



TALLINN UNIVERSITY OF TECHNOLOGY
SCHOOL OF ENGINEERING
Department of Materials and Environmental Technology

ENVIRONMENTALLY FRIENDLY TEXTILE FINISHING BY ELECTROSPRAYING METHOD

KESKKONNASÕBRALIK KANGATÖÖTLUS ELEKTROPIHUSTUS MEETODIL

MASTER THESIS

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Tallinn 2022

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THESIS TASK

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(in Estonian) Keskkonnasõbralik kangatöötlus elektropihustus meetodil

Thesis main objectives:

Study electro spraying as fabric finishing method

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- To apply water-repellent finishing to cotton and polyester fabric by electro spraying method
- Testing the fabrics with water-repellent finishing
- Study morphology of the coating

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PREFACE

This current master's thesis topic is „Environmentally friendly textile finishing by electrospraying method“. The idea of this research is to study electrospraying as a fabric finishing method. As objectives set for the experiment, this method will be enforced onto 100% CO and 100% PES fabrics to enhance the textile water-repellent properties and afterward conclude the result for the behavior of tested textile substrates. Electrospraying allows for finishing process to happen under more controlled conditions than it could be followed for traditional wet treatment of fabrics. Performed finishing was analysed under scanning electron microscopy for determining the success of sprayed coating. The durability of the coating was tested for abrasion resistance, surface wetting and domestic washing procedures. From different testings could be concluded that the results of the treated samples were not as successful as hoped before experimenting. By changing solution concentration and altering distance between nozzle and collector plate, finishing can be more successful.

All the work was done under the supervision of the research scientist Illia Krasnou and Tiia Plamus in the Department of Materials and Environmental Technology at Tallinn Technical University. Author of the Master's thesis would like to thank supervisors for encouragement and relevant feedback throughout this study.

Keywords: electrospraying, fabric finishing, textile treatment, nanotechnology, textile testing

List of abbreviations and symbols

CO – cotton

PES – polyester

EtOH – ethanol

H₂O – water

APTES – (3-Aminopropyl) triethoxysilane

TEOS – tetraethyl orthosilicate

HCl – hydrochloric acid

CH₃OH – methanol

SEM – scanning electron microscopy

CAM – contact angle measurement

INTRODUCTION

Fabric finishing technologies have been invented to improve the fabric's qualities or appearance. This way, it is possible to change the feel or touch of the fabric or make them more suitable for specific uses. The term "finishing," in its broadest meaning, covers all processes that fabric undergoes after production. Even if materials inherit naturally good properties, some finishing is always necessary to lengthen the life of textile products. Topics on eco-friendly finishes performed for textiles refer to methods that do not hamper the surrounding atmosphere and environment and deliver sustainable products. Nowadays, the world around us is becoming increasingly aware of the implications of the damages already done or happening to our ecosystem. To prevent this and reduce damage to our environment, many norms/standards for finished textile products have been taken into action regulating the use of chemicals in manufacturing. [1, 2]

Textiles nowadays tend towards "bio, natural, and environmentally friendly" garments because of the increasing consciousness of consumers. Therefore, companies need to develop new and greener ways to replace hazardous chemical usage in textile production. Although ingredients may be biological or natural, the same kind of substances are often not used in the end-user processes.

The textile sector shows promising results for developing and applying nanotechnology and textile-related nanotechnology studies, including modifying textile materials and upgrading chemical finishes to improve dyeability, water- and oil-repellency, antibacterial and other properties via nanoparticles. Nanofinishing techniques allow the finishing process to be more precise and recur under more controllable conditions. One possible approach for this is electrospraying, in which the nanoparticles layer is deposited onto the fabric surface. The electrospraying setup consists of charged syringe fed with polymer solution onto a grounded collector plate with the substrate material. [3]

The electrodynamic spraying technique used in the study, also known as electropraying, can help produce diminutive droplets with submicron sizes by employing an electric field. A typical setup for the electrospraying process consists of a high-voltage power supply, a syringe capped by a capillary to hold a polymer solution, a syringe pump to control the flow of the solutions, and a grounded collector. With the applied electrical charge, the liquid jet breaks up to form droplets which form small particles onto a collector plate. The size of the sprayed particles can be restrained to an extent with

applied voltage, solution concentration, and distance between the needle tip and collector plate. [4]

Over the years, there have been developed many eco-friendly fabric finishing techniques. With these techniques they try to substitute harmful chemicals, but a larger side of them is other ways harmful to the ecosystem or rather costly to use in manufacturing; therefore, companies do not yet justify their adoption. Consumers become more aware of the possibilities and demand sustainable products with everyday changes. Hence it is vital to acknowledge the future opportunities for fabric finishing techniques.

The main aim of the thesis is to study electro spraying as a fabric finishing method. As objectives set for the experiment, this method will be enforced onto 100% CO and 100% PES fabrics to enhance the textile water-repellent properties and afterward conclude the result for the behavior of tested textile substrates. The environmental aspect of the study is concentrated on solvents used for electro spraying.

The thesis is divided into six chapters. The first chapter includes a literature overview explaining overall finishing techniques for textiles and highlights the leading environmentally friendly techniques used in manufacturing. The second chapter explains the electro spraying method's work basics and setup techniques. Different testing methods for finished textiles are discussed in the fourth chapter.

The experimental part and aim of the study are emphasized in chapter five, giving an overview of the experimental plan composed for the study. The chapter explains the materials and methods used for the study, how the experimental part was performed, and what tests were accomplished. The last, sixth chapter explains the results and conclusions obtained from the study.

1. ENVIRONMENTALLY FRIENDLY FINISHING METHODS OF TEXTILES

The general trend in the textile industry tends to lean towards using less water and less harmful chemicals. Eco-friendly textiles are considered as products that do not contain any hazardous or toxic substances and are biologically degradable so that they do not cause any damage to the environment. Due to growing consumer perception, environmental considerations are becoming vital factors in consumer habits. Awareness about eco-friendliness in textiles is one of the crucial issues in recent times. A systematic approach towards eco-friendliness involves identifying and avoiding harmful chemicals, minimizing the quantities of restricted chemicals, and recycling wherever possible. [5, 6, 7]

The concept of sustainability reflects the continuity of human society's economic, social, institutional, and environmental aspects. Sustainability affects every level of organization, from the local neighborhood to the entire planet. In which nonaqueous green solution treatments should replace the conventional wet processing of textiles. Demand for environmental friendliness of finishing processes is in growing needs and the interest in the functionality of fabrics in physically induced techniques for surface modifications and coatings. An economical alternative to water-based treatment is plasmatechnology. Also as other options could be considered microencapsulation and nanotechnology. [8]

1.1 Textile finishing

Textile materials are used in various ways according to the surface, properties, applications, etc. However, the surface properties are often not suitable for the end-use of textiles. Therefore, it is necessary to modify the textile materials to provide the required performance for the product they have been designed to become. [9]

In its broadest sense, the term "finishing" has been meant to cover all the processes that modify fabric after ready-made fabric leaves the loom or knitting machine. Finishing would also include bleaching and dyeing, but in this case, we view them in separate categories. [9]

It is evident that fibres have characteristic properties, but we can alter or improve those qualities in the finishing stage. The object of finishing is to improve the attractiveness and performance of the fabrics. In today's industries, fabrics require more and more chemical treatment to withstand heat, moisture, sunlight, etc. and last longer in their original condition. A more restricted view of finishing is that the fabric is prepared for end-use by manufacturer after weaving or knitting. A simple definition for finishing is the sequence of operations, other than sourcing, bleaching, and colouring, to which fabrics are subjected after leaving the loom or knitting machine. [9]

The technique of finishing is affected by many factors and can be held in wide variations, but mainly, it may be held to depend on four factors [9]:

1. fabric type and yarn arrangement in fabric;
2. fibre physical properties;
3. fabric capability of absorbing various finishing preparations;
4. chemical modification receptivity of the material.

Most of the simpler finishing processes are merely concerned with the effect of pressure, moisture, and heat. It is possible to divide finishing processes into two main categories: mechanical and chemical. The majority of surface enchantments are done by wet chemical processing. Wet chemical modification can improve the specific characteristics of fabrics by altering their chemical structure due to chemical reactions. Furthermore, the usage of chemicals often results in a decrease in fiber strength. [10, 9]

Synthetic fibers have been widely used for textiles because of their high mechanical strength, good stretchability, heat-stability, rapid dry-ing, wrinkle resistance, resistance to many common organic solvents, antibiotic properties, and weathering resistance, etc. However, they are inherently hydrophobic, which leads to wearing discomfort, low color strength, electrostatic charge build-up, a tendency to pill formation, and insufficient washability. To enhance the hydrophilicity of the synthetic fiber, surface modifications have been carried out for many years. [10]

1.1.1 Different textile finishings

Finishing processes for textiles have various functions intended to make the fabric more suitable for its intended use. Mainly finishes are done for five following reasons [11]:

1. To accentuate or inhibit the natural characteristics of the fibres. Like softening, stiffening, delustering, brightening, and changing surface characteristics.

2. Impact new properties or characteristics on the fabric, like durable press finishes, flame retardant finishes, and many other chemical treatments.
3. To increase the life and durability of the fabric.
4. To maintain the fabric's shape and structure.
5. For setting dyes.

Treatments added to the textiles can be classified as mechanical or chemical depending on the desired characteristic for the end-use of the fabric. Chemical finishes are often treated with aqueous solutions or chemicals, and mechanical finishes are modifications to dry fabric by a machine. Some overlap in the classification of finishing processes is inevitable since there are over 100 different types of fabric finishing techniques. The textile industry's most used fabric finishing techniques and treatments are presented in Figure 2.1.1.1. Mechanical finishes involve calendering, raising, mechanical stabilization, flocking, and mechanical softening. Chemical finishes for textiles are easy-care finishes, flame retardant finishes, water- and soil- repellent finishes, and antimicrobial and antistatic finishes. [11, 9]

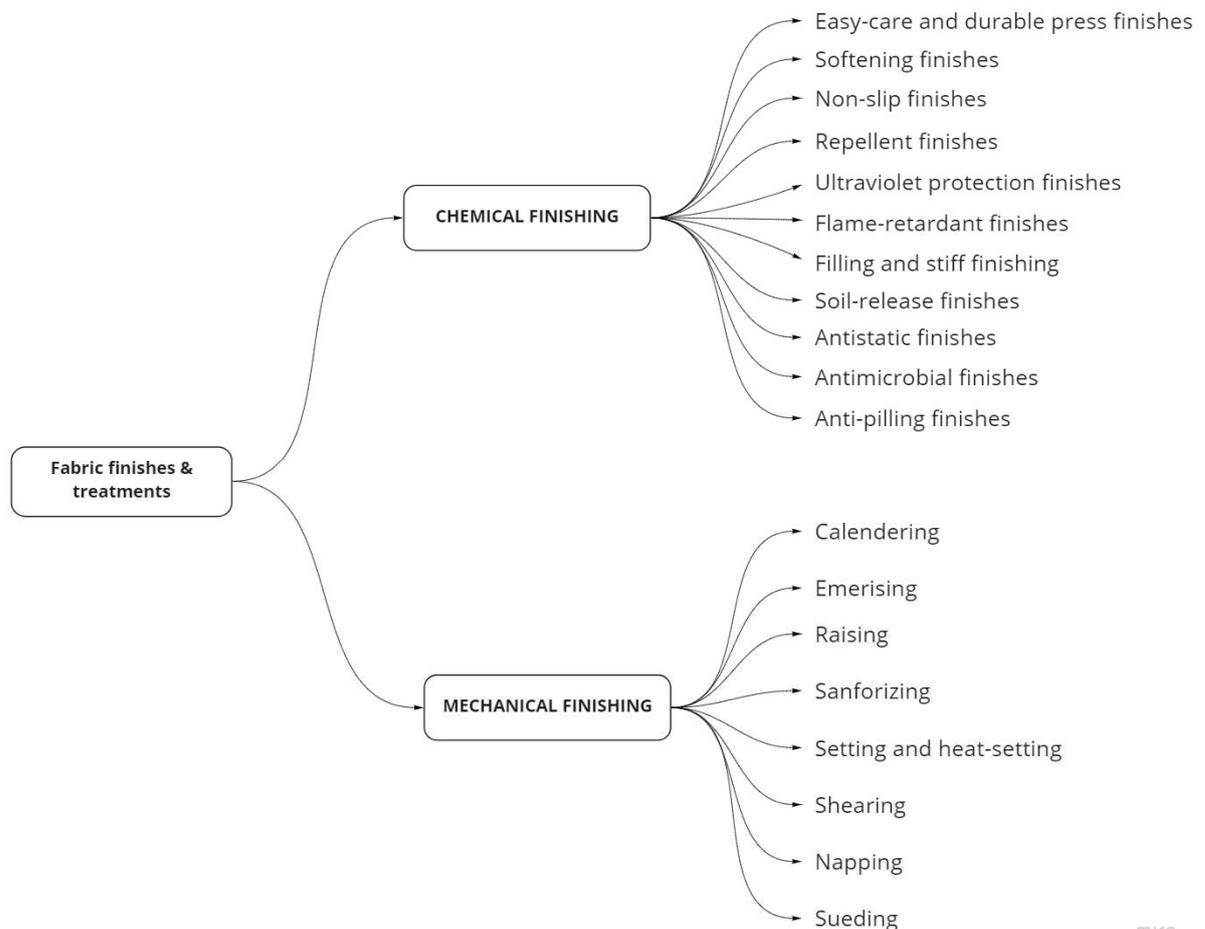
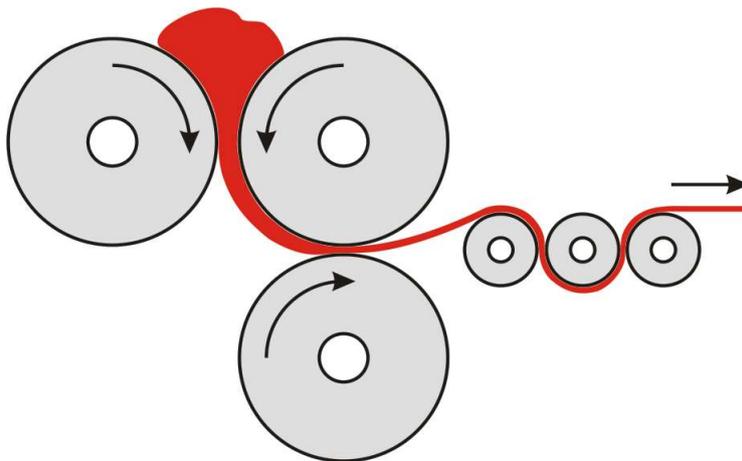


Figure 2.1.1.1 List of textile finishes and treatments [11]

Calendering

Finishing fabric with calendering method changes the appearance of the fabric surface or, in other words, increases luster. In the process, the fabric is passed between rolls under pressure. This increases the fabric's luster because the yarns are flattened, making the fabric surface smoother and improving light reflective abilities. Calendering process is explained in Figure 2.1.1.2. Calendering process effect can be affected by adding moisture to the fabric and heating the calender rolls. When adding resins to the fabric before calendering durability of the luster can be improved. [12, 11]



Picture 2.1.1.2. Calendering process [12]

Mechanical stabilization

Mechanical stabilization is most often done to prevent excessive shrinkage of fabric when exposed to heat or when laundered. Manufacturing processes increase the tension and stress in fibres, yarns, and fabrics when processing them with water and heat that allows them to give in and relax. The heat setting of thermoplastic fibres usually stabilizes these fabrics sufficiently. Woven fabrics that contain non-thermoplastic fibres such as cotton can be mechanically stabilized by preshrinking the fabric. [11]

Raising

Raising refers to lifting fibres from yarns near the fabric's surface to produce a hairy or fuzzy surface. Commonly used names for specific raising processes are teasing, napping, sueding, and shearing. Woven fabrics intended for napping may contain soft twist warp yarns, which respond to the action caused by wires. The napping technique uses small wires to pluck fibres from the yarns near the fabric surface, which is shown in Figure 2.1.1.3. [13, 11]

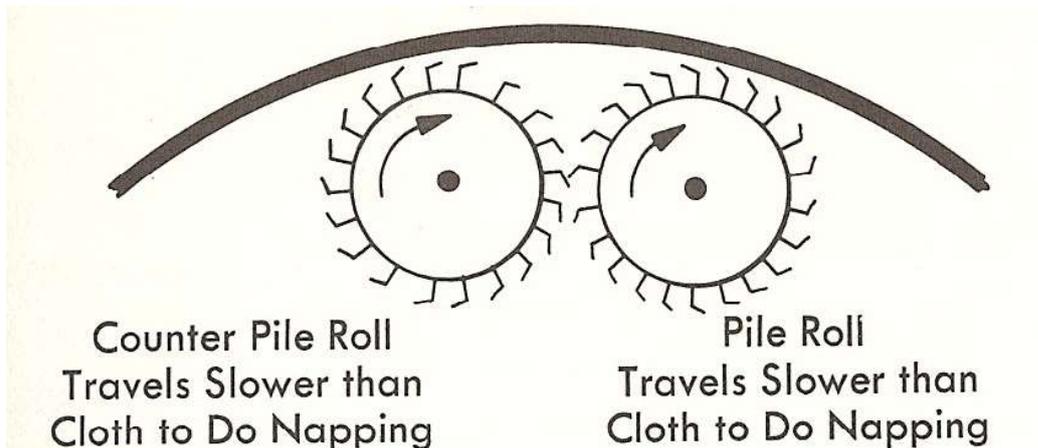


Figure 2.1.1.3. Napping rolls [14]

Mechanical softening

If yarns and fibres are not flexible and can move freely in the fabric structure, fabrics become stiff. Plus, drying fabrics under tension tends to set fibres, making them stiffer. And chemical agents make fabrics even stiffer because they bind fibres and yarns together. Mechanical softening consists of bending, flexing, or pounding the fabric to cause adhesions between the fibres and yarns to break. For mechanical softening, a variety of machines can be used. [15, 11]

Flocking

Gluing short fibres to the surface of the base fabric is called flocking. Flock can be applied to the entire surface of the fabric to produce a raised surface or to only certain areas for producing patterns. At first, adhesive is applied, and then the fabric passes the flock applicator, where short fibres are sifted onto the fabric. Most of the flock must be perpendicular to the fabric surface to achieve the desired effect. After the flock is applied, the adhesive is cured by convection, conduction, infrared, or dielectric heating to bind the flock to the fabric permanently. The electrostatic flocking process is explained in Figure 2.1.1.4. [16, 17, 11]

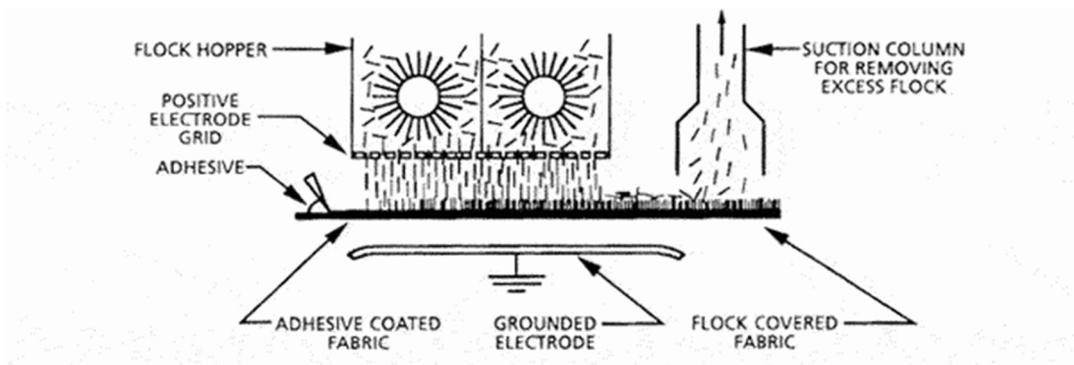


Figure 2.1.1.4 The electrostatic flocking process [17]

Easy-care finishes

Wrinkling occurs when the fibre is severely bent. Hydrogen bonds between the molecular chains in the amorphous regions of fibres break, allowing the chains to slip past one another. The bonds reform in new places and hold creases in the fibre and fabric. When adding chemical crosslinks between the molecular chains in the amorphous regions of the fibre or by deposition of a polymeric substance in amorphous areas, chain slippage can be restricted. For easy-care finishes, many terms are used, like wash-and-wear, durable press, and crease resistance. For finished fabrics, creases in the fabric may occur when wearing but are designed to smooth out after washing and drying. [11, 18]

Flame retardant finishes

If the conditions are severe enough, all organic fibres burn. Cellulosic fibres ignite at noticeably low temperatures and burn rapidly. Polyester and polyamide do not ignite but melt and recede from the flame. Nonetheless, when blended, cellulosic and synthetic fibres burn vigorously. Often a mixture with different fibres will burn more quickly than a fabric made from only individual fibres. Flammability standards, in general, require textile products to be self-extinguishing. That means that material cannot support combustion if the ignition source is removed. Flame retardant finishes are usually applied by the pad-dry-cure process. The fabric is treated with a mixture of antimony oxide, a chlorinated wax, and a binder chemical. [11, 19]

Water and soil repellent finishes

By limiting the wettability of a fabric, repellency is attained, meaning that a substance does not spread on the fabric surface. Some essential water-repellent finishes are wax emulsions, pyridinium compounds, silicones, and fluorochemicals. These finishes may be used to impact a wide variety of both natural and artificial fibres. The difference between repellent finishes is the final bond with the fibres or fabric. In wax emulsion finishing, the film of polymers or crosslinks reacts to the fabric. The pyridinium compound reacts with cellulose leaving its hydrocarbon group attached to the fibre, making the fibre surface more hydrophobic and water-repellent. Fluorochemical finishes impact both water- and oil-repellency. Fluorocarbon groups must fully cover the surface of the fibres to work. [11, 20]

Soiling is mainly the result of adhesion and physical entrapment of particulate matter onto the fabric. Particulate matter mixed with oily soil holds chemically to hydrophobic fibres making its removal more difficult. Soil release finishes make the fibre's surface more hydrophilic so that adhesion between the fibre and hydrophobic soil is lowered.

High application temperature for chemical substances is required to achieve good penetration and bonding of agents to fibres. [11]

Antimicrobial finishes

The growth of bacteria and fungi in textile fabrics is usually undesired. Laundering with hot water and disinfectants such as chlorine or peroxygen bleaches destroys many microorganisms in fabrics. Antimicrobial agents are chemicals that prevent the growth of bacteria. Many chlorinated organic compounds and organometallic compounds contain copper, silver, iron, manganese, or zinc, making textile materials resist microorganism growth. However, many of these agents are toxic to higher organisms. [11, 21]

1.2 Waste minimization in textile finishing processes

In general, minimization of waste from finishing formulations or coating pastes can be achieved by taking into account the following process aspects [22]:

- limitation of use of water,
- reduction of chemicals,
- minimizing the emission of toxic substances.

For this reason, a good choice of chemicals is required: substituting hazardous substances, using biobased, biodegradable, or reusable materials, and finally looking into new technologies that use no water. Currently used wet finishing processes produce waste containing organic and inorganic compounds. The effluents are rich in chemicals, of which some are persistent or resistant to water treatment methods. Removal of these substances from wastewater is expensive and difficult to achieve. Because of this, effluent segregation and source reduction methods are preferred as economically attractive alternatives. New finishing processes are developed to reduce energy consumption substantially and use a limited amount or no water because traditional fabric finishing processes require vast amounts of energy and water. [22]

Finishing textiles with water-free technologies assure that no water is present in the finishing process. Thus no water emission occurs, resulting in a more controlled environment for adding chemical finishing to textiles. In general, removing water-based treatment from finishing processes helps lessen the effect on the environment by lessening energy costs and removing the drying step of the manufacturing. With water-

free technologies, exposure to chemicals in textiles happens under more controlled conditions. [22]

1.3 Plasmatechnology

Plasma is defined as a partially or wholly ionized gas with an equal number of positively and negatively charged particles. It offers an energy-efficient and economical alternative to the classical wet finishing process. Within plasma processes, a high reactive gaseous phase interacts with the surface of a substrate. In principle, all polymeric and natural fibres can be plasma treated. As textile manufacturers and end-users alike are exploring different ways to improve the surface properties of natural and artificial fibres, a similar result to conventional pre-treatment with the chemical can be achieved with a shorter treatment time. During plasma treatment process, no wastewater is generated, very minimal or no amount of chemicals are needed, and the textile stays dry. Further advantages of plasma technology express short treatment time and low temperature for application. The effectiveness of the treatment depends on the nature of the plasma gas used. [23, 24, 25, 26]

The plasma treatment shortens the process time compared with conventional pre-treatment with chemicals, with similar achieved end-results. Over the past three decades, low-temperature plasma technology has been the focus of much research to improve the surface properties of polymeric materials without changing the bulk properties. There also has been research to improve wettability, water-repellency, soiling, soil release, printing, dyeing, and other finishing processes of textile fibers and fabrics by using plasma technology. In most of these studies, two significant discharges have been considered: high-frequency discharge (low-pressure plasma) and low-frequency discharge (corona discharge). [25]

Conventional wet processing of fabrics, such as de-sizing, scouring, bleaching, dyeing, and finishing, consumes large amounts of water and energy and eventually causes water and air pollution. Therefore, pre-treatment and finishing fabrics by plasma technologies have become popular because such techniques offer numerous advantages over the traditional wet chemical processes. In general, plasma treatment is a dry surface modification process allowing modification of surface properties without changing the bulk properties of the substrate. The low power needed to maintain plasma gas activation in the chamber saves large quantities of electrical energy. Besides, from an economic point of view, the small consumption of chemicals makes the process superior for the textile industry. The extensive work in the last two decades on the possible

application of plasma to different textile materials indicates that this technology can be efficiently exploited for obtaining plenty of advantageous effects, including high efficiency, economic feasibility, environmental acceptability, and flexibility. [25]

1.4 Nanotechnology

The recent advancement in the field of fabric finishings can extensively be written down as the achievements in the area of nanotechnology. By combining the nanoparticles with the organic and inorganic compounds, the surfaces of the fabrics treated with abrasion resistant, water repellent, ultraviolet, electromagnetic, and infrared protection finishes can be appreciably modified. Nanotechnology can best be described as activities at the level of atoms and molecules that have applications in the real world. In the commercial product range, nanoparticles commonly are in the range of 1 to 100 nm. This is one of the most common ways to create stain and water resistance in the textile industry. The coating compositions that can modify the surface of textiles are usually composed of nanoparticles, a surfactant, ingredients, and a carrier medium. [27, 28, 26]

The impact of nanotechnology in the textile finishing area has brought up innovative finishes and new application techniques. Particular attention has been paid to applying chemical finishing by nanomaterials in textiles. More efficiently, discrete molecules or nanoparticles of finishes can be brought individually to designated sites on textile materials in a specific orientation and trajectory through thermodynamic, electrostatic, or other technical approaches. Nanoparticles such as metal oxides and ceramics are also used in textile finishing altering surface properties and imparting textile functions. Because nanosize particles are transparent and do not blur the color and brightness of the textile substrates, preventing nanoparticles from aggregating is the key to achieving the desired performance. Nanoparticles can be pre-engineered to adhere to textile substrates using spray coating or electroplating methods. Finishing with nanoparticles can convert fabrics into sensor-based materials. Suppose nanocrystalline piezoceramic particles are incorporated into fabrics. In that case, the finished fabric can convert exerted mechanical forces into electrical signals enabling the monitoring of bodily functions such as heart rhythm and pulse if worn next to the skin. [28]

1.5 Microencapsulation

As the textile industry moved to the 21st century, the number of commercial microencapsulations grew. Microencapsulation is a micro-packaging technique involving the deposition of the thin polymeric coating on tiny particles of solid or liquid. This process is more advantageous to conventional procedures in terms of economy, energy-saving, eco-friendliness, and controlled release of substances. The capsules embedded within a fiber or applied to the fabric's surface allow the effect to operate irrespective of the ambient conditions, even when the material is compressed or crushed. [26, 29]

Encapsulation of active ingredients for a wide range of industries is carried out for one or more of the following purposes [29]:

- a. Rendering liquids into powders to prevent clumping and improve mixing.
- b. Protecting active ingredients from oxidation, heat, acidity, alkalinity, moisture, or evaporation.
- c. Preventing ingredients from interacting with other compounds in the system, which may result in degradation or polymerization.
- d. Masking the taste of unpleasant flavors or odors.
- e. Improving handling of an ingredient before processing.
- f. Release active chemicals in a controlled or targeted fashion.
- g. Protecting workers or end-users from exposure to hazardous substances.

The choice of the available microencapsulation methods depends on the functionality of the fabric. What are the density and stability requirements for capsulated ingredients, and what processes will the capsule survive. [29]

2. ELECTROSPRAYING METHOD

The phenomenon of electrohydrodynamic atomization was first observed and recorded by William Gilbert in 1600. Around two centuries later, the terminology for electrospaying was generated. In 1882, a study theoretically evaluated the charge that a liquid droplet could carry to the greatest extent, also known as the "Rayleigh limit". This was confirmed experimentally nearly 100 years later. Electrohydrodynamic atomization technique started attracting a lot of attention in the 1990s for producing and processing micro-/nanoparticulate materials for a rich variety of applications. Nowadays, it is namely known as electrospaying. [30]

Electrospray is an electrohydrodynamic technique similar to electrospinning. This process is conducted by using a similar principle, and often in, the process is used identical apparatus. Through electrospaying is possible to produce diminutive droplets with submicron sizes through an electric field. This generates nano-sized droplets on the target material. The simplicity and flexibility of the experimental setup of electrospaying has been successfully employed to generate particulate materials with controllable compositions, structures, sizes, morphologies, and shapes. Electrospaying method used for textile coating is a method of forming fine polymer particles through electrostatic charging and attachment of particles to the substrate. In a typical electrospaying process, the main variables are applied voltage, nozzle collector distance, and solution concentration. [31, 32, 33, 30]

In this process, a solvent-based liquid passes through a nozzle where fine droplets are generated by electrically charging the liquid to a very high voltage. Electrospaying is carried out under room temperature and atmospheric pressure, and there is no further drying step required since the solidification of particles occurs instantaneously during the electrospaying process. If the ejected stream of prepared solution does not have sufficient molecular cohesion to withstand the electric pull to form nanofibre, it breaks and creates drops of the polymer. At the tip of the cone, the liquid becomes unstable as it is forced to hold more and more charge, and eventually, it disperses into numerous, micron-sized, highly charged droplets at the tip of the nozzle. As the particles fly about, they rapidly shrink as the solvent molecules in micron-sized droplets evaporate. [33, 34]

To achieve uniform electrospaying on the fabric surface, many interrelated parameters influence the outcomes during the process. By controlling pre-optimized parameters of applied voltage, flow rate, working distance, spraying mode, nozzle gauge, collection

method, environmental temperature, pressure, and humidity, different structures and morphologies of electrosprayed particles can be obtained. Electrospraying process outcomes possess various functional and structural advantages are shown in Figure 3.1. [33, 34, 35]

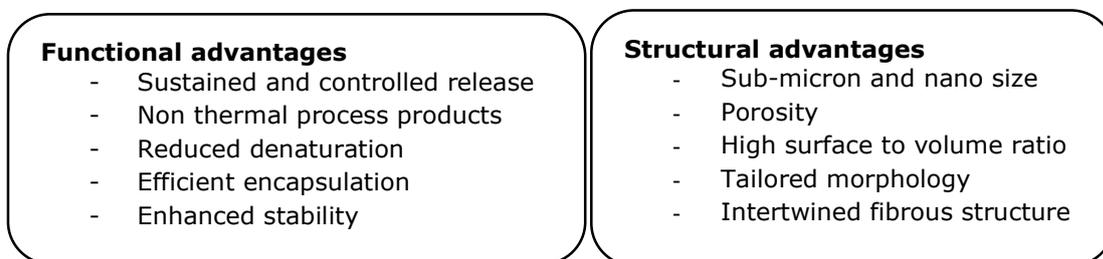


Figure 3.1 Functional and structural advantages of electrospraying process outcome [35]

From an application standpoint, electrostatic spraying is successful in non-textile applications, including solar panels, microencapsulation, electro emulsification, and fine powder formation. In the past few years, a wide range of synthetic and natural polymers in pure or blend solutions have been used for coating textile substrates. This method could also be actively used in the field of the textile coating due to the following advantages [33, 36]:

- a. Mechanical energy is not required for forming the liquid droplets.
- b. The charged droplets can be controlled by applied voltage and solution flow rate.
- c. Coating thickness is thinner than conventional textile coating techniques.
- d. Charged droplets are self-dispersing in the atmosphere, which results in no agglomeration and coagulation.
- e. An electric field can control the motion of charged droplets.
- f. The deposition efficiency of electrosprayed polymer droplets is higher than uncharged droplets.

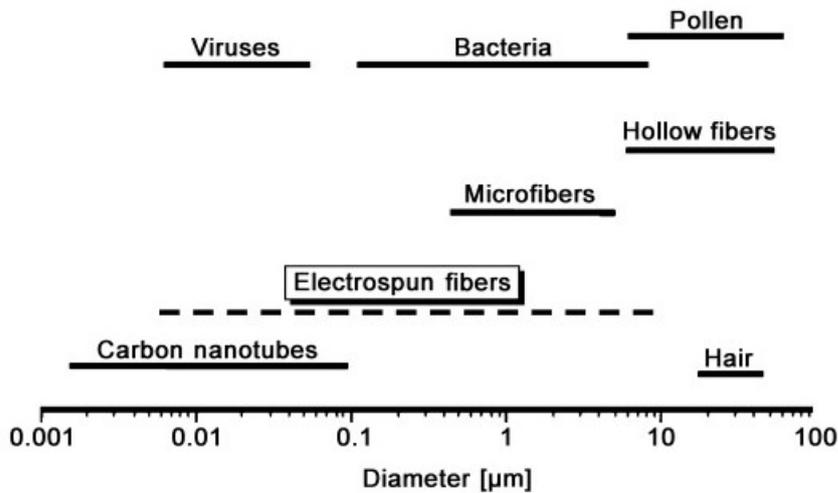


Figure 3.2 Comparison of the diameters of electrospun/sprayed fibers to those of biological and technological objects. [37]

This study of environmentally friendly textile finishing by electrospaying method is done with reference to a report written by J. M. Cuevas; B. Gonzalo; C. Rodrigues; A. Dominigues; D. Galan, and I. G. Loscertales discussing grafting electrospayed silica microspheres on cellulosic textiles. The report concluded a versatile process of covalent grafting of solid, hollow, or core-shell submicron capsules onto cellulosic textiles by conventional dyeing processes. [38]

2.1 Experimental procedure of electrospaying

Electrospaying comprises a syringe, syringe pump, nozzle, high-voltage generator, and collector. A light source and a high-resolution camera connected to a computer are also often used in electrospaying to facilitate observation and analysis. At first, an electrospaying solution is added to a syringe and further transported to the nozzle under an adjustable flow rate via a syringe pump. The nozzle is linked with one end of the high-voltage generator that is primarily positive. The other end of the high-voltage generator is connected to the grounded collector. The distance between the nozzle's tip and collector usually ranges from a few centimeters to several tens of centimeters. Equipment setup for electrospaying process is given in Figure 2.1.1. Due to the strong electric field between nozzle and collector and the Coulomb repulsions, the electrospaying solution will be stretched and further dispersed into smaller droplets ejected from the tip of the Taylor cone. During the flight of droplets to the collector, solvents in these droplets will evaporate, and solid particles can finally be obtained in the collector. [34, 36]

Further, these particles could be placed into a vacuum oven to remove the residual solvent if needed. Sometimes, to further stabilize the process of electro spraying and cone-jet, a ring-shaped electrode is placed between the nozzle and the collector. Meanwhile, the size and distribution of electro sprayed particles will change with the electric field's changing among the nozzle, and collector. [34, 36]

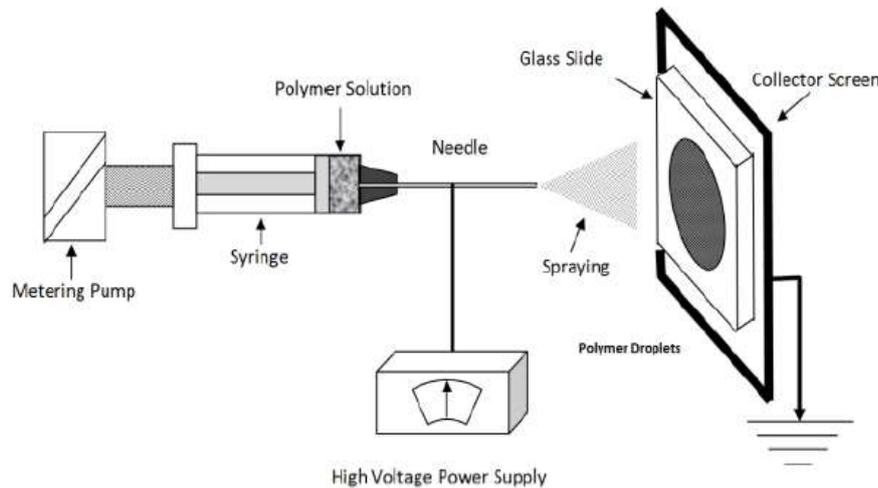


Figure 2.1.1 Equipment setup for electro spraying process [39]

The electro spraying process is governed by six types of forces [34]:

- the gravity of polymeric solution,
- the electrostatic force generated from an external electric field between nozzle and collector,
- Coulomb repulsion force among adjacent charged carriers on the surface of jet,
- a viscoelastic force of polymeric solution,
- surface tension in the interface between air and liquid, and
- the frictional force between charged jet and surrounding air.

When polymeric solution flows out of the nozzle, the charge distribution and carried charge quantity on the surface of the polymeric solution will change in varying degrees according to its electrical conductivity and dielectric constant because of the polarization effect coming from the external electric field. At the same time, initially, the uncharged liquid becomes charged jet and is further stretched towards the direction of electrostatic attraction. However, compared to gravity and electrostatic force that accelerates the moving and stretching of polymeric solution from the nozzle to the collector, the surface tension and viscoelastic ones prevent this moving and elongation because of their opposite behavior on the electro sprayed solution. When these forces reach a balance at a specific range, the droplets at the tip of the nozzle are stretched from the spherical shape into the conical surface. [34]

3. DIFFERENT TESTING METHODS OF WATER REPELLENT FINISHED TEXTILES

3.1 Characterization of woven fabrics

Woven fabric is made in a pattern of interlacing yarns or threads at a correct angle from a fabric. Warp yarns are longitudinal yarns, and the lateral threads are called weft yarns. The characteristics of woven fabrics depend on the weaving method of making the fabric and which fibre yarns are interwoven. Fabrics woven in plain weave are the simplest types of weaving, which allows to produce from very coarse and heavy fabric to finest and lightest. [40]

There are three types of woven fabric fibre: pure woven fabric, interwoven fabric, and mixed or blended textile fabric. Pure woven fabrics are made from the same kind of fibre for warp and weft yarns, also referred to as pure spun yarn woven fabric. For interwoven fabrics, warp and weft yarns have different fibre compositions. Blended fabrics involve using two or more varieties of fibre blended yarn woven fabric. Two of the most popular textile fibres globally are cotton and polyester for fabric weaving. [41]

Cotton fabric is one of the most versatile and commonly used textile fibre worldwide. It is made from natural cellulosic fibres collected from plant seed hairs. Cotton textiles are breathable and absorb water but typically dry slowly and wrinkle easily. They can be damaged by mildew and prolonged exposure to sunlight. However can withstand heat, detergents, and bleach. [42]

Polyester fabric is a synthetic woven material made from the polymerization of petroleum-derived ethylene glycol and purified terephthalic acid. Its most characteristic properties are durability, moisture-resistance, shape-retaining, and coarseness. The environmental impact of most polyester yarns is quite the opposite of natural fibres because, typically, it is non-biodegradable—polyester doesn't easily break down and is not generally biodegradable. [43]

3.1.1 Substrate content analysis

A content analysis methodology is to objectively and systematically study the content of communications in various formats. Researchers use content analysis to determine

the presence of certain concepts within given data to qualify and analyze for bias or partiality. [44]

Optical microscopy analysis

Microscopy analysis is essential to understanding the microstructure or nanostructure of materials. In the optical microscope, as the light passes through the specimen, it is rendered one-half wavelength or 180 degrees out of step with the direct light that has passed through undeviated. The one-half wavelength out of phase, caused by the specimen itself, enables this light to cause destructive interference with the direct light when both arrive at the intermediate image plane located at the fixed diaphragm of the eyepiece. The eye lens of the eyepiece further magnifies this image, which finally is projected onto the retina, the film plane of a camera, or the surface of a light-sensitive computer chip. [45]

Scanning electron microscopy analysis

SEM (scanning electron microscopy) gives a three-dimensional response to the specimen surface. This technique uses a 2 to 3 nm spot of electrons to enhance linear magnification by using an electron beam as source radiation. SEM scans the surface of the specimen and, over time, as the entire surface of the specimen is examined, produces an image of the sample due to interactions between the electron beam and sample surface. Its high depth of field is an essential feature of SEM as it allows to analyze fracture surfaces of a specimen. [46, 47]

Contact angle measurements

CAM (contact angle measurement) is an essential part of determining surface tension. Performing CAM for surface energy determination is an appropriate method to investigate textile surface characteristics, such as adhesion properties to textile surfaces. This measurement was done by following ISO 19403-2:2017, Determination of the surface free energy of solid surfaces by measuring the contact angle. This standard is applicable for the characterization of substrates and coatings. A minimum of at least three drops of distilled water is applied with a needle to test the flat specimen surface. Measurement is recorded for 45 seconds, and results are calculated by dividing the polar and dispersive fractions. Conditions for testing must meet a relative temperature of (23 ± 2) °C and humidity of $(50 \pm 5)\%$ minimum of 16 h before testing according to ISO EN 23270:2000. [48, 49]

Determination of mass per unit area using small samples

Fabric mass per unit area is expressed either as grams per square meter or grams per linear meter. Determination of mass per unit area measurements using small samples was done according to ISO EN 12127:2000, which applies to woven and knitted fabrics. Before cutting the pieces, the fabric must be kept in a flat tension-free state for at least 24h and under conditioning to achieve the standard atmosphere for measuring, performed following ISO 139:2005/A1:2011 defining standard atmospheres for conditioning and testing. There can be no folds, creased areas, or spots with a visible fault on samples. [50, 51]

Samples are cut following the pattern shown in Figure 4.1.2.1. Each test specimen is weighted individually to the nearest value of 1mg. The expression of results is calculated with the formula $M = \frac{m \times 10000}{A}$ where m is the mass of a conditioned sample, and A is the area of the same test sample. Measurement results are expressed in grams per square metre. [50, 51]

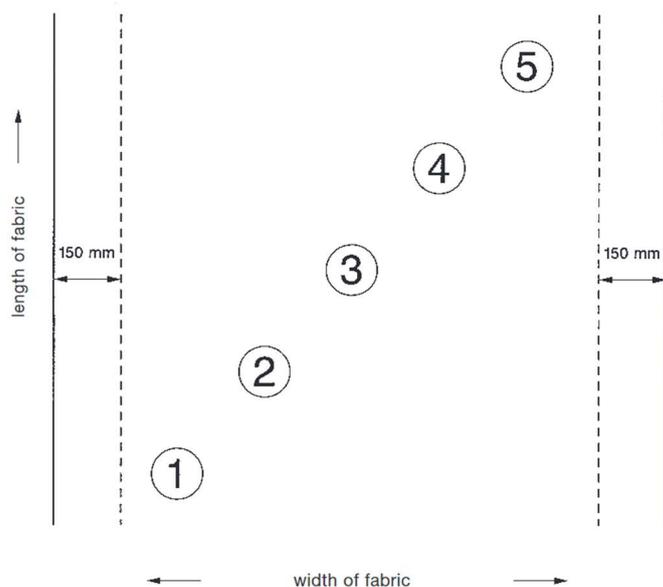


Figure 4.1.2.1. Example of cutting samples for testing in a laboratory [51]

Determination of number of threads per unit length

Determination of number of threads per unit length was done in accordance with ISO EN 1049-2:2000, Determination of a number of threads per unit length. Beforehand textile samples were put under conditioning according to ISO 139:2005/A1:2011 defining standard atmospheres for conditioning and testing. A number of threads per unit typically expresses the coarseness or the fineness of the fabric. Thread count is a reliable guide for fabric quality up to a point, but a higher thread count does not always

mean better quality. Measurements are done according to method C by traversing a thread counter suitable for all fabrics. [50, 52]

Five samples from the fabric must be cut in the weft and warp directions. Threads are counted from a thread in the weave to the appropriate minimum measuring distance. The minimum distance for measuring distance for 25 to 40 number of threads per centimeter is with a measuring length of 3 centimeters given according to ISO 7211. [50, 52]

3.2 Determination of the abrasion resistance of fabrics by the Martindale method

The abrasion resistance is determined following the determination of the abrasion resistance of fabrics by the Martindale method – Part 1: Martindale abrasion testing apparatus ISO EN 12947-1:2001. This testing method subjects a circular specimen to a defined load and rubs it against an abrasive material. The lower specimen holder contains an abrasive medium, and the upper holder contains test samples used in a freely rotatable plate around its own axis. [53]

For testing, the abradant fabric is used, a polyetherurethane foam material with mass per unit area less than 500 gm^{-2} , and auxiliary material. Upper holder circular pieces are cut with a diameter of 38.0 mm and placed into the holder insert. Lower specimen holder specimens are cut with a diameter of 140.0 mm. Loading pieces used for testing apply nominal pressure of 9 kPa or 12 kPa during testing. [53]



Figure 4.2.1 Abrasion and pilling testing machinery [54]

3.3 Determination of resistance to surface wetting

Determination of resistance to surface wetting is performed according to ISO EN 4920:2012, which describes a spray test for determining water-resistant or water-repellent properties to surface wetting. The spray test consists of a (150 ± 5) mm funnel held vertically with a metal nozzle connected to the end of the stem by rubber tubing. Distilled water with an amount of 250 ml is sprayed onto a specimen on a ring placed at an angle of 45° . The duration of flow shall be between 25 s and 30 s. The specimen holder consists of two plastic rings that fit into each other to secure the specimen. Test specimens have to be under conditioning for at least 4 h. The machinery used for spray rate testing is shown in Figure 4.3.1. [55]



Figure 4.3.1 Spray rate testing machinery setup [56]

After spraying, the test specimen holder must be taped against a solid surface to remove excess drops that do not affect surface wetting. Results of the spray test are held by visual determination according to AATCC photographic scale shown in Figure 4.3.2. [55]

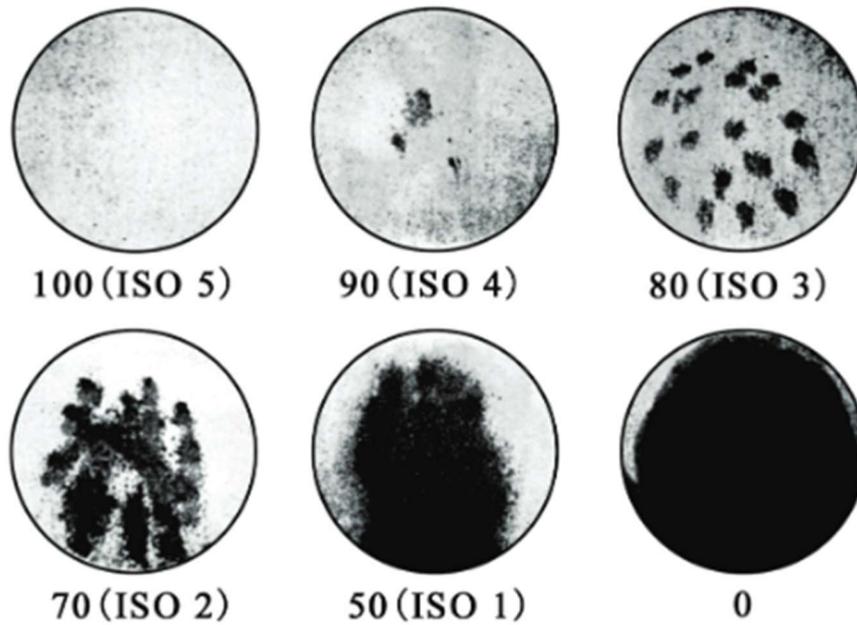


Figure 4.3.1 Descriptive rating scale for ISO EN 4920:2012

3.4 Domestic washing procedures for textile testing

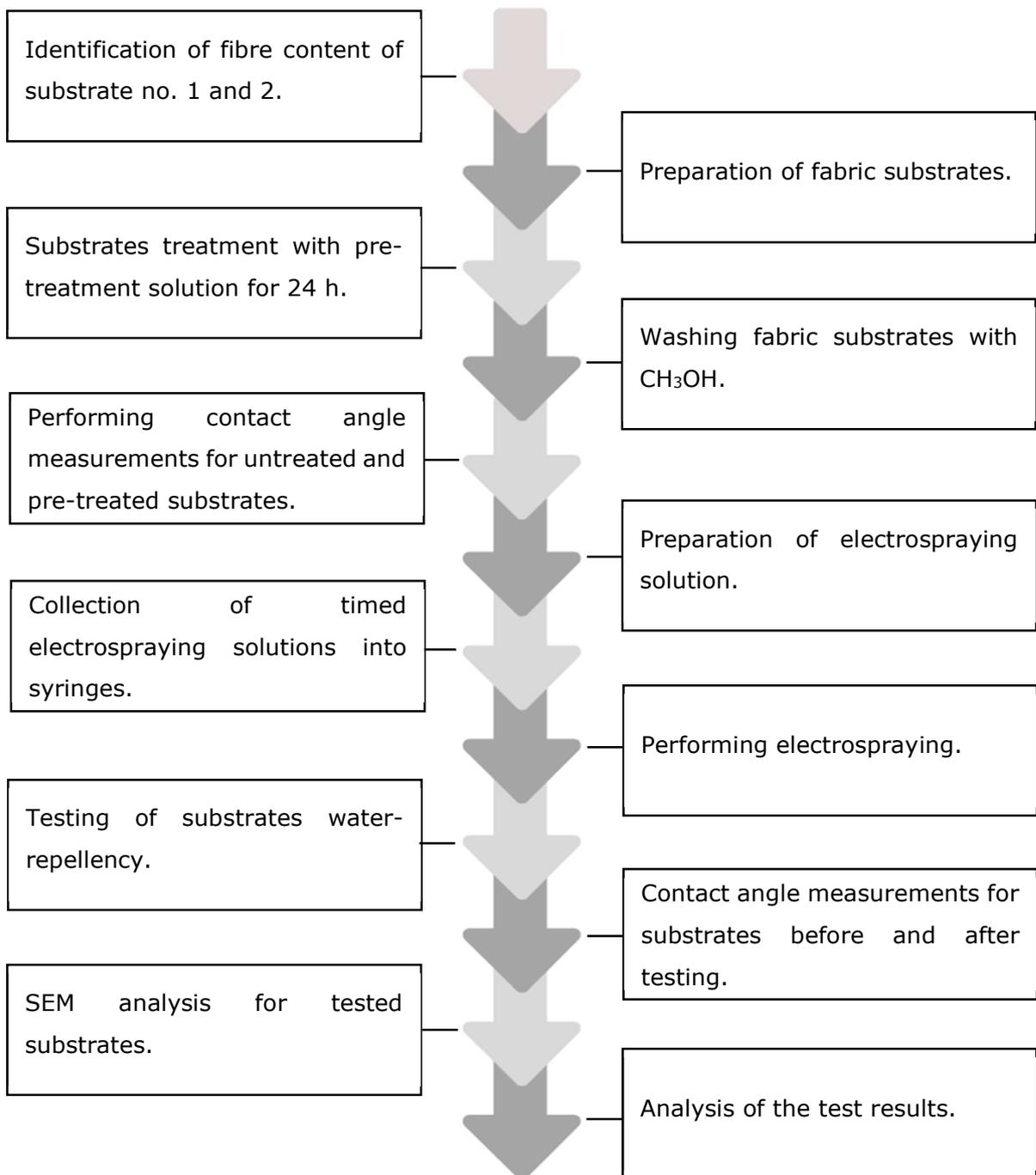
Washing procedures for substrates are performed according to ISO EN 6330:2021. Domestic washing and drying procedures for textile testing are applicable to textile fabric, garments, and other textile articles. Used detergent for testing is according to detergent type 2, suitable for machines type a and type b. Containers for washing are prepared with a laundry solution of 150 ml with a detergent dosage of (20 ± 1) g. As ballast inside the container for testing must be added beads to fill the mass required for washing. Tumbler washing temperature is set to 30 °C, and water pressure shall be higher than 150 kPa. A number of test specimens required for testing come from a number of different substrates or materials used for the study. The apparatus for conducting laundry testing is shown in Figure 4.4.1. [57]



Figure 4.4.1 Testing apparatus for color fastness to laundry testing [58]

4. EXPERIMENTAL PLAN AND THE AIM OF THE STUDY

The electrospaying study was performed in the TalTech Laboratory of Polymers and Textile Technology. The method was imposed on 100% CO (cotton) and 100% PES (polyester) fabrics to enhance the textile water-repellent properties with the electrospaying method. Experimental plan constructed for the study is shown in Graph 4.1.



Graph 4.1 Experimental plan for the study

5. MATERIALS AND METHODS

5.1 Materials

Two different substrate materials with different fibre content were chosen for the study, which were later studied under similar conditions to determine effectiveness and propriety of electrospaying.

5.1.1 Substrate materials

Substrate materials were purchased from a fabric store and are commercially available. Substrate materials used for the study are presented in Table 5.1.1.1. Substrate number 1 is white 100% CO fabric in plain weave, and substrate number 2 is white 100% PES fabric in plain weave..

Table 5.1.1.1 Substrate materials

| Material | Fibre content | Weave type |
|-----------------|---------------|-------------|
| Substrate no. 1 | 100% CO | Plain weave |
| Substrate no. 2 | 100% PES | Plain weave |

5.1.2 Chemical components

For substrates, pre-treatment has selected a solution containing CH₃OH (99%) (methanol) and APTES (3-Aminopropyl triethoxysilane). To perform electrospaying was chosen to use TEOS (tetraethyl orthosilicate), EtOH (ethanol), H₂O (water), and HCl (37%) (hydrochloric acid). Substrates used in the study and substrate acquiring places are presented in Table 5.1.2.1.

Table 5.1.2.1 Chemical components of the solvents

| Solution types | Chemical components | Supplier |
|-------------------------|--------------------------|------------------|
| Pre-treatment solution | CH ₃ OH (99%) | Keemia kaubandus |
| | APTES | Alfa Aesar |
| Electrospaying solution | TEOS | Sigma-Aldrich |
| | EtOH | Keemia kaubandus |
| | H ₂ O | Laboratory |
| | HCl (37%) | Stanchem |

5.2 Methods

5.2.1 Characterization of woven fabrics

To determine the fibre content of test substrates and suitability for the experimental part, several tastings were performed according to ISO standards – determination of mass per unit area using small samples and determination of the number of threads per unit length. Also, different microscopical analyses (light microscopy and SEM) were performed for substrate materials.

Determination of mass per unit area using small samples

According to ISO EN 12127:2000, to determine mass per unit area of fabric substrates, small samples had to be cut from textile substrate 1 and substrate 2. In total, five samples in different places in a pattern where no warp or weft threads are matching on the samples from both fabrics by the following the way given in Figure 1. Samples are in standard size cut with a circular cutting device to avoid distortion of the material. All test specimens were measured in a measuring container.

Determination of number of threads per unit length

The number of threads per unit length was determined according to ISO EN 1049-2:2000. Five samples in the warp direction and five samples in the weft direction were cut in the size of 5x10cm. Samples were cut in various places of fabric. The minimum measuring distance for thread count given was 3.0 cm which was done according to method A specified in ISO 7211.

5.2.2 Substrate content analysis

Content analysis for substrate materials was used to determine fiber content's accuracy. With SEM, it was possible to examine the fabric surface conditions before pre-treatment, analyze the changes to electrosprayed substrates, and evaluate surface changes to different testing methods afterward.

Optical microscopy analysis

Optical microscopy was done in Taltech Laboratory of Polymers and Textile Technology using a microscope from Euromex series BioBlue with a magnification of 100x and 400x, shown in Figure 6.2.2.1.1. At analysis time, the room was under conditioning determined by ISO 139:2005/A1:2011, which defined standard atmospheres for conditioning and testing.

Scanning electron microscopy analysis

The surface morphology of the electrosprayed specimens was studied using a scanning electron microscope. SEM analysis was performed in TalTech in the Department of Mechanical and Industrial Engineering with tabletop microscope TM-1000 from Hitachi to untreated, electrosprayed, and tested specimens.

Contact angle measurements

The hydrophobic properties of test specimens were studied under contact angle measurements. CAM was done in the laboratory at Taltech Laboratory of Wood Technology. All measurements were performed according to ISO 19403-2:2017 with machinery from Dataphysics with Contact Angle System OCA support for programming given in Figure 6.2.2.3.1. The room temperature for testing was 20° with a relative humidity of 37%. Measurements were done on all specimens included in this study.

5.3 Electro spraying setup

Pre-treatment for substrates surface was done with 50 ml of CH₃OH and 2 ml of APTES, which was under vigorous stirring for 24 h. Pre-treatment is essential to remove any existing residue and deteriorate the surface of the substrate for better adhesion to the electro spraying solution. Afterward, substrates were washed with CH₃OH to remove any leftover residue of APTES.

For the electro spraying process, four solutions with different crosslinking stages were prepared. Solvents are shown in Table 5.3.1, for a solution was added TEOS of 26 g in H₂O of 3.375 g, EtOH of 5.75 g, and hydrochloric acid (37%) of 0.0625 g. It was stirred in a sealed flask at room temperature, which was about 23 °C, for 10 minutes and warmed to 80 °C for further 10 minutes. Afterward, 2ml of the solution was taken for

electrospraying on the first test specimen. This step was repeated after 10 minutes to collect four different morphology solutions up to 40 minutes of stirring.

The Electro spraying study setup is presented in Figure 5.3.1, and high-voltage equipment is shown in Figure 5.3.2. Electro spraying was performed in the following steps:

1. A syringe with 2 ml of solution was placed on the machine that controls the amount of solution sprayed onto the fabric substrate.
2. This was set to spray out 5ml of solution in 1h.
3. Onto collector plate was placed textile specimen, which was grounded so electricized droplets from syringe would spray directly onto the textile substrate.
4. To the syringe needle was attached a voltage clamp with 15 kV, and the process was started.
5. With electro spraying nanoparticle layer is placed on the specimen.

Table 5.3.1 Electro spraying solutions used in the study

| Solution | Stirring time [min] | Solvents | Amount [g] |
|-----------------|----------------------------|------------------|-------------------|
| Solution no. 1 | 10 | TEOS | 26 |
| | | EtOH | 5.75 |
| | | H ₂ O | 3.375 |
| | | HCl (37%) | 0.0625 |
| Solution no. 2 | 20 | TEOS | 26 |
| | | EtOH | 5.75 |
| | | H ₂ O | 3.375 |
| | | HCl (37%) | 0.0625 |
| Solution no. 3 | 30 | TEOS | 26 |
| | | EtOH | 5.75 |
| | | H ₂ O | 3.375 |
| | | HCl (37%) | 0.0625 |
| Solution no. 4 | 40 | TEOS | 26 |
| | | EtOH | 5.75 |
| | | H ₂ O | 3.375 |
| | | HCl (37%) | 0.0625 |



Figure 5.3.1 Electro spraying experiment set up



Figure 5.3.2 High-voltage equipment

5.4 Different testing methods for finished textiles

Testing of finished textiles helps to understand how finishing affects usable fabric. This chapter is covering what were different testing methods performed for finished textiles after the electro spraying process.

5.4.1 ISO EN 12947:2001 – Determination of the abrasion resistance of finished fabrics with the Martindale method

Determination of abrasion resistance of finished fabric by the Martindale method is performed according to ISO EN 12947-1:2001 in TalTech Laboratory of Polymers and Textile Technology. Testing was performed on 8 different specimens. Samples placed in the holder were in the size of 38.0 mm; the exact sizes apply to polyetherurethane

foam. Martindale abrasive cloths, wool felt abrart fabric and polyurethane foam are from James Heal, made in England. Wool felt and abrart fabric is placed in the lower holder with a sample diameter of 140.0 mm. The nominal pressure applied for testing is 9 kPa with 1000 rubs. [53]

5.4.2 ISO EN 4920:2012 – Determination of resistance to surface wetting

Resistance to surface wetting is a test to determine the water-repellency of materials using a "shower test". Determination of resistance to surface wetting measurements was done according to ISO EN 4920:2012 in TalTech Laboratory of Polymers and Textile Technology. James Heal's Spray Rate tester combines the comprises of stainless-steel framework incorporating a funnel. The spray nozzle is mounted on the neck of the funnel. The precision spray nozzle contains 19 holes – one central hole surrounded by two concentric rings of 6 and 12 holes.

The test was performed on 10 different finishing test specimens. Samples were placed inside the metal ring for secure positioning, and 250 ml of distilled water was sprayed onto them. Afterward, a visual analysis of the specimen was performed according to the descriptive rating scale for ISO EN 4920:2012, shown in Figure 4.3.1.

5.4.3 ISO EN 6330:2021 – Domestic washing procedures for textile testing

Domestic washing procedures were performed in TalTech Textile laboratory following ISO EN 6330:2021. Testing was carried out in separate containers on 8 test specimens. Each container included four ballast balls and 150 ml of detergent solution. At the time of testing, the washing machine temperature was set to 30 degrees for testing of 30 minutes.

6 RESULTS AND DISCUSSION

6.1 Characterization of substrate materials

6.1.1 Determination of mass per unit area using small samples

Mass per unit area for substrates was measured to determine the suitability of substrates for performed study to conclude the weight of the materials. The results for the determination of mass per unit area using small samples for substrate no. 1 is 191 g/cm². The calculated standard deviation for mass per unit area is 4. Results for substrate no. 1 are given in Appendix 1.1. The results for determining mass per unit area using small samples for substrate no. 2 is 188 g/cm², with a standard deviation of 1. Results for substrate no. 2 are shown in Appendix 1.2.

6.1.2 Determination of number of threads per unit length

The results for determination of the number of threads per unit length performed for substrate no. 1 in the weft direction is 75 threads per unit length with a standard deviation of 1. In the warp direction 74 threads per unit length with a standard deviation of 2. The outcome of measurements for substrate no. 1 is given in Appendix 2.1. The results for the determination of number of threads per unit length performed for substrate no. 2 in the weft direction is 68 threads with a standard deviation of 3, and in the warp direction 70 threads with a standard deviation of 2. The outcome of measurements performed for substrate no. 2 is shown in Appendix 2.2.

6.1.3 Substrate content analysis

Optical microscopy

Optical microscopy analysis was performed on substrate no. 1 and substrate no. 2 to make sure that the fabric content was correct — the analyzed fabric substrates were white, 100% CO and 100% PES without finishing. Figure 6.1.3.1 and Figure 6.1.3.2 present fabric substrate no. 1, which is analyzed under 100x and 400x magnification to

conclude that the fibre content is 100% CO. Under microscopy, cotton fibres appear as flat twisted ribbon-like fibres.

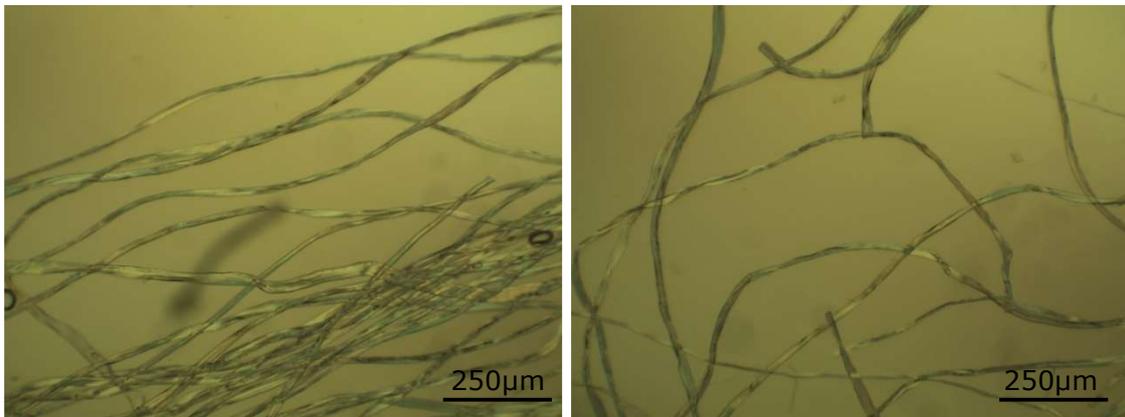


Figure 6.1.3.1 Microscopy pictures of 100% CO warp (left) and weft (right) with a magnification of 100x.

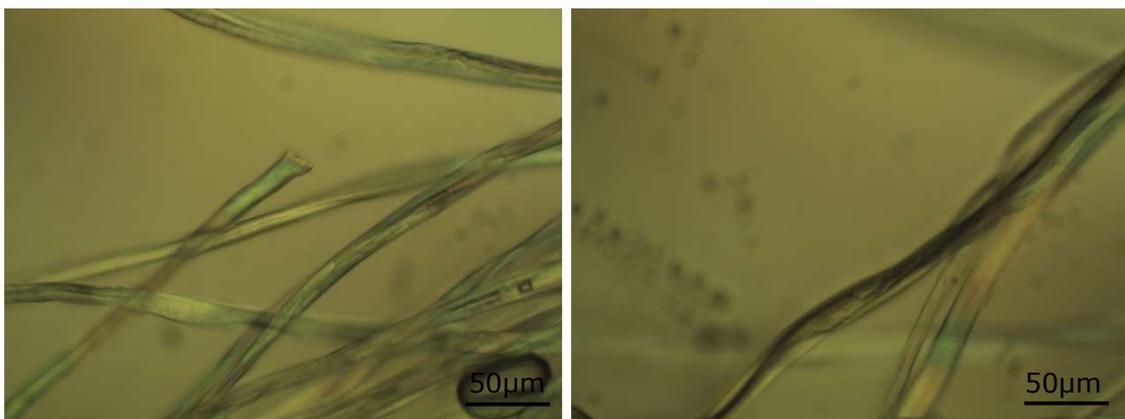


Figure 6.1.3.2 Microscopy pictures of 100% CO warp (left) and weft (right) with a magnification of 400x.

Figure 6.1.3.3 shows fabric substrate no. 2 under 100x magnification, and Figure 6.1.3.4 shows substrate under 400x magnification for understanding that it is 100% PES. Polyester fibres appear structureless and very uniform under microscopy, while cross-sectional views of the fibres can vary from round, triangular, to square-shaped.

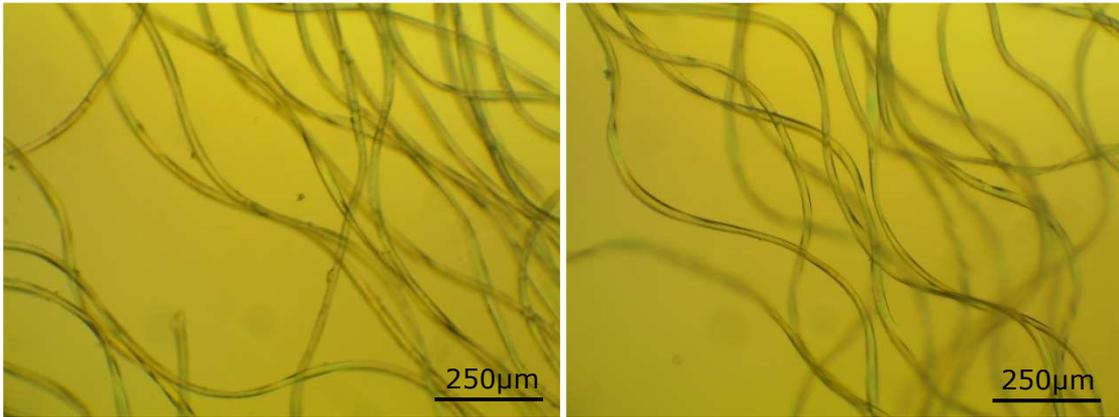


Figure 6.1.3.3 Microscopy pictures of 100% PES warp (left) and weft (right) with a magnification of 100x.

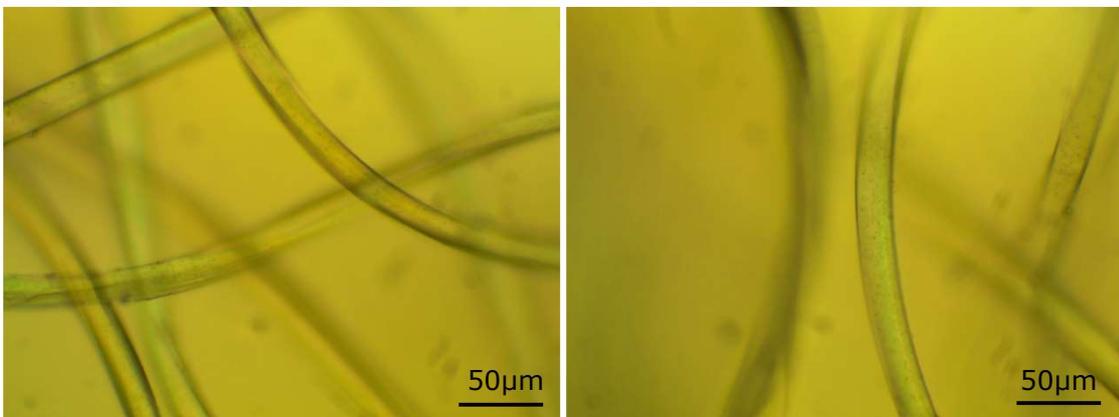
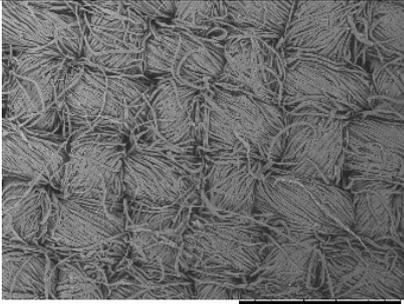
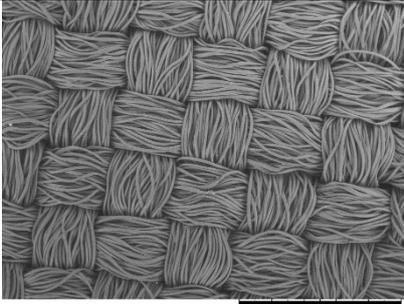
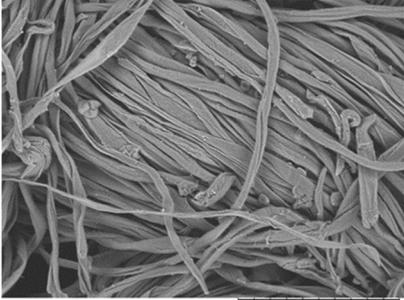
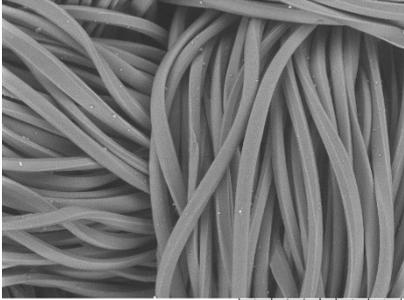


Figure 6.1.3.4 Microscopy pictures of 100% PES warp (left) and weft (right) with a magnification of 400x.

SEM analysis

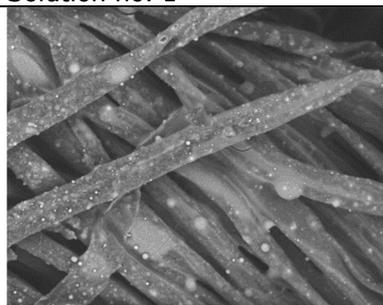
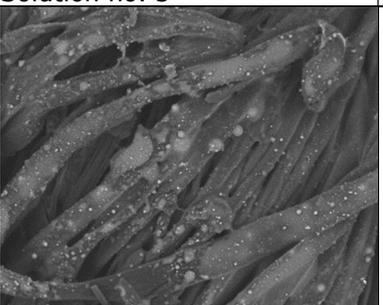
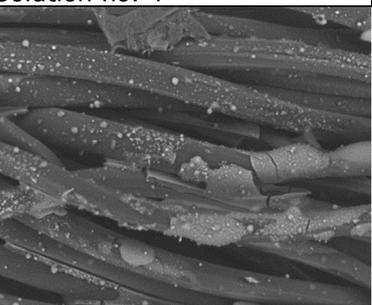
First, SEM analysis was conducted on untreated substrates that were commercially purchased. Pictures show the structural view of woven fabric and demonstrate that no finishing was noticeable on the substrates before the experimental part of electrospaying. Results are shown in Table 6.1.3.1.

Table 6.1.3.1 Untreated substrates under SEM analysis

| | Substrate no. 1 | Substrate no. 2 |
|--|---|--|
| Untreated substrates under magnification x100. |  |  |
| Untreated substrate materials under magnification of x500. |  |  |

The effect of electrospayed solution on pre-treated substrates is given in Table 6.1.3.2. SEM pictures show that electrospayed coating covers substrate materials' surface and overall gave a uniform coating. On fibres are visibly distinguishable larger and smaller particles whose size depends on the crosslinking time of the solution. From analysis could be concluded that particle size grew bigger with heat and end time under stirring.

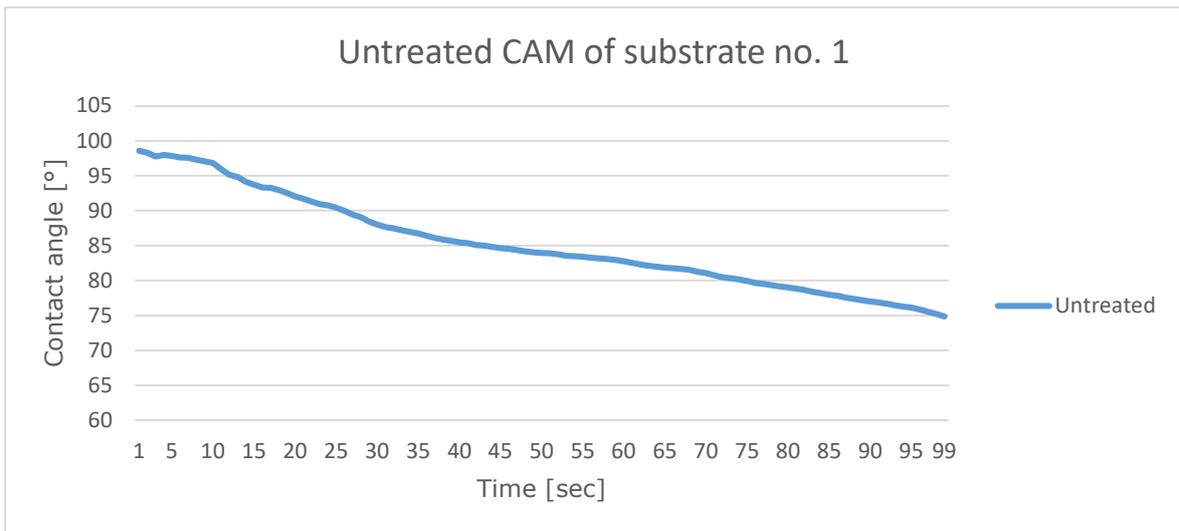
Table 6.1.3.2 Electrospayed substrates under SEM

| Solution no. 1 | Solution no. 3 | Solution no. 4 |
|---|--|---|
|  |  |  |

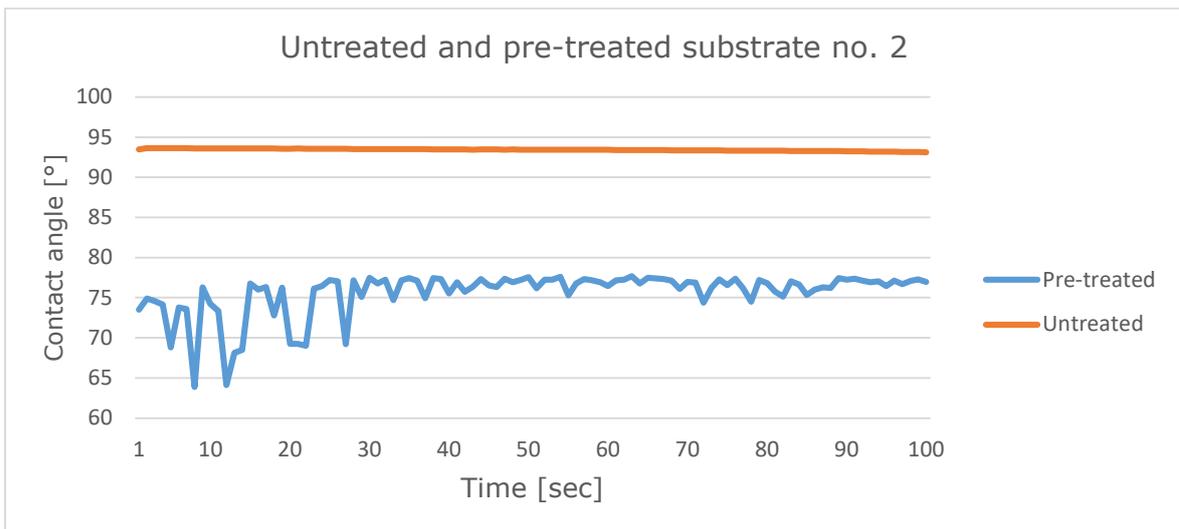
6.1.4 Contact angle measurements

Performed contact angle measurements for untreated textile substrates show the original state of examined fabrics with finishing properties previous to pre-treatment and electrospaying. Pre-treated substrate no.1 CAM could not be recorded because of

the substrate's super hydrophilic properties. For substrate no. 1 CAM result presented in Graph 6.1.4.1 shows that the original state of fabric measurement started at 98.6 ° expressing hydrophobic properties, similar to substrate no. 2, which was at starting point measuring 93.5 ° and also hydrophobic. Substrate no. 1 CAM kept falling through the test, finishing at 74.9 °. However, for substrate no. 2 test result stayed almost the same for further measuring time, showing signs of hydrophobic properties. Results for CAM performed for substrate no. 2 are shown in Graph 6.1.4.2. Both fabrics showed similar results in the original state, which is the untreated state.



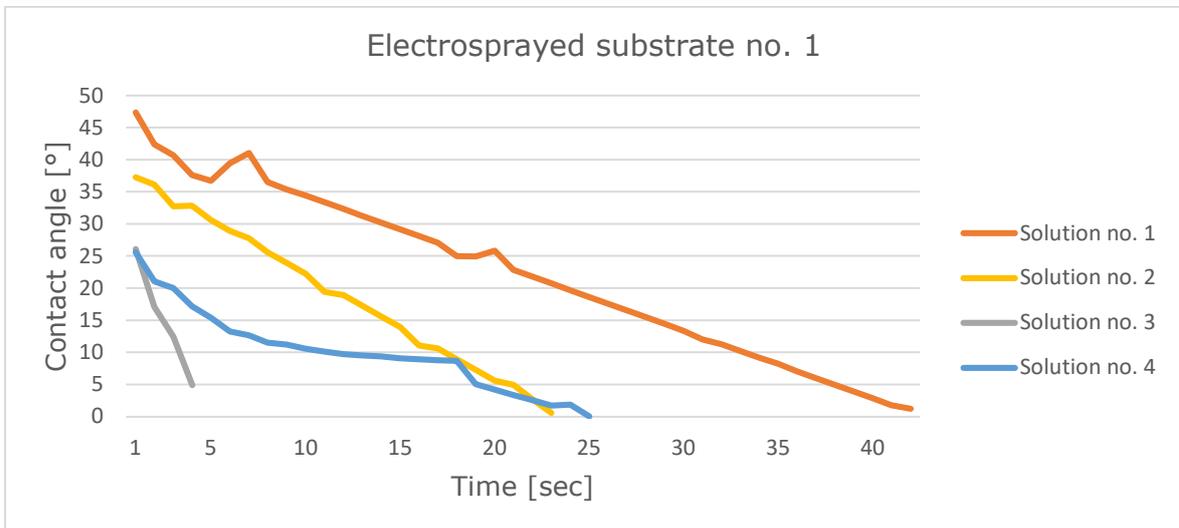
Graph 6.1.4.1 Results for CAM performed on untreated substrate no. 1



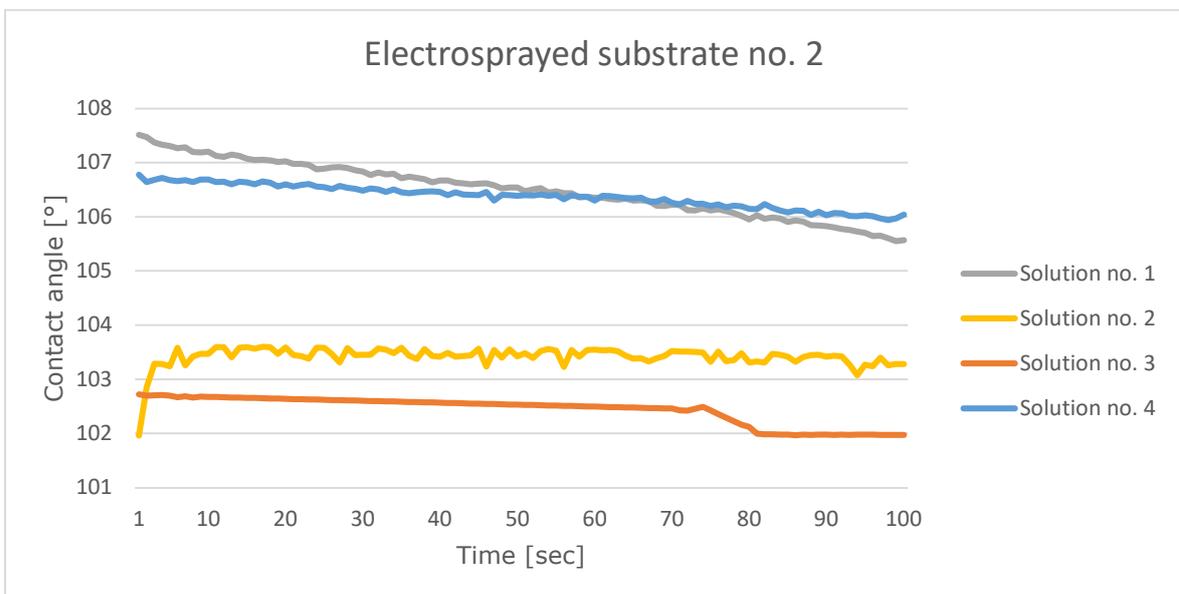
Graph 6.1.4.2 Results for CAM performed on untreated and pre-treated substrate no. 2

Electrospray substrate no. 1 CAM started at 47.4° expressing hydrophilic properties. Solution no. 2 showed lower results than the previous solution, which was 37.2° Result for a solution no. 3 and no. 4 are between 25.5° to 26° degreasing fast. The longest

measurement lasted for 44 seconds, and the shortest was 5 seconds. Throughout all electrosprayed cotton substrates, they express hydrophilic properties. CAM results for electrosprayed substrate no. 1 are shown in Graph 6.1.4.3.



Graph 6.1.4.3 Results for CAM performed to electrosprayed substrate no. 1

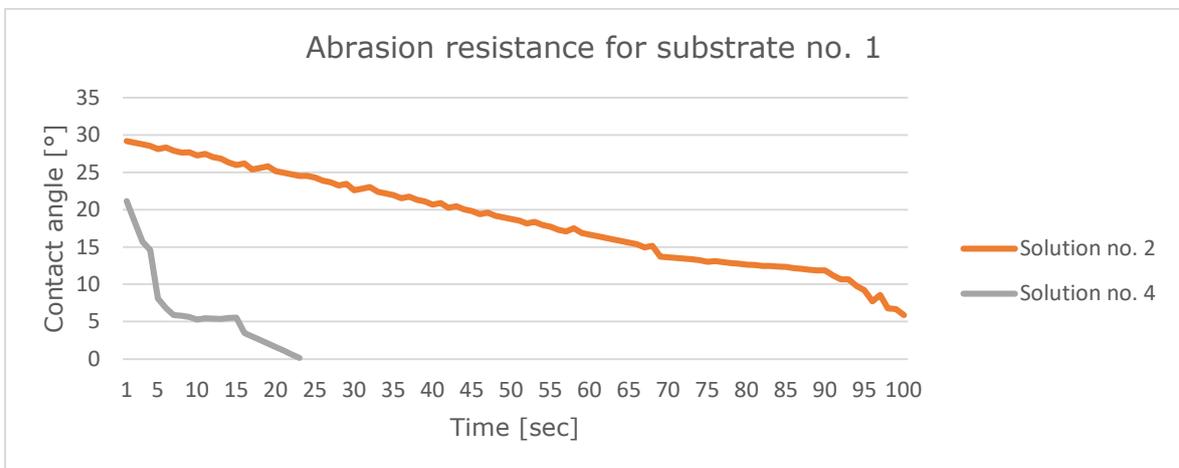


Graph 6.1.4.4 Results for CAM performed to electrosprayed substrate no. 2

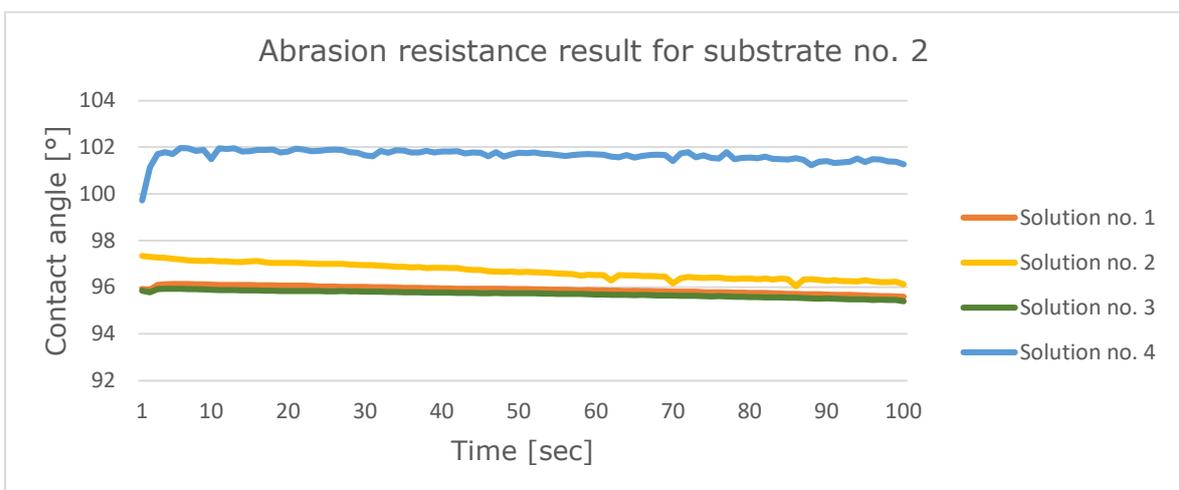
Electrosprayed substrate no. 2 overall expressed hydrophobic properties resulting in all solutions CAM resulting higher than 90°. For solution no. 1 CAM was measured at starting point of 107.5°, which was the highest of all solution results. Solution no. 2 CAM was 101.9°, and solution no. 3 was 102.7°, and for solution no. 4 it was 106.9°. Results for CAM performed for electrosprayed substrate no. 2 are expressed in Graph 6.1.4.4.

Abrasion resistance for substrate no. 1 CAM started at 29.2° expressing hydrophilic properties, and for solution no. 4, the result was even lower, measuring starting point at 21.2°. Electrospayed substrate no. 1 showed very hydrophilic features close to being super hydrophilic. For solutions no. 1 and no. 3, It was not possible to record CAM results because substrates appeared to be super hydrophilic. CAM results for electrospayed substrate no. 1 are shown in Graph 6.1.4.5.

For substrate no. 2 abrasion resistance results all expressed hydrophobic properties. Most promising results gave solution no. 4 which result were the highest of all four solutions. Results for abrasion resistance performed on substrate no. 2 are expressed in Graph 6.1.4.6.



Graph 6.1.4.5 Results for CAM performed to testes substrate no. 1 for determination of abrasion resistance



Graph 6.1.4.6 Results for CAM performed to testes substrate no. 2 for determination of abrasion resistance

6.1.5 Results of different tests performed on finished textiles

Determination of the abrasion resistance of fabrics by the Martindale method

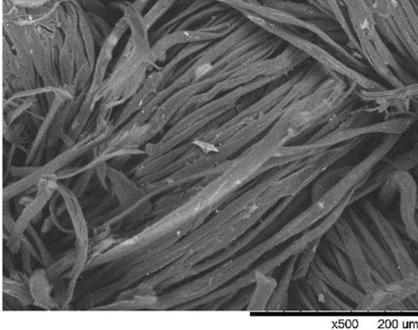
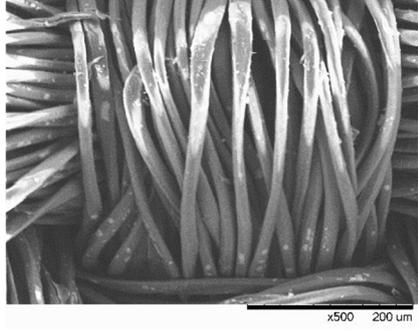
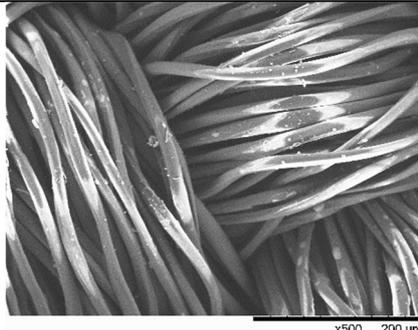
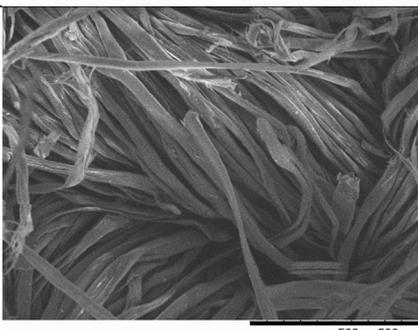
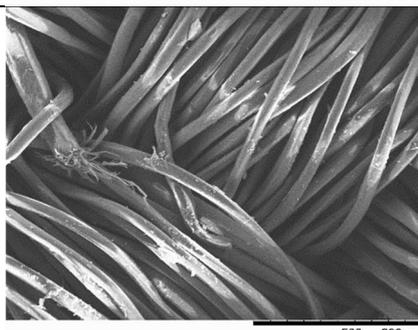
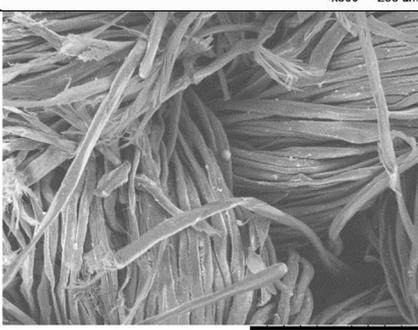
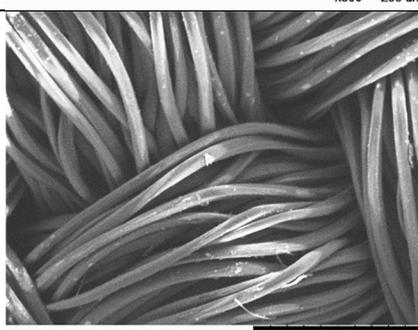
The effect of abrasion on the fabric substrates was analyzed under scanning electron microscopy to conclude an overview of the finishing success and the amount abraded off during testing. Table 6.1.5.1 presents SEM analysis results for test substrates to determine the abrasion resistance of electro sprayed finishing. Before the analysis, specimen surfaces were not coated with gold, which created a surplus charging effect on the specimen.

With solution no. 1, substrate no. 1 show small particles attached to the substrate surface that have not been rubbed off whilst testing. Finishing made with solution no. 1 is almost completely rubbed off to the point where only a few particles are visible on the surface of the fibres. It can be assumed that the abradant has rubbed together some amount of particles and formed lumps on the surface. While substrate no. 2 shows solution marks on the surface, that solution has been rubbed off and over the yarns. Particles have not been rubbed off between intersections of yarns, where small droplets are noticeable.

Solution no. 2 result shows a very thin layer like finishing and small particles on the substrate no. 1. Abradant has damaged the fibre surface, therefore removing the electro spray layer from the substrate surface. There seems to remain some amount of coating still attached to fibre surface. For substrate no. 2, the coating was better attached to fibre surface. However, the abradant still removed a significant amount of finishing from the surface while leaving small particles and spots on the substrate surface.

After the abrasion resistance test, solution no. 3 and solution no. 4 left similar results on the substrate surface. No significant difference between the observed test specimen was detected. Some of the particles were removed in abrasion testing, but distinct particles were visible on the fibres. With substrate no. 1 fibres are noticeably broken whilst electro sprayd particles remain on the severed fibres. For substrate no. 2, the coating is quite well attached to the fibre surface, and very little has worn off in rubbing.

Table 6.1.5.1 SEM analysis results for determination of abrasion resistance

| Solution | Substrate no. 1 | Substrate no. 2 |
|----------------|---|--|
| Solution no. 1 |  |  |
| Solution no. 2 |  |  |
| Solution no. 3 |  |  |
| Solution no. 4 |  |  |

Determination of resistance to surface wetting

Determination of surface wetting shows the result of increase in water-repellency in electrospaying coating to substrates. Results for the determination of resistance to surface wetting are given in Table 6.1.5.2.

When untreated substrate no. 1 was completely wetted, then solution no. 2 already showed better results as the specimen was wetted beyond spray points. Solution no. 4 gave the best result to spray testing for substrate no. 1, which partially wet the specimen surface. During testing, electro sprayed solution no. 4 showed the most promising results for water-repellency.

For substrate no. 2, results stayed overall the same for different solutions. The lowest result was with solution no. 1, and the highest result was solution no. 4. Electro sprayed solution did not improve untreated substrate properties.

Table 6.1.5.2 Results for determination of resistance to surface wetting

| Solution | Material | Result [ISO] | Comment |
|----------------|-----------------|--------------|---|
| Untreated | Substrate no. 1 | 0 | Complete wetting of the surface of the specimen. |
| | Substrate no. 2 | 3 | Wetting of specimen face at spray points. |
| Pre-treated | Substrate no. 1 | 0 | Complete wetting of the entire face of the specimen. |
| | Substrate no. 2 | 1 | Complete wetting of the entire specimen face beyond the spray points. |
| Solution no. 1 | Substrate no. 1 | 0 | Complete wetting of the surface of the specimen. |
| | Substrate no. 2 | 2 | Partial wetting of the specimen face beyond the spray points. |
| Solution no. 2 | Substrate no. 1 | 1 | Complete wetting of the entire specimen face beyond the spray points. |
| | Substrate no. 2 | 2 | Partial wetting of the specimen face beyond the spray points. |
| Solution no. 3 | Substrate no. 1 | 1 | Complete wetting of the entire specimen face beyond the spray points. |
| | Substrate no. 2 | 3 | Wetting of specimen face at spray points. |
| Solution no. 4 | Substrate no. 1 | 2 | Partial wetting of the specimen face beyond the spray points. |
| | Substrate no. 2 | 3 | Wetting of specimen face at spray points. |

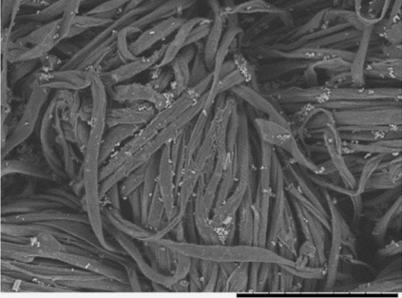
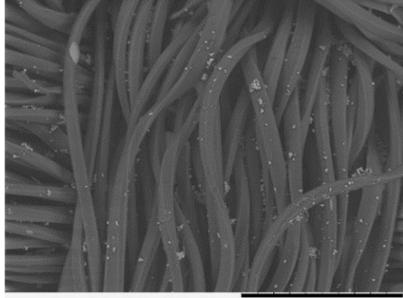
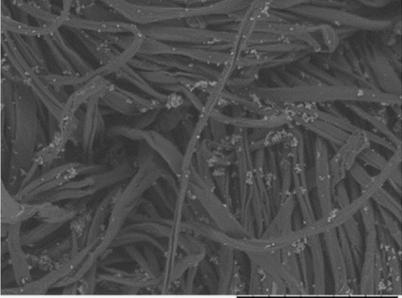
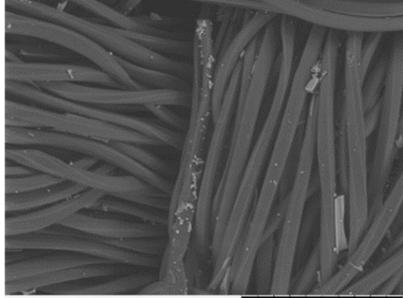
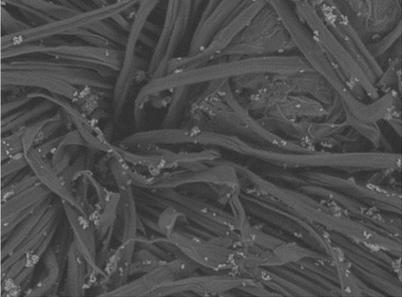
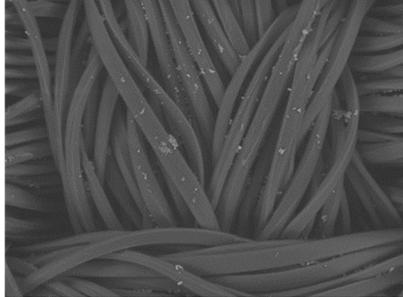
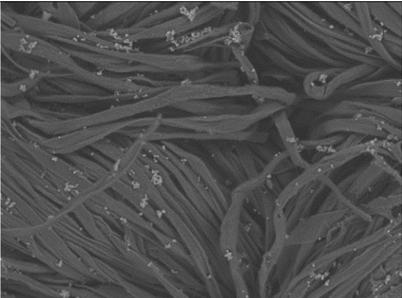
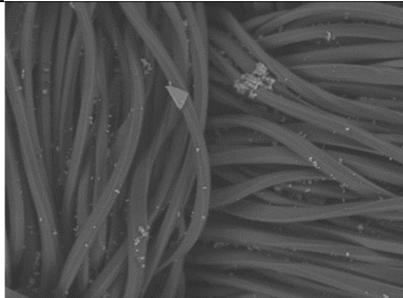
Domestic washing procedures for textile testing

Domestic washing procedures for textile testing results are given in Table 6.1.5.3. In overall, all coatings performed well under washing procedures. From the pictures it could be concluded that the coating is well attached to the substrate's surface and strong enough to stay on the fibres during washing.

For solution no 1, on substrate no. 1 particles have been washed off during domestic washing procedures, and particles have interconnected, thus creating clusters of particles hooked together. For substrate no. 2, washing procedures seem to have peeled off the finishing pieces, meaning that particles were not so firmly attached to the fibre surface. Similarly, to substrate no. 1 also, particles have been stacked together.

With washing procedures performed for solution 2, substrate no. 1 presents bigger clumps of particles stacked together than to solution no. 1.

Table 6.1.5.3 Results for domestic washing procedures for textiles testing

| Solution | Substrate no. 1 | Substrate no. 2 |
|----------------|---|--|
| Solution no. 1 |  |  |
| Solution no. 2 |  |  |
| Solution no. 3 |  |  |
| Solution no. 4 |  |  |

6.2 Concusions of the experimental part

Peformed study was conducted to perform electro spraying to add water-repellent properties to the substrates. During electro spraying each fabric substrate was sprayed with 2 ml of solution. Overall results obtained during SEM and CAM show that sprayed coating is firmly attached to the surface of the substrates and particles appear to be somewhat uniform for each solution. However when crosslinking stage reaches to high, coating appeared to be cracking from places where it has formed layers onto substrate surface. Throughout testing particles still remained on the surface of the specimen showing that with changing the parameters of the solution or spraying setup, better results could be obtained.

Main difference between two substrates stands with their naturally obtained properties from fibre characteristics, cotton in its nature is more prone to take in water while polyester has natural hydrophobic properties. With electro sprayed coating these fabrics showd growth in water-repellent properties and with changing the process parameters more satisfactory results can be acquired for these substrates.

6.3 Recommendations for improvements

Performed study on electro spraying method shows promising results. Although given study on electro spraying did not result in as significant changes as hoped, characteristics of the solution and experimental setup could be altered to achieve better results.

With increasing solution concentration, with electro spraying process it is possible to produce more uniform and homogenous particles. Changes in particle size could lead to better water-repellent properties. However, too big particles will create a thick layer on fabric that could be broken off from the textile surface by bending the fabric by motion. As concluded from the study, sprayed particles would grow more extensive for a more crosslinked solution. Particle size could also be changed by creating a bigger distance between the nozzle and collector plate, so particles have a time to split into smaller particles.

By increasing applied voltage, particles could be pulled more between the fibres than just positioning to the surface. However, this was not the point of the study to alter fiber characteristics, but this could also alter the water-repellent properties of the textiles.

With increased voltage more uniform flow of the particles to the substrate creates a more evenly sprayed layer onto the textile surface.

SUMMARY

The term "finishing," in its broadest meaning, covers all processes that fabric undergoes after production. Even if materials inherit naturally good properties, some finishing is always necessary to lengthen the life of textile products. Therefore, it is necessary to modify the textile materials to provide the required performance for the product they have been designed to become. The textile sector shows promising results for developing and applying nanotechnology and textile-related nanotechnology studies, including modifying textile materials and upgrading chemical finishes to improve dyeability, water- and oil-repellency, antibacterial and other properties via nanoparticles. [1, 2, 9, 3]

One possible approach for this is electrospaying, in which the nanoparticles layer is deposited onto the fabric surface. The electrospaying setup consists of charged syringe fed with polymer solution onto a grounded collector plate with the substrate material. [3] The study was carried out using the electrospaying method. For the process was made four solutions with different crosslinking stages consisting of TEOS, H₂O, EtOH, and HCl. The first solution underwent 10 minutes of heating and stirring second 20 minutes, the third 30, and the fourth 40 minutes. All tested substrate materials, 100% cotton and 100% polyester fabrics underwent pre-treatment prior to being electrospayed with mentioned solutions.

After spaying, the uniformity of the finishing was analyzed under SEM. It could be concluded that the solution prepared for the process was successful, and particles were visible under magnification. The first solution has small particles attached to the fibre surface with almost evenly spread over the substrate. Particle size grew more prominent throughout the solutions, and for 40 minute solution there were distinguishable layers on the substrate surface. There also appeared to be some cracks in the finishing where too much solution was sprayed onto one place.

CAM measurements showed that untreated substrates showed hydrophobic properties, with the result being slightly over 90°. Electrospayed solutions showed the most promising results for cotton substrate for 10 minute solution and 20 minute solution. With polyester substrate most effective result appeared to be with 10 minute solution and 40 minute solution. Abrasion resistance measurements performed for cotton substrate showed very to super hydrophilic properties without achieving the results hoped for to have water-repellent properties. For polyester substrate, the substrate was

expressing hydrophobic characteristics with all solutions CAM maintaining throughout the measuring over 90°.

Laundry test substrates were analyzed under SEM, which showed that solution particles were clumped together on the surface, creating clusters of finishing for both substrates. Performed study of "environmentally friendly fabric finishing by eletrospraying method" showed promising results for future perspective. The expected results set up before the study stated that electrospaying would result in water-repellent properties; however, the results of the treated samples were not as succesful as hoped. Therefore some changes in the process are needed to achieve the expected results. For example, changing the solution concentration, size of the particles could be altered to be more uniform. With increasing distance between the nozzle tip and collector plate, particles would have more time to evaporate into smaller particles. With increased voltage, particle attachment to the surface would be more substantial.

KOKKUVÕTE

Mõiste "viimistlus" hõlmab selle kõige laiemas tähenduses kõiki protsesse, mille kanvas läbib pärast tootmist. Isegi kui materjalid on soovitud omadustega, on tekstiiltoodete eluea pikendamiseks alati vajalik järelviimistlemine. Seetõttu on vaja tekstiilmaterjale järeltöödelda, tagamaks soovitud tulemused kavandatud toote eesmärgipäraseks kasutamiseks. Tekstiilisektor näitab paljulubavaid tulemusi nanotehnoloogia valdkonnas ja tekstiilidega seotud nanotehnoloogia uuringute väljatöötamisel ning rakendamisel, sealhulgas tekstiilmaterjalide muutmisel ja keemilise viimistluse täiustamisel, parandamaks nanoosakeste abil värvipüsivust, vee ja õli hülgavust, antibakteriaalseid ja muid omadusi. [1, 2, 9, 3]

Üks võimalik lähenemisviis selleks on elektropihustus meetod, mille käigus kanga pinnale kantakse nanoosakeste kiht. Elektropihustusseade koosneb vooluringi ühendatud süstlast, mille abil juhitakse polümeerilahus maandatud kollektorplaadile, millele on kinnitatud kangas. [3] Käesolevas magistritöös kasutati viimistlemisel elektropihustusmeetodit. Protsessi läbiviimiseks valmistati neljas erinevas ristsidumisetapis olevat lahust järgmiste koostisosadega: TEOS, H₂O, EtOH ja HCl. Esimest lahust kuumutati ja segati 10 minutit, teist 20 minutit, kolmandat 30 minutit ja neljandat 40 minutit. Kõik testitud materjalid, 100% puuvill ja 100% polüester kangad läbisid eelnevalt eeltötluse.

Pärast pihustamist analüüsiti viimistluse ühtlust SEM-i abil. SEM analüüsi põhjal järeldati, et valmistatud lahused sobisid viimistlemiseks, kuna SEM analüüsi abil sai tuvastada erinevaid viimistluse osakesi kanga pinnal. Esimese lahuse puhul kinnitusid kiu pinnale väikesed osakesed, mis olid peaaegu ühtlaselt üle aluspinna jaotunud. Osakeste suurus kasvas iga lahusega ja 40 minutilise lahuse puhul olid kanga pinnal eristatavad viimistluskihid. Viimistluses ilmsid ka mõrad, mis oli tingitud sellest, et lahust oli liigselt pritsitud ühele kohale.

Kontaktnurga mõõtmisel selgus, et töötlemata kangastel olid hüdrofoobsed omadused, mille mõõtetulemuseks oli veidi üle 90°. Elektropihustuse lahustega esinesid kõige lootustandvamad tulemused puuvillase kanga puhul 10 minuti lahusega ja 20 minutilise lahusega. Kulumiskindluse mõõtmisel puuvillasel kangal töötlus tulemusi ei andnud ning kangad olid väga hüdrofiilsed. Polüesterkanga puhul näitasid kõige paremaid tulemusi 10 minutilise lahuse ja 40 minutilise lahuse. Elektropihustusega töödeldud polüesterkangad tõestasid hüdrofoobseid omadusi kõikidel mõõtmistel ja testimistel, kus kõik kontaktnurga mõõtmised püsisid üle 90°.

Peale pesutestianalüüsi kangaid samuti SEM-iga, tulemustest võis järeldada, et pesemise käigus olid viimistluse osakesed kanga pinnal kokku haakunud, moodustades nii puuvillase kui ka polüesterkanga puhul kangapinnale osakeste klompe. Siiski võib järeldada, et läbiviidud uuring „Keskkonnasõbralik kangatöötlus elektropihustus meetodil“ näitas tulevikuperspektiive silmas pidades paljutöotavaid tulemusi. Enne uuringut püstitatud eeldatavad tulemused väitsid, et elektripihustuse tulemuseks on vett hülgavate omadustega kangatöötlus; viimistletud katsekehade analüüsimise ja testimise tulemused olid oodatust halvemad. Seetõttu on vajalik lahuste kontsentratsiooni muutmine või süstla tipu ja kollektorplaadi vahelise kauguse muutmine, mis tagaks ühtlasema osakeste laotumise katsekehade pinnale või suurendaks osakeste kinnitumise tugevust kanga pinnale.

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APPENDICES

Appendix 1 Determination of mass per unit area using small samples

Appendix 1.1. The result for determination of mass per unit area using small samples for substrate no. 1

| Sample nr. | Formula | m [g] | A [m ²] | Result M [g/m ²] |
|---------------------|--------------------------|--------|---------------------|------------------------------|
| 1 | $M = \frac{m * 1000}{A}$ | 1.9531 | 100.0 | 195.31 |
| 2 | | 1.9513 | 100.0 | 195.13 |
| 3 | | 1.8956 | 100.0 | 189.56 |
| 4 | | 1.8722 | 100.0 | 187.22 |
| 5 | | 1.8791 | 100.0 | 187.91 |
| TOTAL RESULT | | | | 191 |
| Standard deviation | | | | 4 |

Appendix 1.2. The result for determination of mass per unit area using small samples for substrate no. 2

| Sample nr. | Formula | m [g] | A [m ²] | Result M [g/m ²] |
|---------------------|--------------------------|--------|---------------------|------------------------------|
| 1 | $M = \frac{m * 1000}{A}$ | 1.8769 | 100.0 | 187.69 |
| 2 | | 1.9021 | 100.0 | 190.21 |
| 3 | | 1.8850 | 100.0 | 188.50 |
| 4 | | 1.8633 | 100.0 | 186.33 |
| 5 | | 1.8785 | 100.0 | 187.85 |
| TOTAL RESULT | | | | 188 |
| Standard deviation | | | | 1 |

Appendix 2 Determination of number of threads per unit length

Appendix 2.1. The result for determination of number of threads per unit length of Substrate no. 1

| Nr. | Weft direction | Warp direction |
|--------------------|----------------|----------------|
| 1 | 76 | 72 |
| 2 | 75 | 74 |
| 3 | 76 | 74 |
| 4 | 74 | 76 |
| 5 | 76 | 75 |
| Result | 75 | 74 |
| Standard deviation | 1 | 2 |

Appendix 2.2. The result for determination of number of threads per unit length of substrate no. 2

| Nr. | Weft direction | Warp direction |
|--------------------|----------------|----------------|
| 1 | 66 | 71 |
| 2 | 64 | 71 |
| 3 | 71 | 72 |
| 4 | 72 | 67 |
| 5 | 68 | 69 |
| Result | 68 | 70 |
| Standard deviation | 3 | 2 |