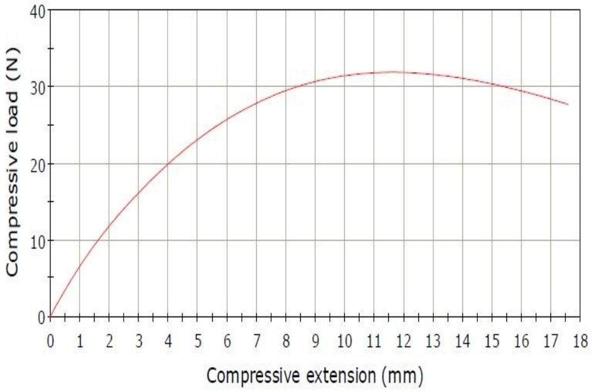

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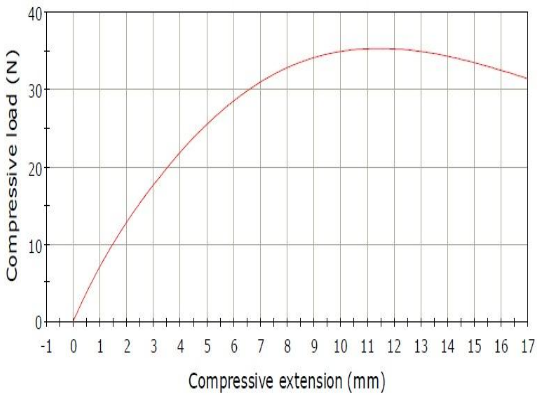

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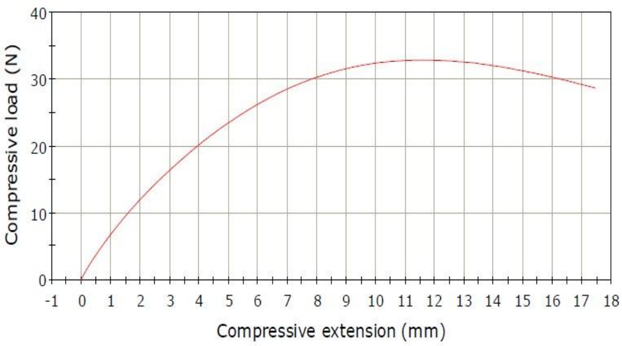


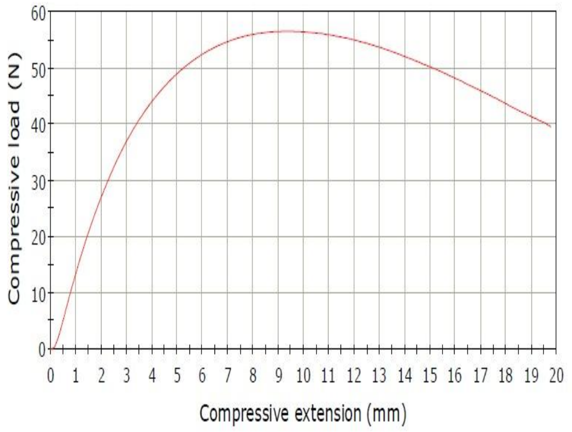
Sample 5

Effect of GF on the flexural properties of LDPE samples

LDPE + 10% PEF

Flexural strength values of LDPE + 10% PEF samples

LDPE + 10% PEF		
Specimen No.	**Maximum Compressive load(N)**	**Flexural Modulus (MPa)**
1	56.45	788.24
2	57.09	693.77
3	44.66	1072.18
4	44.94	1133.18
5	43.91	1098.67
	49.41	**957.208**



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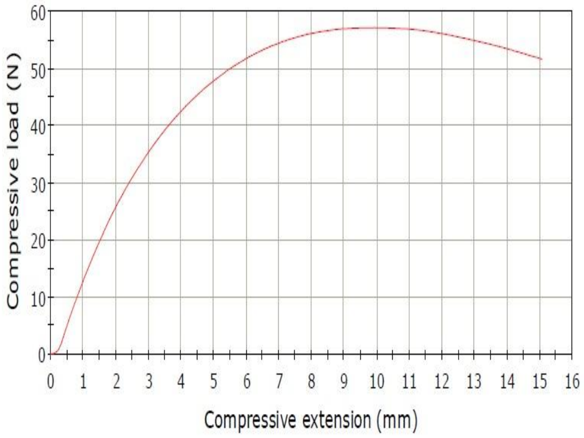

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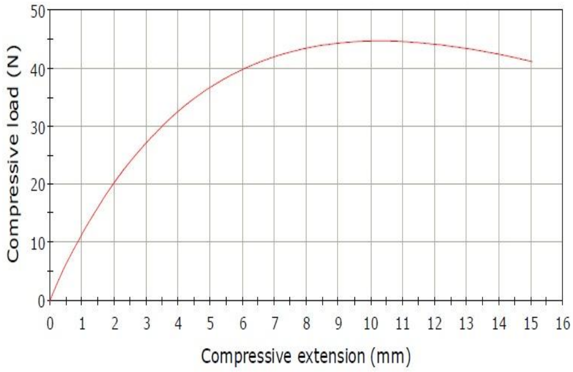

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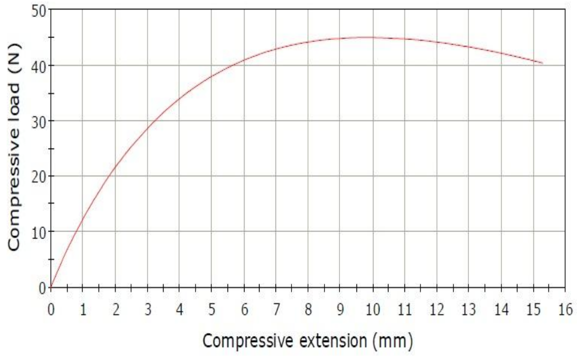

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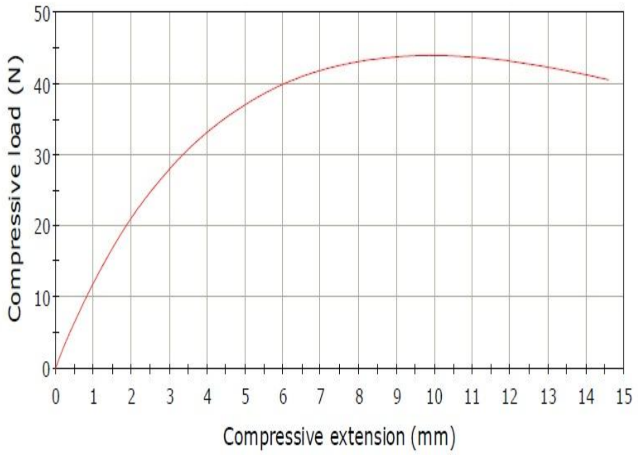


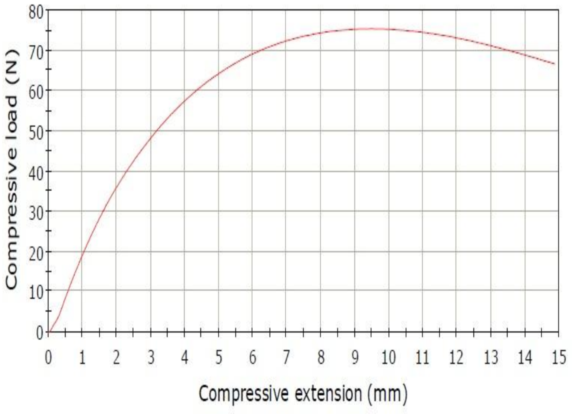
Sample 5

Effect of PEF on the flexural properties of LDPE samples

LDPE + 10% GF + 5% grafted MA

Table 14. Flexural strength values of LDPE + 10% GF + 5% grafted MA samples

LDPE + 10% GF + 5%CoMPatibilizer		
Specimen No.	**Maximum Compressive load(N)**	**Flexural Modulus (MPa)**
1	75.32	1373.08
2	72.35	1846.38
3	74.24	1957.31
4	71.84	1754.44
5	73.72	1777.38
	73.494	**1741.718**



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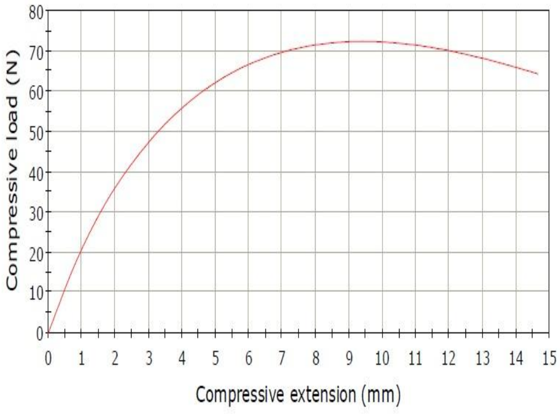

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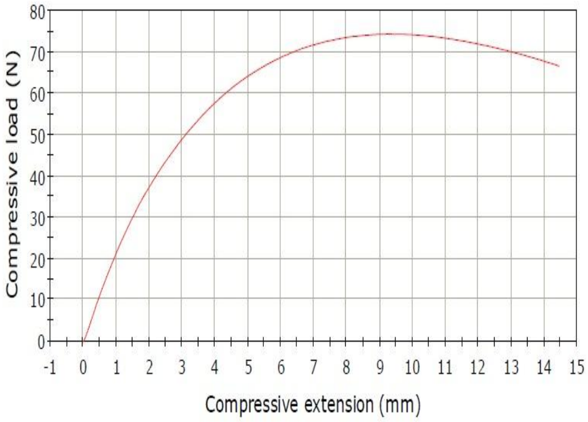

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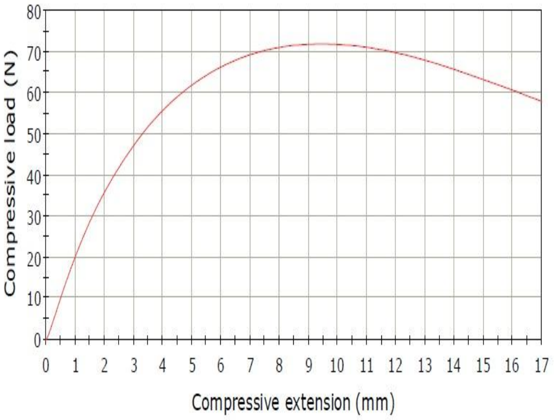

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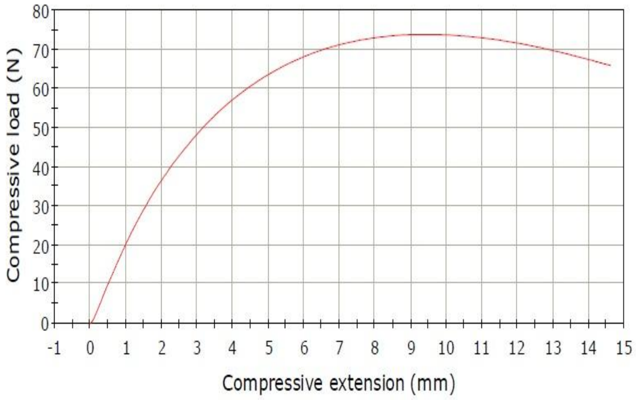


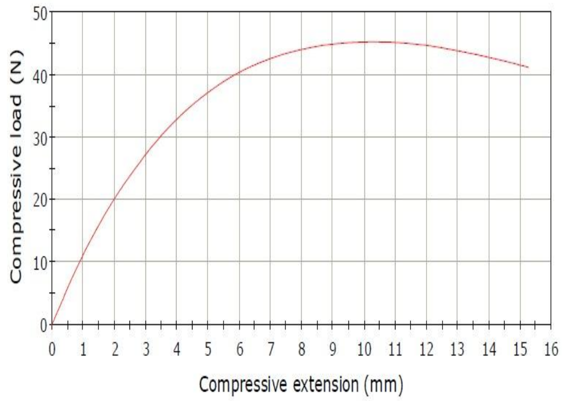
Sample 5

Effect of GF on the flexural properties of LDPE samples with grafted MA as coupling agent

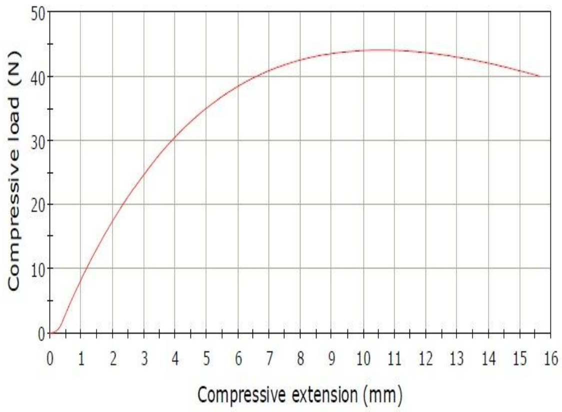
LDPE + 10% PEF + 5% grafted MA

Flexural strength values of LDPE + 10% PEF + 5% grafted MA samples

LDPE + 10% PEF + 5%CoMPatibilizer		
Specimen No.	**Maximum Compressive load(N)**	**Flexural Modulus (MPa)**
1	45.19	969.59
2	44.02	389.85
3	44.61	981.25
4	44.25	731.85
5	44.72	932.08
	44.558	**800.924**



Sample 1



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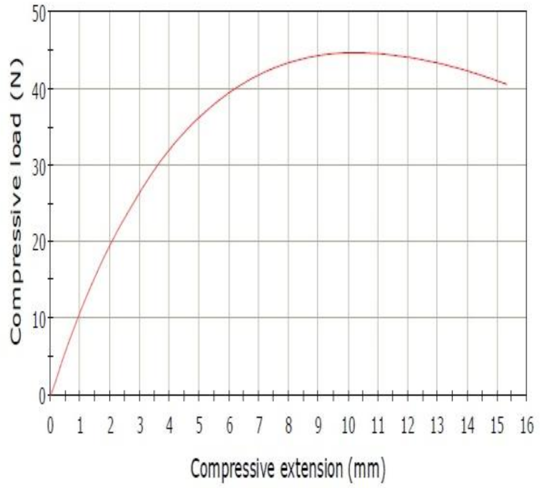

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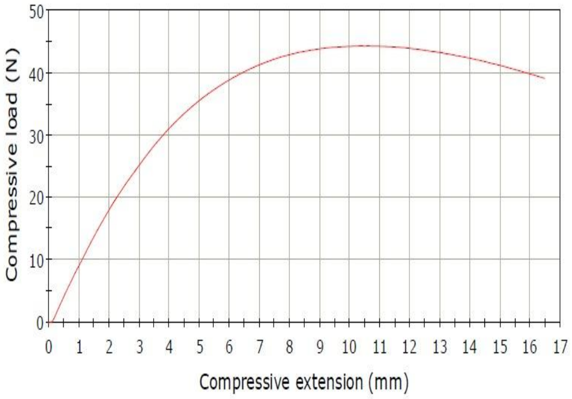

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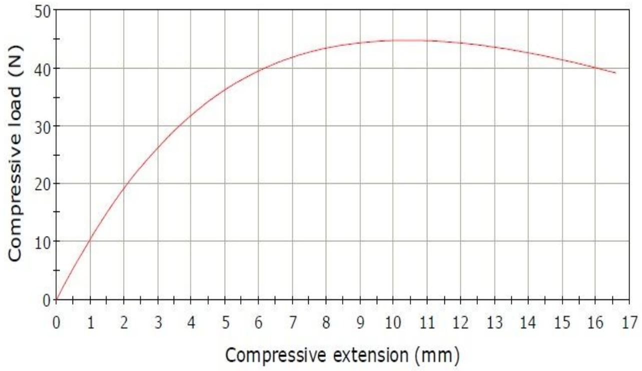


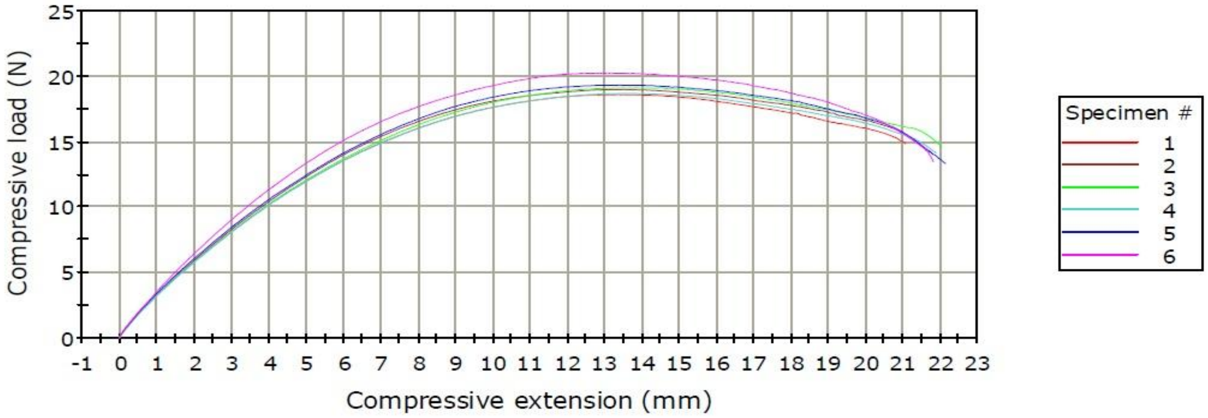
Sample 5

Effect of PEF on the flexural properties of LDPE samples with MA as coupling agent

LDPE + 10% Limestone Ash

Flexural strength values of LDPE + 10% limestone ash samples

LDPE + 10% Limestone Ash			
Sample no.	**Maximum Compressive load(N)**	**Flexural Strength (MPa)**	**Flexural Modulus (MPa)**
1	18.59	10.46	305.42
2	18.99	10.68	296.05
3	19.12	10.76	295.59
4	18.68	10.51	291.17
5	19.32	10.87	302.19
6	20.13	11.38	320.19
Mean	**19.14**	**10.78**	**301.77**

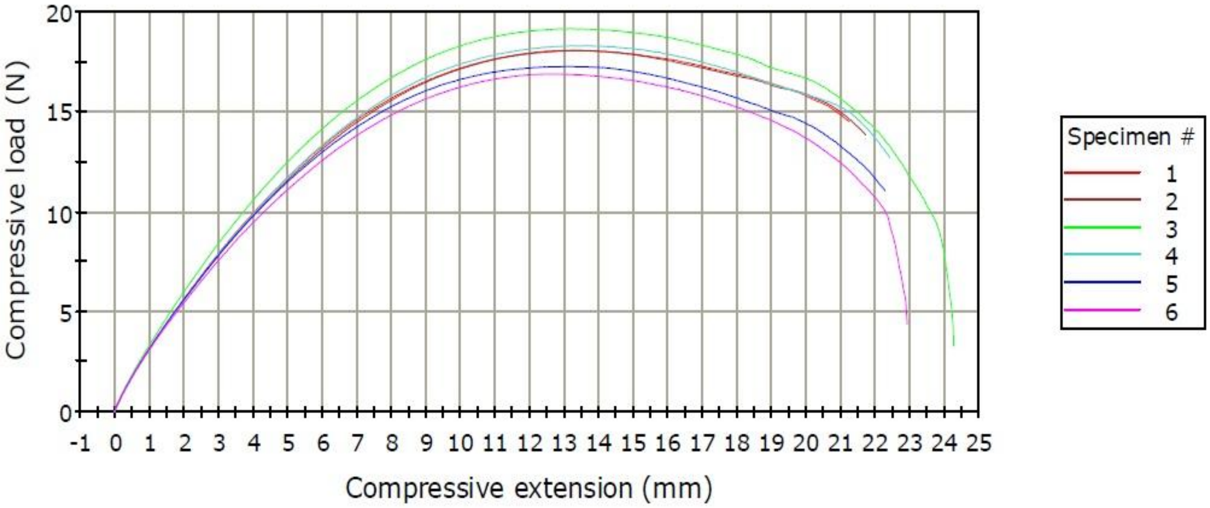


Effect of limestone ash on the flexural properties of LDPE samples

LDPE + 10% Oilshale Ash

Flexural strength values of LDPE + 10% Oilshale Ash samples

LDPE + 10% Oilshale Ash			
Sample no.	**Maximum Compressive load(N)**	**Flexural Strength (MPa)**	**Flexural Modulus (MPa)**
1	18.05	10.16	287.31
2	18.05	10.15	291.80
3	19.13	10.76	309.47
4	18.29	10.29	280.53
5	17.29	9.70	287.00
6	16.87	9.47	289.51
Mean	**17.95**	**10.09**	**290.94**

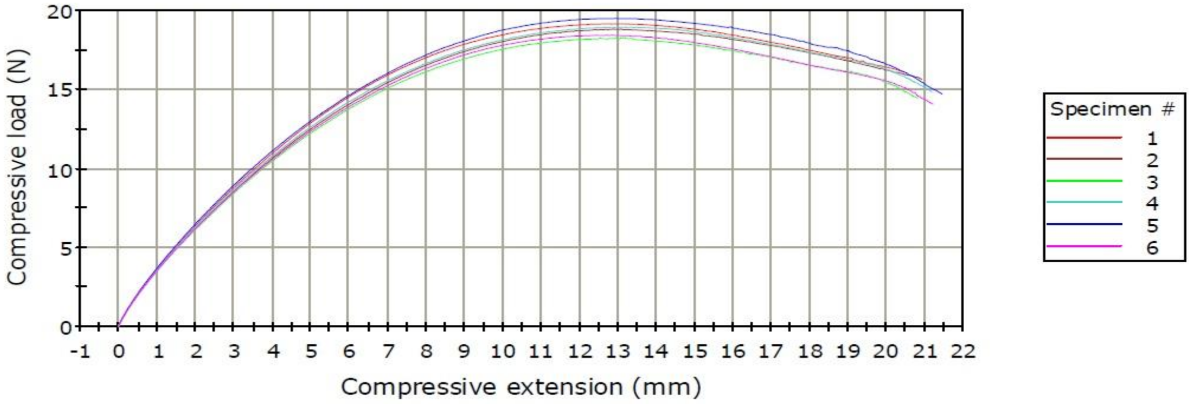


Effect of oil shale ash on the flexural properties of LDPE samples

LDPE + 10% MF

Flexural strength values of LDPE + 10% MF samples

LDPE + 10% MF			
Sample no.	**Maximum Compressive load(N)**	**Flexural Strength (MPa)**	**Flexural Modulus (MPa)**
1	19.13	10.76	336.54
2	18.79	10.57	329.26
3	18.26	10.27	331.68
4	18.91	10.64	333.38
5	19.47	10.95	340.33
6	18.42	10.36	327.91
Mean	**18.83**	**10.59**	**333.18**

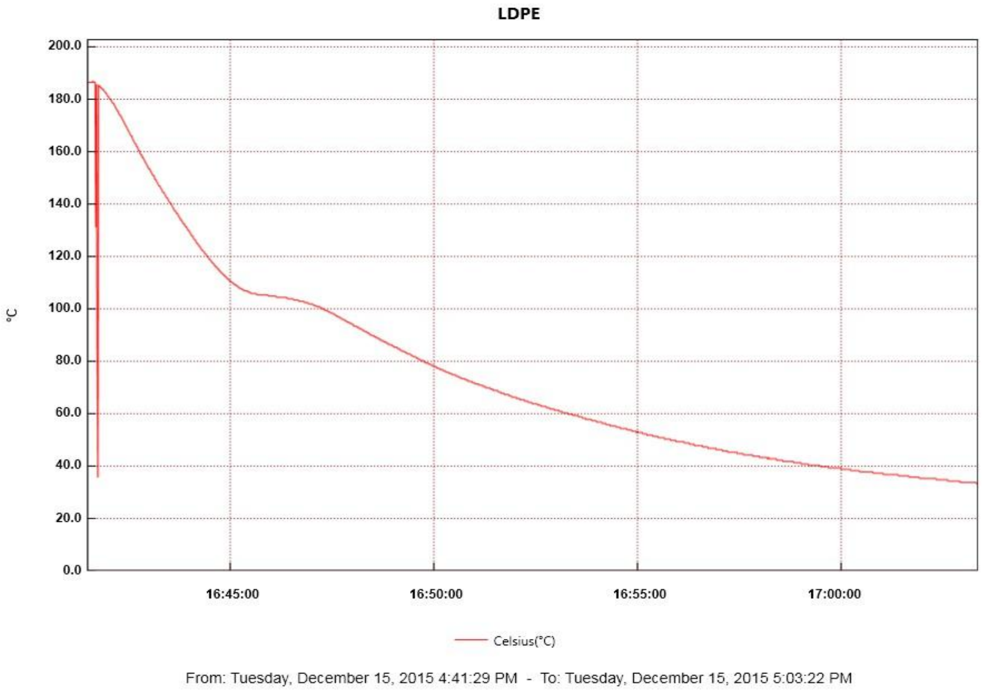


Effect of MF on the flexural properties of LDPE samples

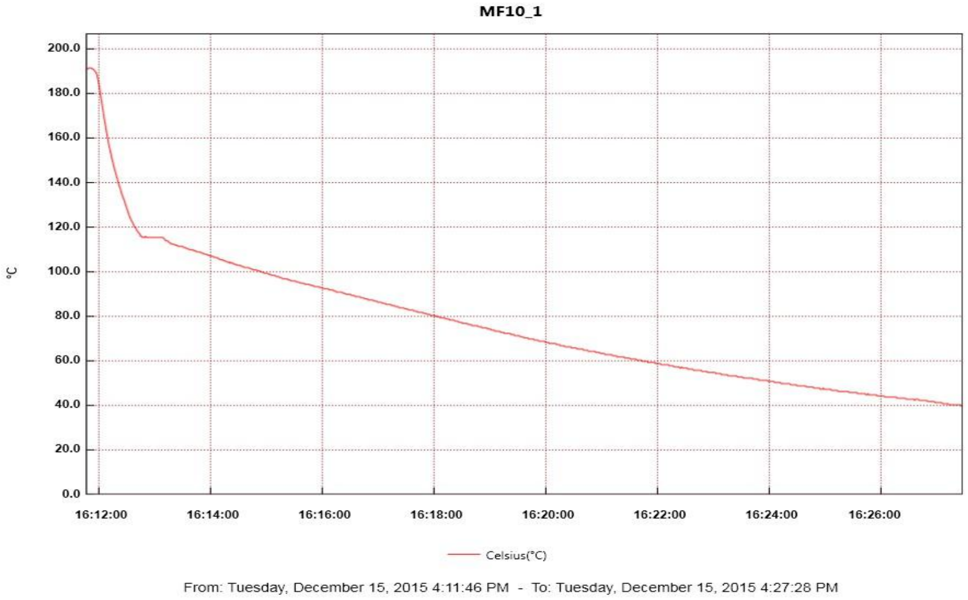
Cooling rate analysis

Cooling speed results for LDPE compoaites

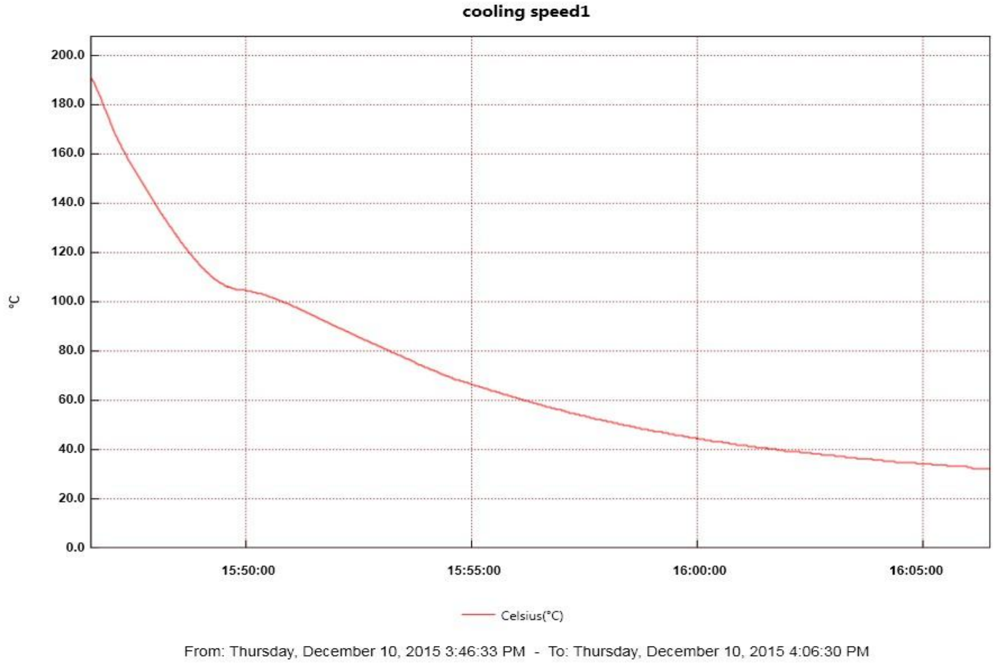
Sample	Cooling Speed (min:sec)
LDPE	18:23
LDPE + Mineral fibre 10 %	15:33
LDPE + Mineral fibre 20 %	15:11
LDPE + Limestone ash 10 %	22:30
LDPE + Limestone ash 20 %	19:20
LDPE + oil shale ash 10 %	18:05
LDPE + oil shale ash 20 %	13:02



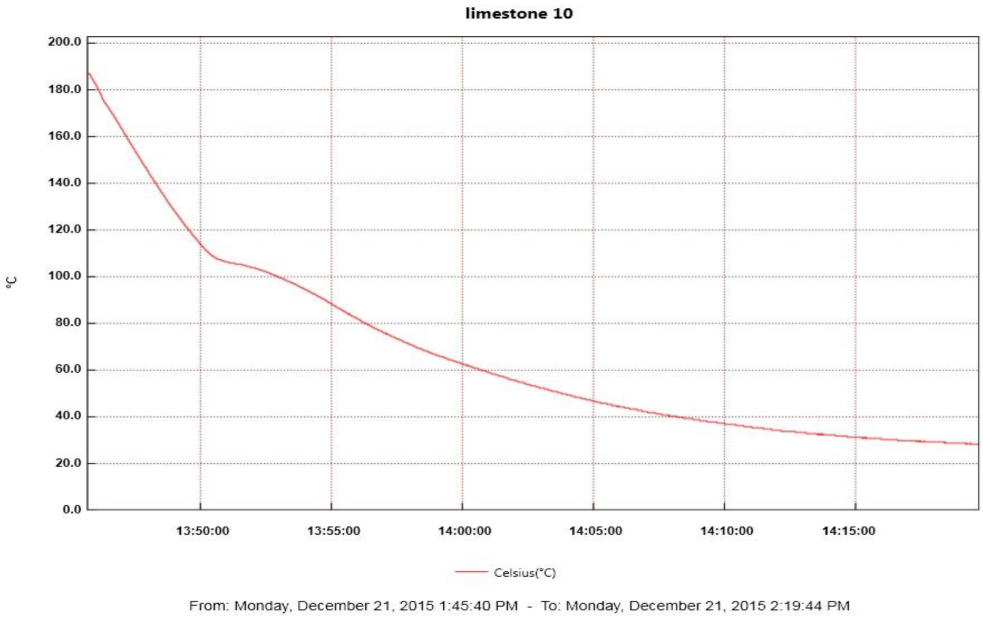
Cooling speed analysis for LDPE



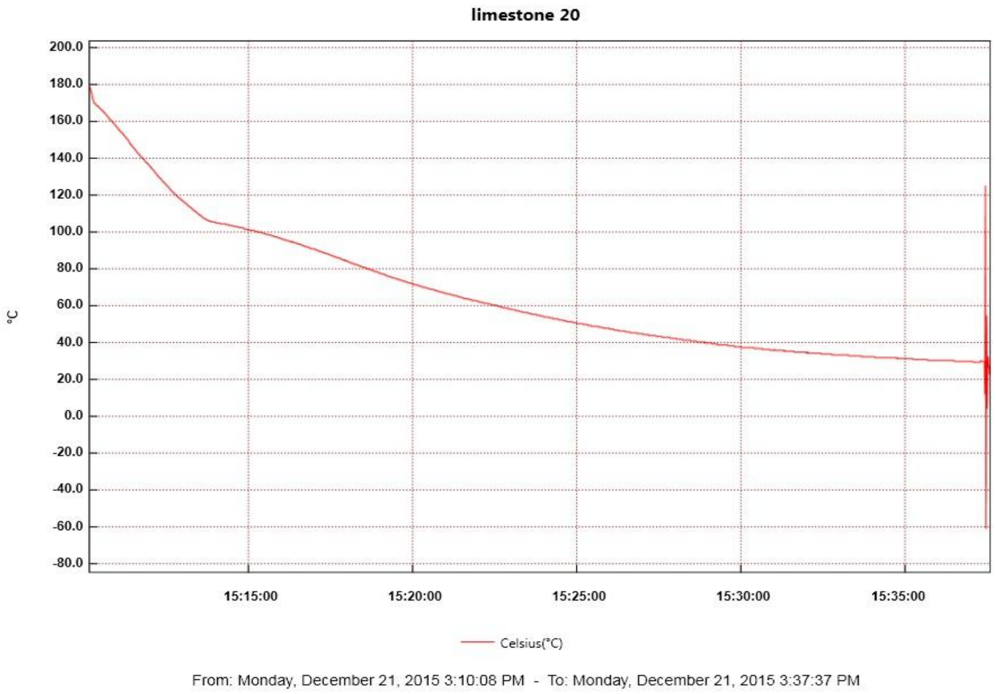
Effect of 10% MF on cooling speed of LDPE



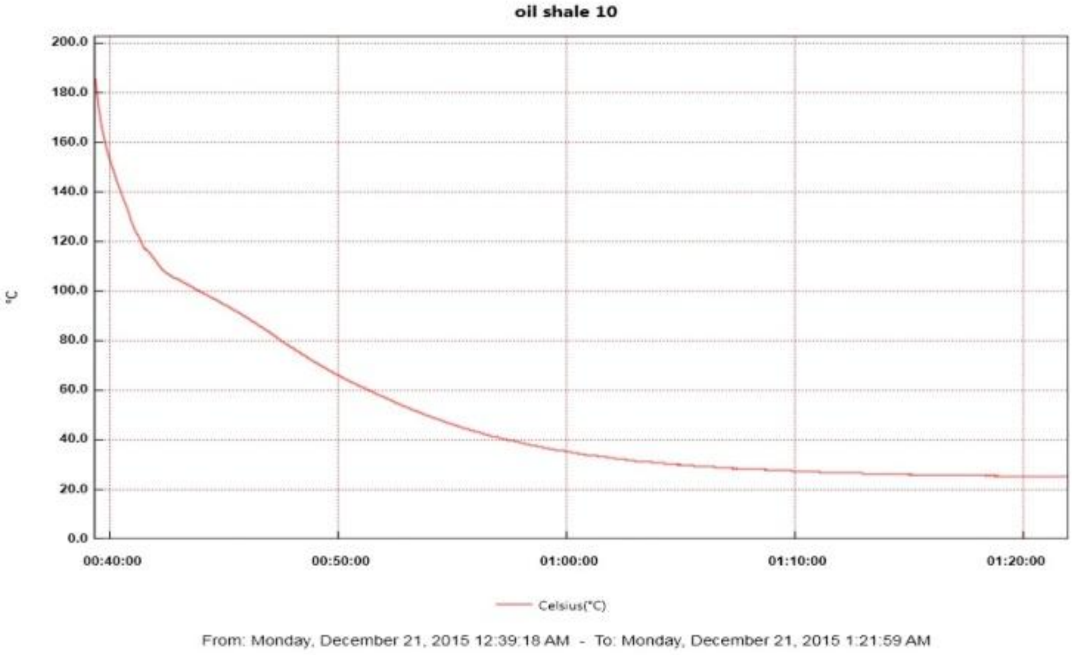
Effect of 20% MF on cooling speed of LDPE



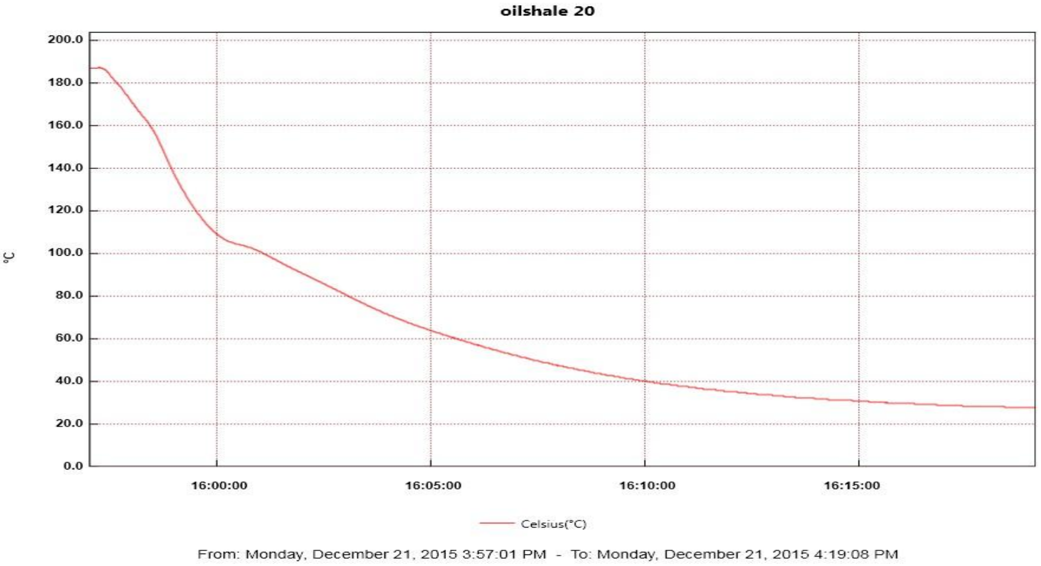
Effect of 10% limestone ash on cooling speed of LDPE



Effect of 20% limestone ash on cooling speed of LDPE



Effect of 10% oil shale ash on cooling speed of LDPE



Effect of 20% oil shale ash on cooling speed of LDPE

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Title of thesis (in English): Effect of Some organic and inorganic fillers on physical properties of LDPE composites
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Supervisor(s): Andres Krumme
Date of defence: 08.06.2017
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