TECHNICAL UNIVERSITY OF LIBEREC
Faculty of Textile Engineering

Antimicrobial treatments for textile materials

Master Thesis

Study programme: N0723A270002 Textile Engineering
Study branch:

Author: **Akshat Tegginamath**
Thesis Supervisors: Ing. Ondřej Novák, Ph.D.
Department of Nonwovens and Nanofibrous materials

Liberec 2021

TECHNICAL UNIVERSITY OF LIBEREC
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Master Thesis Assignment Form

Antimicrobial treatments for textile materials

Name and surname: **Akshat Tegginamath**
Identification number: T19000392
Study programme: N0723A270002 Textile Engineering
Study branch:
Assigning department: Department of Nonwovens and Nanofibrous materials
Academic year: **2020/2021**

Rules for Elaboration:

1. Prepare literature review focused on "Antimicrobial treatment for textile materials".
2. According to the previous research work select appropriate methods for antimicrobial/antibacterial treatments for textile materials made of the polypropylene.
3. Prepare samples and test selected properties.
4. Discuss obtained results and provide recommendations.

Scope of Graphic Work:
Scope of Report: 40-60
Thesis Form: printed/electronic
Thesis Language: English

List of Specialised Literature:

1. HEGEMANN, DIRK, 2021. Plasma polymerization and its applications in textiles. *Nopr.niscair.res.in* [online]. Retrieved z: http://nopr.niscair.res.in/handle/123456789/24497.
2. HASSABO, AHMED and ESRAA EL-SAYED, 2021. Recent advances in the application of plasma in textile finishing (A Review). *Journal of Textiles, Coloration and Polymer Science* [online]. vol. 0, no. 0, pp. 0-0. Retrieved z: doi:10.21608/jtcps.2021.67798.1050
3. CHEN, CHONYU and WEN-YA CHANG, 2021. Antimicrobial activity of cotton fabric pretreated by microwave plasma and dyed with onion skin and onion pulp extractions. *Nopr.niscair.res.in* [online] .. Retrieved z: http://nopr.niscair.res.in/handle/123456789/304
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Date of Thesis Assignment: November 1, 2020
Date of Thesis Submission: August 30, 2021

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Liberec November 1, 2020

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Acknowledgements

I express my sincere thanks to the supervisor Prof. Ondra Novak for his inspiring guidance, constructive criticism and valuable suggestions provided during the course of this research. I also express my gratitude to my colleagues and my parents for their constant support. I would also like to thank the staff of the faculty for helping me in conducting my experiments during this period of the epidemic.

Akshat Tegginamath

Abstract

One of the major causes for chronic infections can be linked to bacterial infections which fester at a very high rate, thus the need for the usage of antibacterial materials is of paramount importance. The development of antimicrobial textiles in this day and age has seen an exponential growth, which has also been influenced by the ongoing pandemic which has disrupted all forms of normalcy around the world. Antimicrobial textiles are not only being used for medical purposes but also for personal protection against the variety of microbes which are present in the environment. With textiles being utilised widely in everyday activities, the textile material can be used to effectively counteract microbes and bacteria. In the recent years, nanotechnology has advanced at a blistering pace and with the advent of electrospinning and the production of nanofibers in a large scale, nanofibers are also being utilised to produce antimicrobial/antibacterial textiles, with a large surface area, antibacterial nanofibers allow for the efficient integration of antibacterial agents. The various techniques used to produce antibacterial/antimicrobial finishes are discussed here along with the various viruses present in the atmosphere along and potential agents to be used against these microbes. The method of metallisation via galvanisation of textile materials and the usage of textiles produced via the hybrid process known as spun blown process as antimicrobial/antibacterial textiles is studied here. In the study conducted it was seen that process of galvanisation wasn't adept in producing a suitable antibacterial/antimicrobial textile material. According to previous research work conducted on silver based antibacterial/antimicrobial textile materials was supposed to have produced suitable results, the spunblown material could have shown better results in this regard. It can be recommended that the spunblown materials can be tested further since the properties of fibers obtained via the process of the spun blown process are comparatively better than using the conventional SMS materials.

Key words

Antimicrobial, antibacterial, electrospinning, sputtering, galvanisation, spun blown

Abstrakt

Jednou z hlavních příčin chronických infekcí mohou být bakteriální infekce, které hnisjí velmi rychle, takže potřeba použití antibakteriálních materiálů má prvořadý význam. Vývoj antimikrobiálních textilií v této době zaznamenal exponenciální růst, který byl také ovlivněn pokračující pandemií, která narušila všechny formy normálnosti po celém světě. Antimikrobiální textilie se používají nejen pro lékařské účely, ale také pro osobní ochranu před různými mikroby, které jsou přítomny v životním prostředí. Vzhledem k tomu, že textilie jsou široce využívány při každodenních činnostech, lze textilní materiál použít k účinnému potlačení mikrobů a bakterií. V posledních letech nanotechnologie pokročily v prudkém tempu a s příchodem elektrospiningu a výrobou nanovláken ve velkém měřítku se nanovlákna používají také k výrobě antimikrobiálních/antibakteriálních textilií s velkou plochou, antibakteriální nanovlákna umožňují účinnou integraci antibakteriálních látek. Jsou zde diskutovány různé techniky používané k výrobě antibakteriálních/antimikrobiálních povrchových úprav spolu s různými viry přítomnými v atmosféře a potenciálními činidly, která mají být použita proti těmto mikrobům. Je zde studován způsob metalizace galvanizací textilních materiálů a použití textilií vyrobených hybridním procesem známým jako spun-blown pro výrobu antimikrobiálních/antibakteriálních textilií. Ve studii bylo vidět, že proces galvanizace není vhodným adeptem pro výrobu antibakteriálních/antimikrobiálních textilií. Na základě předchozích výzkumů na antibakteriálních/antimikrobiálních textiliích by měly být vhodné materiály na bázi stříbra, spun-blown materiál mohl v tomto ohledu dosáhnout lepších výsledků. Lze doporučit, aby spun-blown materiály byly dále testovány, protože vlastnosti vláken získaných procesem spun-blown jsou poměrně lepší než použití konvenčních SMS materiálů.

Klíčová slova

Antimikrobiální, antibakteriální, elektrospining, rozstřikování, galvanizace, spun-blown

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1.0 Introduction:

The growth of antimicrobial textiles in this day and age has seen an exponential growth, which has also been influenced by the ongoing pandemic which has disrupted all forms of normalcy around the world. Antimicrobial textiles are not only being used for medical purposes but also for personal protection against the variety of microbes which are prevalent in the environment. With textiles being utilised widely in everyday activities, the textile material can be used to effectively counteract and safeguard against microbes and bacteria [1]. When microbes/microorganisms are present on textiles and start growing a variety of undesirable qualities are introduced like unpleasant odour, discolouration of fabrics and stains, which are unwanted for the wearer as well as the fabric [73]. To produce an antibacterial or antimicrobial textile, various wet treatment processes are utilised, but these processes produce a large amount of waste products leading to these processes being non-environmentally friendly processes. With the upcoming rise in requirements for antimicrobial/antibacterial textiles new process of producing these products are to be utilised which are comparatively environmentally friendly in nature [1]. The antibacterial/ antimicrobial textiles can also be used in the fields of wound care/ wound dressing since, one of the major causes for chronic infections can be linked to bacterial infections [2-5], which fester at a very high rate on existing wounds, thus the need for the usage of antibacterial materials is of paramount importance. With the rise in nanofibers and nontechnology, nanofibers can also be utilised in the production of antimicrobial/antibacterial products, with a large surface area, antibacterial nanofibers allow for the efficient integration of antibacterial agents [6]. The areas of research under nanotechnology are also expanding at an exponential rate. One of the areas of research under this revolution is nanomedicine and over the recent decades, this field has shown a great potential of becoming a major field of research. Research in this field has led to drastic improvement of human health [7]. A number of techniques have been utilized for the production of nanofibers such as melt spinning, chemical vapour deposition, sinter technology, solution spinning and electrospinning. Among these techniques it is found to be that the electrospinning technique is the most cost-effective method in producing continuous nanofibers from numerous polymers or compounds [8-11]. The nanofibers produced via electrospinning have a large specific area, a high porosity and also have a wide range of applications in the fields of tissue engineering, regenerative medicine and wound dressing. The wound healing process is continually put on the line and tested with

the presence of bacteria, which when present lead to the inflammation of the wound and delay the process of healing. Bioactive wound dressings which is a new field of wound dressing, has shown a great potential in displacing the conventional methods of wound dressing. Since these bioactive wound dressings are made of nanofibers from natural polymers containing antibacterial agents and the field of skin wound dressing is projected to have a $20.4 billion global market by the year 2021 [12]. A study conducted has shown that the production of antimicrobial textiles has increased more than 15 % in a year in Western Europe between 2001 and 2005 [159], and the production of socks, shoe lining and sports wear made up 85 % of the whole production [73].

One of the most commonly and widely used synthetic fibers in the industry is the polypropylene (PP) fibers. Other than being cheap and stronger than the other synthetic fibers, PP also has numerous applications in the fields of carpet manufacture, cover stock, cables, napkins, auto motive interior trims, packaging, films and many more [13]. PP can also be used to produce sanitary products, which need to inherently possess the antibacterial/antimicrobial property, like hygienic bands, diapers, surgical masks, filters, etc [14]. The usage of silver in the role of an antibacterial treatment material has come forth over the recent years owing to the ever-increasing demand for protection arising from rising environmental pollution and the spread of diseases. Medically it has been seen that silver can kill more than 650 various disease-causing organisms, and silver is one of the safest organic compounds which can be used in antibacterial treatment without causing any bodily harm to the to the human body, when compared to the other organic compounds which are present and can be potentially used in this role [13]. The antibacterial effect of the silver used can be increased, as the potency of the antibacterial material used depends on the surface area available, and smaller the silver particles used the greater is the surface area [15,16]. Polymer composites with nanoparticles are proving to have great potential in the field of medicines, this is because it improves the properties of the materials used along with providing an effective method targeting for their applications [17,18]. The antibacterial activity shown by Silver (Ag) and Copper (Cu) is very high [19-22]. To enhance the antibacterial property of the polymers and the surface various techniques such as sputtering, spraying, layer by layer deposition and plasma deposition can be made use of [23-26].

1.1 Antimicrobial finishing requirements:

To maximise the benefits of a textile finished with antibacterial treatments, the antibacterial treatment should full fill a number of prominent requirements. The following are the requirements:

- The treatment should be viable against a spectrum of bacteria and fungal species and must be non-toxic to the wearer.
- The finishing applied must be durable, ie it must have the ability to sustain repeated laundry, dry cleaning and hot pressing process.
- The physical characteristics of the fabric onto which the finishing has been given must not be adversely affected.
- The finishing process should be compatible with the other textile chemical process, should be cost effective and also not produce any hazardous waste products which could affect the environment adversely [73].
- The antibacterial treatment given to the fabrics must also not get rid of the non-pathogenic bacteria present on the skin of the wearer, as these bacteria are important in maintaining the health of the skin of the individual and also provide an environment where pathogenic bacteria cannot grow [160].

1.3 Action of Antimicrobial Treatments:

The antimicrobial treatments like triclosan, silver, quaternary ammonium compounds and polyhexamethylene biguanide (PHMB) are known as biocides, they act against the bacteria by the permeability of the cell wall of the bacteria or destroying the cell wall. The cell wall protects the cell from the external environment as well as the maintaining the structural integrity of the cell. Furthermore, the antibacterial agents prevent the production of lipids, stop the activity of the enzymes and denature the proteins. These process which the antibacterial treatments prevent or destroy are inherently important for the functioning of the bacteria [73].

The desired antibacterial property can be imparted to the textile material by utilising various techniques.

1.4 Usage of Antimicrobial agents:

Depending on the fiber type used and the type of active agent used, there are numerous methods of application of antimicrobial agents onto the fibers/textiles. In case of synthetic fibers, the antimicrobial agents can be introduced at the extrusion stage or blended at the fiber forming stages, to incorporate the antimicrobial property. Since the active agents are physically embedded into the fiber structure in this process, this provides highly durable antimicrobial fibers, which can release the antimicrobial agents slowly over the period of usage of the textile material. Trevira and Novaceta employ this method to produce silver-containing Bioactive polyester and triclosan containing Silfresh cellulose acetate fibers. The pad-dry-cure process which is a conventional process to apply the antimicrobial finishing is also utilised for natural as well as synthetic fibers, to apply biocides such as triclosan and PHMB [73]. Silicone based quaternary agent AEM 5700 can be applied using padding, spraying or foam finishing [161]. Numerous other methods such as nanoscale shell-core particles [162,163], nano sized colloidal solutions [70], polymerisation grafting [164], cross linking of active agents onto a fiber [165,166] and chemically modifying the biocide in order to form a covalent bond with the fiber [167,168]. With the rise in sol-gel technology, sol-gel nanoparticles have been used to obtain textiles with a wide variety of functionalities including antimicrobial functionalities [169,170].

1.5 Testing of Antimicrobial agents:

Various testing methods for testing the efficacy of the antimicrobial agent used have been developed, these methods are generally divided into two types: suspension test and agar test [171,172]. Staphylococcus aureus (Gram positive) and Klebsiella pneumoniae (Gram negative) are utilised in the test methods, these bacteria are pathogenic in nature and must be handled with the utmost care and require appropriate containment facilities [73].

1.5.1 Suspension test:

The AATCC 100-2004, JIS L 1902-200 and SN 195924-1992 define the method of suspension testing. These tests provide a quantitative analysis on the antimicrobial finishing used. These when compared to Agar diffusion tests are exhaustive and time consuming. In these tests, the fabric of suitable size is made to absorb typically 1ml of bacterial inoculum in a growth medium. Which makes certain that there is a constant contact between the bacteria and the fabric sample. The inoculated fabrics are incubated in sealed jars at a temperature of 37°C or 27°C for 24 hours, after which the bacteria are removed from the fabric and via serial dilution and plating on agar nutrient plates, the total amount of bacteria is determined. The activity of

the antimicrobial agent is determined by calculating the difference in population of the bacteria before and after the incubation stage. To make certain that the numbers obtained are strictly due to the antimicrobial finishing numerous samples are to be utilised which have gone through the same procedure except antimicrobial finishing. The suspension tests are usually conducted under artificial conditions and this done to increase the growth of bacteria [73]. The JIS L 1902-2002 method of testing is suggested to recreate realistic scenarios. According to ISO 20743 bacteria are printed on to the textile substrate and incubated at 20°C for 18 to 24hours in humid conditions after which the cells which have survived are determined [173]. The antimicrobial tests conducted only give a measure of effectiveness of textiles that have been treated.

1.5.2 Agar Diffusion Test:

AATCC 147-2004, JIS L 1902-2002 and SN 195920-1992, give the method of agar diffusion test to be conducted and theses tests are purely qualitative in nature, simple to conduct and are apt for testing large samples for antimicrobial activity. Here the bacterial cells are inoculated on agar nutrient plates, above which textile fabrics are placed to ensure a constant contact between the bacteria and the textile fabric. The plates are then incubated at 37°C for a period of 18 to 24hours. The bacterial growth is examined under the fabric and at the edges of the fabric sample. If there isn't any bacteria present under the examined fabric sample, it indicates that there was antibacterial activity which took place [73].

1.6 Antimicrobial Agents used in Textiles:

The antimicrobial agents used in textiles are not exactly new in the industry as these antimicrobial agents have been utilised in various other industries like the food preservative industry, disinfectants, wound dressings or in swimming pool santitisers. The Minimal Inhibitory Concentration (MIC) values gives the potency of the antimicrobial agents used and the agents used in these industries have a very high potency, but these agents when used on textiles or when embedded into textile fibers are limited in their activity as their availability is limited. Also, since textiles are washed, these agents face the danger of being washed off during the rinsing process. Thus, a large amount of these agents must be utilised or applied on textiles to effectively perform its function as an antimicrobial agent. The following are the various antimicrobial agents that can be utilised on textiles:

- Quaternary Ammonium Compounds
- Triclosan
- Metals and Metal Salts

- Polyhexamethylene biguanide (PHMD)
- Dyes
- Chitosan
- Regenerable N-halamine and Peroxide[73]

1.6.1 Quaternary Ammonium Compounds (QACs):

To produce disinfectants from QACs, generally chains which are made of 12-18 carbon atoms are utilised. The positive charge of the N atom of the compounds cases the cell membrane of the microbes to get damaged or they help in the disruption of the structure of the cell or they help in the denaturation of the proteins present in the microbes [182]. The ammonium group stays active as long as the group is bonded on to the textile substrate [183]. The QACs attach to the textile substrate due to the interactions between the textile substrate and the QACs which are anionic and cationic in nature respectively [184,74]. Fibers containing carboxylic or sulfonate groups such as Acrilan and Orlon can be exhausted with QACs at the boiling points of the compounds [185-187]. Woollen fabrics can be made antimicrobial in nature for at least 10 laundry cycles, by treating it QACs [188,75]. Cotton fabrics can be made antimicrobial in nature by treating them with 4-aminobenze-nesulfonic acid-chloro-triazine, which helps the QACs to be exhausted on to the fabric surface with ease [189]. For fibers like Nylon66, QACs can be applied under alkaline conditions, once these fibers are dye with acid dyes. This is due to the fact that these fibers do not contain many reactive cites and are resistant to chemical modification processes. This procedure produced an antibacterial/antimicrobial coating which was semi-durable finishing as shown by Sun et al [183]. BIOGAURD by AEGIS Environments is an active QAC which is commercially available, and has an MIC of 10-100mg/l acting against gram-negative and gram-positive bacteria [190]. This can be applied onto textile materials via padding, spraying and foam finishing, upon drying the silane which is non-volatile in nature forms a covalent bond with the textile material to result in a finishing with a high degree of reliability.

1.6.2 Triclosan:

Since the early 1960s, this has been extensively been used in many consumer products such as hand soaps, deodorants, toothpastes, shower gels, mouthwashes and surgical scrubs. Triclosan works by inhibiting the synthesis of lipids thus leading to the inhibition of the growth of the bacteria, and has a MIC of less than 10ppm against a variety of bacteria [191,192,193]. A method of treating cotton and cotton blends with a mixture of triclosan, polyurethane resin and

a plasticizer was patented by Payne in 2004 [194]. Via the processes of met-mixing and suspension polymerisation Triclosan can be directly integrated into synthetic fibers [195,196]. The antibacterial activity of Triclosan has been found to be less than adequate [197] and breaks down into 2,8-dichlorodibenzo-p-dioxin when exposed to sunlight [198,199] which is related to toxic polychlorinated dioxins [200]. Thus, owing to such environmental concerns, the usage of Triclosan on textile materials in Europe has been banned [73].

1.6.3 Metals and Metal Salts:

Microbes can be easily eliminated with the usage of heavy metals and in the recent years metals like copper, zinc and cobalt have also been researched in eliminating microbes due to the antibacterial properties [73,182,201]. The use of silver in the role of an antibacterial agent has been some concerns, due to the act that some bacteria developing resistance against the action of silver [202,203]. In the case of synthetic fibers, the silver particles can be integrated into their structure before the formation of nanofibers or before extrusion [204-206].

1.6.4 Polyhexamethylene biguanide (PHMB):

PHMB also commercially known as Vantocil has a molecular weight of 2500Da [73]. This has been used as a disinfectant in swimming pools and the possibility of its usage is being looked at in mouthwashes and wound dressings [73,182]. PHMB works by compromising the integrity of the cell membrane and increases with the increase in weight [182]. Onto cotton PHMB can be applied by exhausting it at room temperature or by pad-dry-cure process [73].

1.6.5 Dyes:

The usage of metallic dyestuff which offer antibacterial properties in the textile industry leads to achieving both dyeing as well as an antibacterial finishing while dyeing [73,207]. The usage of azo dispersed dyes for nylon and wool has given excellent antibacterial results [208].

1.6.6 Regenerable N-halamine and Peroxide:

To obtain a durable finish, the regenerable the usage of N-halamine has been explored [73], the N-halamine containing compounds are used in water treatment [77]. The N-Cl bond is the key in acting against microbes and microorganisms. This was studied in 1998 by Sun and Xu [73], after which various compounds have been attached covalently to nylon, polyester, keratinous fibers and cotton or for cellulosic and synthetic fibers, it can also be grafted on [73,209,210].

2.0 Theoretical part:

2.1 Galvanisation:

Since the seventh century, the process of galvanisation has been known to mankind. Galvanisation is a common procedure applied to prevent the corrosion of metals [27]. This is a zinc-based coating process, where a physical barrier is created on the surface of the steel object. This is a total immersion process where the steel is immersed in a bath of molten zinc. The process of galvanisation is used in many industries such as heat exchangers, chains, protective barriers, bolts, nuts, lighting poles electricity distribution towers etc [28,29]. Hot-dip galvanisation and electrodeposition are the main process used to coat the material. In electrodeposition a pure layer of Zn is coated on the steel surface, and this applied with the help of electric current. The layer of coating obtained by this process is generally thinner when compared with the layer obtained via hot-dip galvanisation [30]. The primary element in the process of galvanisation is Zinc (Zn) [27]. The various process in which the Zn coating can be achieved are:

- Hot-dip galvanisation
- Electroplating
- Spraying
- Painting
- Plating [31]

Process of Hot-dip Galvanisation:

The process of hot-dip galvanisation follows the following steps:

- Degreasing
- Rising
- Pickling
- Rinsing
- Flux solution
- Drying
- Zinc bath
- Cooling and clearing [27]

The quality of the coating applied depends on the cleanliness of the surface, thus the first step in the process is to degrease the surface. degreasing is done to remove impurities such as oil, paints, grease or rust from the surface, if rust is present on the surface, the material must be subjected to blast cleaning first. Here the material is immersed in a degreasing liquid, the degreasing liquid is usually an acid bath. After degreasing is completed, the material is then rinsed with water and depending on the end use the temperature of the water is regulated. The following processes of pickling, rinsing, fluxing, drying, galvanisation and cooling are followed in the order stated, after cleaning of the material. Pickling is the process of cleaning the material in an acid bath filled with sulfuric acid or hydrochloric acid. These chemicals are utilised as they generally remove metal oxides and mill scale. Rinsing of the material takes place again after the process of pickling. The process of binding the molten Zn is performed during the fluxing stage. The temperature of molten Zn in the bath is around 450°C. the schematic given below in Figure 1 shows the process of hot-dip galvanisation [27].

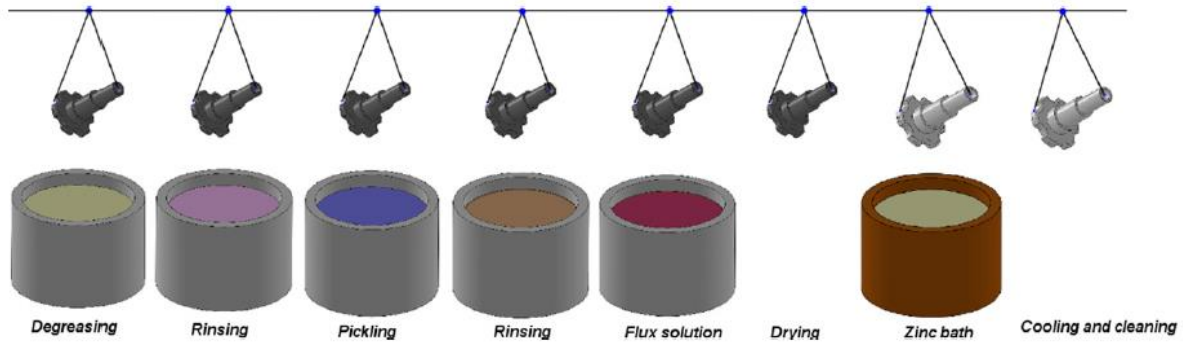


Figure1: Process of hot-dip galvanisation [27]

2.2 Active Silver:

The successful diagnosis and management of diseases depends on the preventing and getting rid of the pathogenic microorganisms. In the recent years it has been found that the various microorganisms have developed a resistance against antimicrobial drugs. But, with the recent advances in the field of medicines based on nanotechnology, it has been found that that the multi drug resistant microorganisms can be overcome with the use of silver nanoparticles (AgNPs). The potential in using AgNPs for treating combating multi drug resistant microorganisms is very high as it has been seen that AgNPS are very potent. The size, surface charge, shape, concentration and colloidal state are the physio-chemical parameters which are critical in determining the potency of AgNPs against the microorganisms. Adhesion to the microbial cell walls, generation of free radicals, generation of reactive oxygen species (ROS),

modulation of the signal transduction pathways and penetrating the cell walls are the mechanisms employed by AgNPs to combat against multi drug resistant microorganisms [32]. The schematic shown in Figure 2 shows the action of AgNPs on the microorganism.

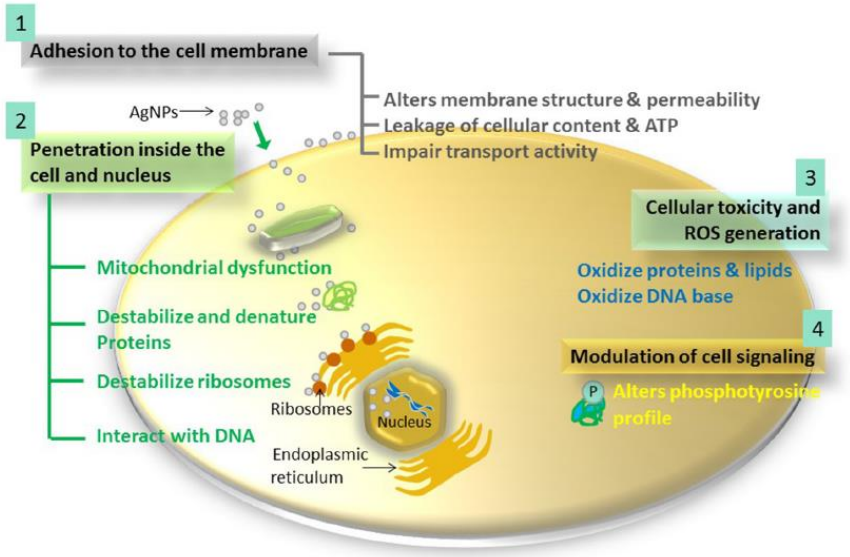


Figure 2: Action of AgNPs on a microorganism [32]

The following table gives the action of AgNPs on a bacteria:

Table 1: Action of AgNPs on a bacteria

Slno	**Bacteria**	**Size of AgNPs(nm)**	**Action**	**Reference**
Gram Negative				
1	Klebsiella pneumonia	<50	Interacts with DNA, prevents cell division	[32]
2	Pseudomonas aeruginosa	5 ± 2	Interaction with cell membrane	[33]
3	Pseudomonas aeruginosa	28	Quorum sensing reduction	[34]
4	Pseudomonas aeruginosa	10	Cell penetration	[35]
5	Vibrio cholera	5 ± 2, 90-100	Interaction with cell membrane,	[33],[36]

			metabolic pathways inhibited.	
6	Salmonella typii	5 ± 2, 2-23	Interaction with cell membrane, lysis of cell wall	[33],[37]
7	Escherichia coli	5 ± 2	Interaction with cell membrane	[33]
8	Escherichia coli	10	Interacts with S and P containing compounds	[38]
9	Escherichia coli	5	Damages cell membranes	[32]
10	Escherichia coli	1-10	Increases membrane permeability and components of the cell are leaked out	[39]
11	Escherichia coli	25	Interacts with S and P containing compounds	[40]
12	Escherichia coli	16	Interacts with S and P containing compounds and Interaction with cell membrane	[32]
13	Escherichia coli	9.3	Interaction with cell membrane	[41]
Gram Positive				

1	Staphylococcus aureus	5	Damages the cell membrane	[32]
2	Staphylococcus aureus	25	Interacts with the cell membrane and inhibits respiration	[40]
3	Clostridium diphtheria	28.42	Cell wall is ruptured and denaturation of proteins takes place	[32]
4	Bacillus subtilis	5	Damages the cell membrane	[32]
5	Bacillus subtilis	10	Chromosomal DNA is degraded and ROS levels are increased	[32]
6	Listeria monocytogenes	23 ± 2	Increases levels of ROS and electron transportation chain is disrupted	[32]

With the introduction of therapeutic agents, the at the site of a wound, the process of wound healing can be accelerated. The local delivery of therapeutic agents like antioxidants, anesthetics, enzymes, growth factors and antimicrobial agents can be comprehensively achieved with the help of electrospun nanofibersn[42]. The advantage of using electrospun nanofibers to deliver theses agents over the commonly used methods of drug delivery system is that, the nanofibers have a fast response rate with a greater control over release rate [43,44]. The therapeutic agents can be introduced into the electrospun nanofibers via the method of co-

axial electrospinning or emulsion electrospinning [45,46]. The process of CO_2 impregnation or infusion or surface immobilization can be utilized to introduce the therapeutic agents into the electrospun nanofiber [47,48]. The table given below gives a list of therapeutic agents that can be incorporated with electrospun nanofibers.

Table 2: List of therapeutic agents

Slno	**Purpose**	**Nanofiber**	**Therapeutic agent used**	**Reference**
1	Antibacterial	Chitosan/PVA	Lysozyme	[49]
2	Antibacterial	Gelatin/polyurethane; gelatin; polyurethane; poly(ethylene-*co*-vinyl alcohol)	Silver	[50-53]
3	Antibacterial	PCL; alginate/PVA	ZnO	[54]
4	Antibacterial	PLGA	Cefoxitin sodium	[46]
5	Antibacterial	Chitosan	Gentamicin	[55]
6	Antibacterial	Polyurethane/dextran; PVA/ poly(vinyl acetate)	Ciprofloxacin HCl	[56]
7	Antibacterial	Cellulose acetate/polyester urethane	Polyhexamethylene biguanide	[57]
8	Pain management and antibacterial	PLLA	Lidocaine, mupirocin	[58]
9	Hemostasis	PLLA	Fibrinogen	[50]
10	Antioxidant	PCL	Curcumin	[59]
11	Angiogenesis	Chitosan/PEO; HA/collagen	VEGF	[60]

12	Angiogenesis, granulation tissue formation	Polyurethane; HA/collagen	PDGF-BB	[61]
13	Keratinocytes migration and maturation, angiogenesis	PCL–PEG/PCL; poly(l-lactic acid)-*co*-poly-(ε-caprolactone); HA/collagen; PCL/PEG	EGF	[62]
14	Cell adhesion, proliferation, ECM secretion, re-epithelialization and skin appendages regeneration, angiogenesis	PELA; HA/collagen	Basic-FGF	[63]

2.2.1 Electrospinning:

In 1934 Formhals patented the process of electrospinning, which explained the production of polymer filaments with the help of an electrostatic force. Thus, the name electrospinning was used for the spinning of fibers using this method. Numerous investigations on the process were conducted [175-180]. In the most uncomplicated form, the electrospinning equipment consists of a pipette to hold a polymer solution, a DC voltage supply and two electrodes. The polymer solution is drawn in the form of fibers from the tip of the pipette ant the fibers are collected at the grounded collector [181]. The needleless electrospinning technique is a very well-known technology and in the recent years, drum electrospinning is being taken over and replaced by continuous wire electrospinning [174].

The table below gives the common types of antimicrobial agents utilised:

Table 3: Common types of antimicrobial agents

Slno	Type	Applied onto	Action	Reference
1	Metals like Ag and others	Cotton, silk, wool, polyester, nylon, viscose	Destroys proteins, lipids, DNA and produces reactive oxygen species.	[64-74]
2	Polybiguanide	Cotton, polyester and nylon	Damages the lipids and causes cytoplasmic sources to leak	[73,72]
3	N-halamines	Cotton, wool, polyester, nylon	Prevents enzymatic and metabolic processes and binds the microbes	[74,75]
4	Triclosan	Acrylics, cellulose acetate, nylon, polyester and polypropylene	The cell membrane integrity is compromised and lipid biosynthesis is prohibited	[73,74,76]
5	QACs	Cotton, wool, nylon and polyester.	Disturbs the DNA, denatures proteins and forms complexes with microbes.	[73,77]

6	Chitosan	Cotton, wool and polyester.	Prevents solutes from being transported towards the cells and prohibits protein synthesis.	[73,78-81]

2.2 Sputtering:

The process of sputtering involves microscopic solid particles being ejected from its after being bombarded by energetic particles of a gas or plasma [82].

2.2.1 Plasma Treatment:

Plasma is known as the fourth state of matter, which is a hot ionised gas which consists of approximately equal number of positively and negatively charged ions. Plasmas are classified as "thermal plasma" or "non-thermal plasma" depending on the relative temperatures, electrons, ions and neutrals. [83]. The classic definition of plasma is a quasi-neutral collection of charged and neutral particles which exhibit collective behaviour.

Plasma is basically a hot ionised gas which contains approximately equal numbers of positively charged ions and negatively charged electrons. The charges are distributed in such a way that the charges are cancel out each other, thus plasma can be considered as electrically neutral. Coulomb's law governs the interaction of the charged particles. Coulomb's law states that like charges repel and the opposite charges attract with a force which is proportional to the product of the charges and is inversely proportional to the square of the distance between the particles. A plasma can be created by coupling electromagnetic power with a gas, the plasma consists of ions, electrons, neutrons, photons, free radicals, meta-stable exited species and molecular and polymeric fragments and the while system is at room temperature.

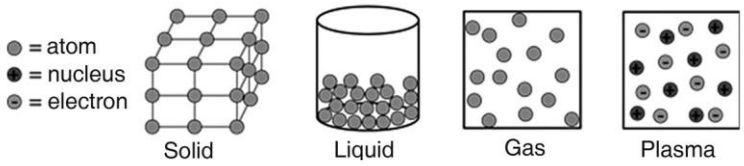


Figure 3: States of matter

With the introduction of the plasma technology in the 1960s, a whole new arena of possibilities was opened up. When the technology was still in its infancy, it was being used

primarily in the non-textile related fields, like in the micro-electronics where low pressure plasma treatments were being used. In the early 1980s plasma treatment was being applied to textiles, to enhance properties such as wettability, adhesion, biocompatibility, anti-wear, chemical affinity. Depending on the relative temperatures of ions, electrons and neutrals the plasmas are classified as thermal plasma or non-thermal plasma. Usually, the non-thermal plasma has low heat content which doesn't heat up the surface of the material which is being treated to such an extent that the bulk properties of cannot be modified. Thus, the non-thermal plasma is used for the modification of the surfaces. A plasma is called a non-thermal plasma if the temperature of the electrons is much higher than the ions and the neutrals. The non-thermal plasma consists of free radicals, ions and other free radicals. The reactive species modify the physical and chemical properties of the materials. But when textiles are treated with plasma the species interact with the surface leading to reactions taking place which would otherwise take place only at a higher temperature. Thus, during plasma treatment of textiles, the thermal degradation of the fabric or textile material is minimal. When the temperatures of the electrons, ions and the neutrals are similar, the plasma produced is known as a thermal plasma, which has high heat content. When this type of type of plasma is used for treatment purposes degradation of the material takes place. The temperature of electrons in non-thermal plasma is in the range of 0.1 eV and the temperatures of the electrons in the thermal plasma can be of the order1.0 eV. Figure 4 shows the entire plasma space [83].

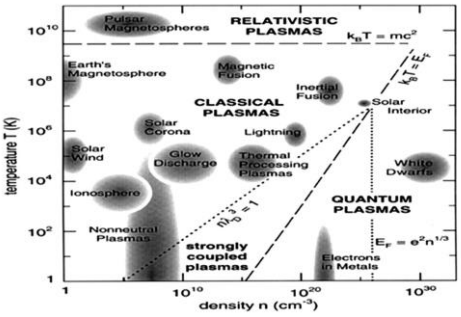


Figure 4: Plasma parameter space showing plasma with different density and temperature

The plasmas are created with the electric break down of the gases, when an electric breakdown occurs a conductive channel is formed. The creation of the plasma occurs mainly when the electron avalanche occurs. Figure 5 shows how the electron avalanche occurs from the anode to cathode.

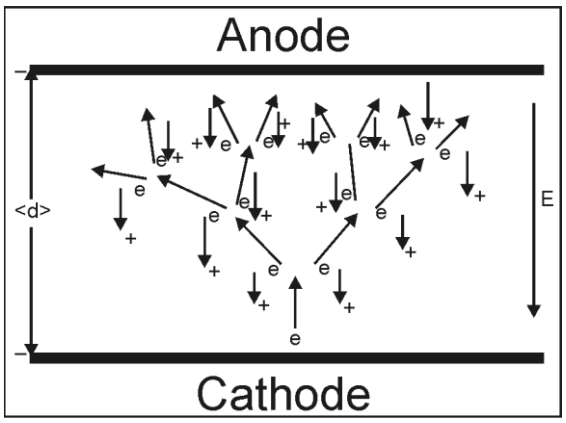


Figure 5: Electron avalanche from Anode to cathode

2.2.2 Plasma Types:

Plasma systems can be used in various applications, the plasma used in the industries can be divided into 2 main forms as given in Figure 6. The "thermal plasma" is produced by dc-ac current sources or by a microwave source under high pressures. This can be utilised to produce anti-wear coatings, gaseous, solid, liquid radioactive halogenated compounds, anti-corrosion coatings or thermal barriers [84]. The cold or non-balance plasma is the second type of plasma utilised. This is usually considered as a low-pressure plasma [85]. This plasma is used for surface modification purposes [84].

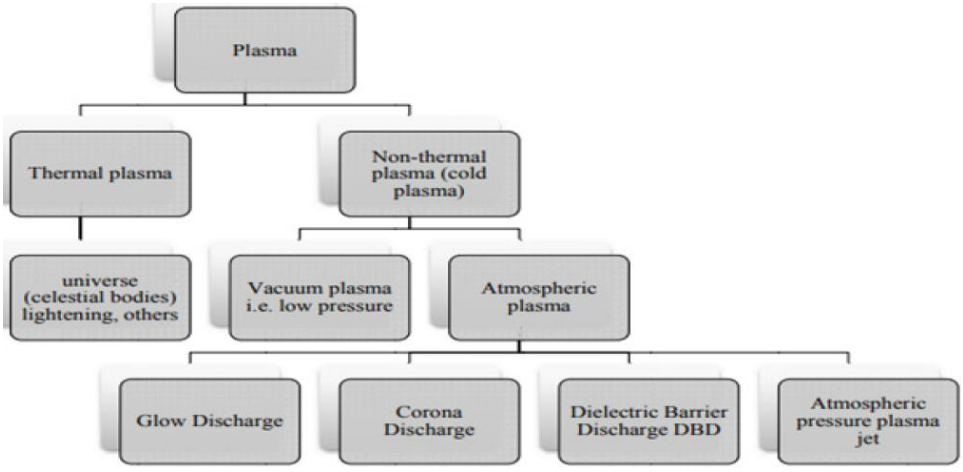


Figure 6: Types of plasma [85]

2.2.3 Plasma treatment in textiles:

The processes of finishing are used to elevate the serviceability or attractiveness of a given textile product [86-92]. The antibacterial, ultraviolet protection, antiseptic properties and flame

retardant properties of fabrics can be improved by treating the fabrics with a low temperature plasma (LTP). The bulk and internal characteristics of the given material are not altered by the plasma but only the surface characteristics are altered when the given material/fabric undergoes a plasma treatment [93,94]. Depending on the plasma gas used and the treatment parameters the chemical properties of the surface can also be altered [95]. An ideal antibacterial finishing should have the following properties:

- Washable
- Non-toxic in nature
- Environmentally friendly
- Durable [96]

The following are the advantages of using plasma in the functionalisation of textiles:

- The risk of fabric exposure is reduced as the plasma is applied at a low temperature.
- The surface of the cloth can be modified effectively due to the wide thermal, chemical and physical ranges.
- Adhesion can be improved.
- Thin films of organic impurities can be removed from the fiber surface.
- Hydrophobic properties can be imparted using plasma treatment.
- The process of plasma treatment is a dry process, thus making it an environment friendly process. [84]

Plasma treatment can be utilised to produce the desired antibacterial finishing on textile fibers and the following table gives a list of common treatment techniques employed on various fibers.

Table 4: Common treatment techniques employed

Slno	**Fibers used**	**Plasma type used**	**Antimicrobial agent utilised**	**Reference**
1	Cotton	DBD (Ar)	Ag, dichlorophenol, triclosan and diphenyl alkane derivative	[97]
2	Cotton	DBD (He and He/O2)	Chitosan	[98]

3	Cotton	Corona (air)	Ag nitrate	[99]
4	Cotton	Corona (air)	Cu and ZnONPs	[100]
5	Cotton	Low-pressure CF4 plasma	AgNPs	[101]
6	Cotton	Sputtering	AgNPs	[102]
7	Cotton	APPJ	AgNPs	[103]
8	Cotton	Low-pressure O2 plasma	ZnONPs	[104]
9	Cotton	APPJ (N2)	5,5-dimethyl hydantoin (DMH)	[104-108]
10	Cotton	Low-pressure plasma (O2 and N2)	Thymol	[109]
11	Cotton	Low-pressure plasma (air or O2)	Neem leaf extract	[110-114]
12	Cotton	Low-pressure plasma (air)	Neem oil vapor	[115]
13	Cotton	Low-pressure O2 plasma	Onion skin extract	[116]
14	Cotton	Low-pressure plasma (Ar)	Ag and Zn	[117]
15	Cotton	Low-pressure plasma (Ar, O2, and N2)	Titanium oxynitride (TiON)/Cu	[118]
16	Cotton	Low-pressure plasma (Ar)	Ag and SiO2	[119]
17	Cotton	DBD (H2/Ar)	AgNPs	[120]

18	Cotton	APGD (Ar)	AgNPs	[121]
19	PP	APGD (He/O2)	DADMAC	[122]
20	PP Mesh	Low-pressure Ar plasma	Ampicillin	[123]
21	PP &PET	DBD (air)	Octenidine	[124]
22	PP	DBD and diffuse coplanar surface barrier discharge (DCSBD) (air)	AuNPs	[125]
23	PP	DBD (H2/Ar)	AgNPs	[126]
24	PET	DBD (air)	Chitosan	[127]
25	PET	DBD (Ar)	Chitosan	[128]
26	PET	DBD (air)	Alkyldimethylbenzylammonium chloride (ADBAC)	[129,130]
27	PET	Low-pressure air plasma	AgNPs	[131]
28	PET	DBD (CO2)	Ag nano-gel and chlorhexidine	[132]
29	PET	APPJ (O2/N2)	Ag, Cu, and ZnONPs	[133-135]
30	PET	Low-pressure plasma	Ag/TiO2	[1]
31	PET	Hollow discharge cathode plasma	Ag	[136]

		(Ar)		
32	PET/Silk	Low-pressure plasma (Ar)	Cu	[137]
33	Nylon/Cotton	APGD (He/O2)	DADMAC	[138]
34	Nylon	APPJ (air)	Chitosan	[139]
35	Nylon	DBD (air)	Poly(*N*-vinylpyrrolidone) (PVP)-coated AgNPs	[140]
36	Nylon	DBD (air)	AgNPs	[141,142]
37	Viscose	Low-pressure O2 plasma	Chitosan	[143]
38	Viscose	DBD (air)	Ag+ and Cu2+ ions	[144]
39	Cotton &Wool	Low-pressure O2 plasma	Chitosan	[145-150]
40	Wool	Low-pressure O2 plasma	Berberine	[151]
41	Wool	DBD (air)	Berberine	[152]
42	UHMWPE	DBD	TMG	[153]
43	Bamboo	DBD (air)	AgNPs	[154]
44	Bamboo	Low-pressure air plasma	Extract of *Aloe barbadensis miller* leaves and *Rosa damascene* flowers	[155]
45	Cotton/PET	DBD (air)	Ag+ ions	[156]
46	Modal	APGD	ZnONPs	[157]
47	Banana	DBD (air)	Green tea and tulsi extracts	[158]

2.3 Spun Blown:

This is a hybrid technology, which is considered to bridge the gap between conventional spun bonding and melt blowing process. The process of production includes muti-rows of spinnerets like the spun bonding process but here the filaments produced are cooled down and attenuated at the tips of the nozzles like in the melt blowing process. Spun blown fibers can be utilised in the following applications:

- Face masks
- Air filtration
- Hygiene products
- Battery separators
- Acoustic insulation
- Thermal insulation
- Sorbents
- Wipes [211]

The schematic in Figure 7 shows the spun blown process:

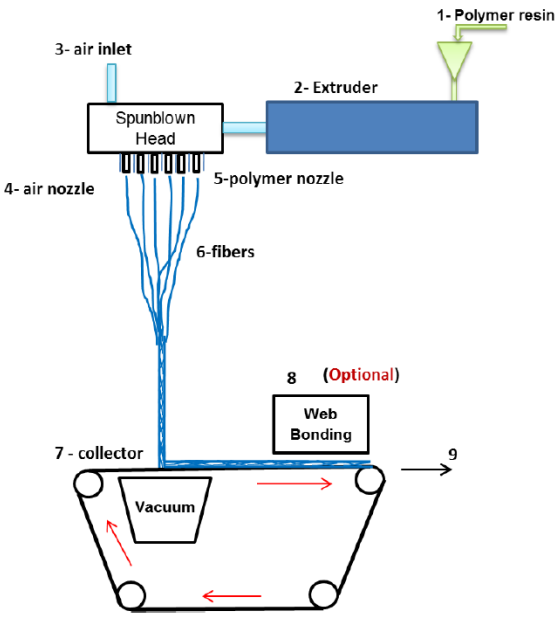


Figure 7: Spun blown process [211]

Parameters of the spinneret used in the spun blown process:

- Upto 104 holes per cm

- Flexible air temperatures
- 2 to 16 rows of holes
- Parallel air jets [211]

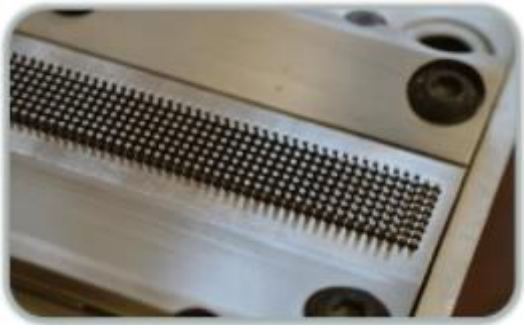


Figure 8: Spinneret of the spun blown machine

Figure 9: Formation of fibers on the Spun-blown machine

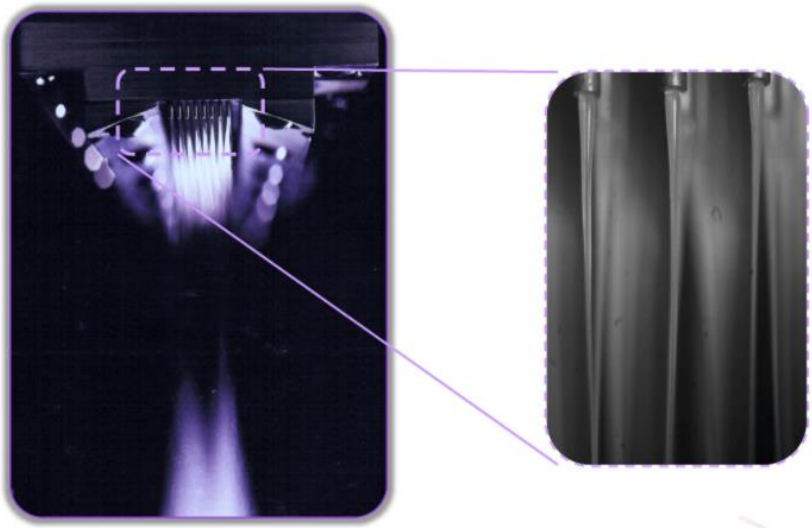


Figure 10: Enhanced image of fiber formation on the spun blown machine

Table 5: A comparison between melt blown, spun bonding and spun blown technologies:

Technology	**Mean Fiber size (µm)**	**Throughput (Kg/m/hr)**	**Nozzle Density (holes per cm)**	**Fiber Strength**
Melt blown	1-5	10-100	10-20	Minor
Spun bonded	3-15	150-300	20-50	High
Spun blown	1-15	10-500	20-104	Medium

The various polymer resins that can be utilised in the spun blown process are:

- PP
- PPS
- PET
- PBT
- PE
- PLA
- Nylon 6

Some of the advantages of using spun blown fibers are as stated below:

- The fibers produced via the spun blown technology are stronger than melt blown fibers and are also smaller in size.
- The spun blown process is cost effective

- The spun blown process can utilise polymers resins used in the spun bonding process as well as the melt blown process.
- Spun blown process has excellent spinning performance when it comes to spinning elastomers.
- The spun blown process has a wide fiber size distribution which can be controlled.
- The fibers produced via the spun blown technology have a high filtration efficiency at lower pressure drops and also have a high dust holding capacity.
- The spun blown process can utilise both cold and hot air.
- The size of the fibers produced from the spun blown process can vary from 0.2 to 15µm. [211]

3.0 Design of Experiment:

To observe and test the antimicrobial/antibacterial activity, agar diffusion tests and suspension tests can be carried out. An Spunbonded-Meltblown-Spunbonded (SMS) nonwoven material deposited with copper via the process of sputtering was utilised in one part of the investigations. In the second part of the investigation polypropylene granulates with active silver were used to produce a spun-blown material to check for its antibacterial/antimicrobial properties.

4.0 Experiment:

4.1 Experiment 1:

In this experiment the materials to be used in antimicrobial/antibacterial roles were prepared via the method of galvanisation. But this method turned out to yield any desirable results, as the coating on the fabrics were negligible or very thin. This led to the experiment not being followed up for further investigations.

4.1.1 Materials Used:

The polypropylene nonwovens of 20 GSM treated with 99.9% silver was utilised in this experiment, the nonwovens were metallised via the process of sputtering and immersed in a solution of $AgNO_3$.

4.1.2 Method of preparation:

The samples used in this experimental method were sourced from Turnex spol. s.r.o. The surface of the fabric was coated with silver via plasma treatment, this was donr to ensure the

conductivity of the polypropylene spunbonded textile material. The thickness of copper obtained was less than 5mm, hence economically the variation in price wasn't significant.

4.1.3 Device used:

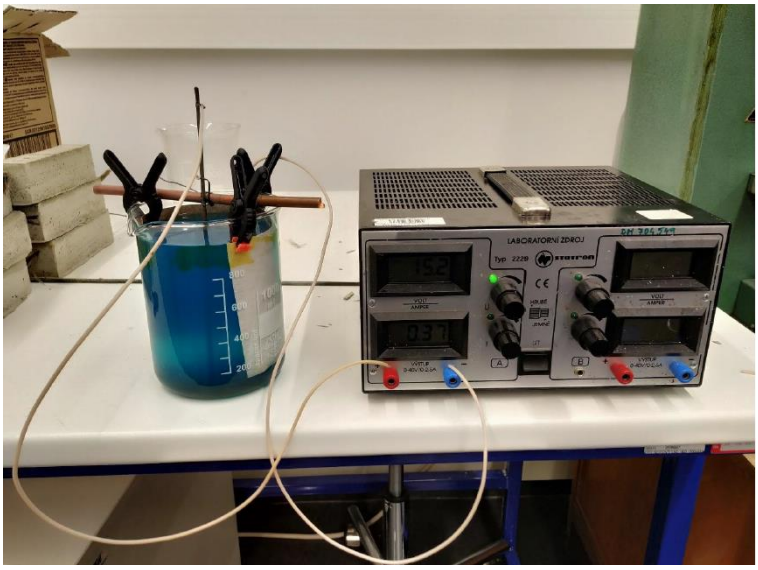
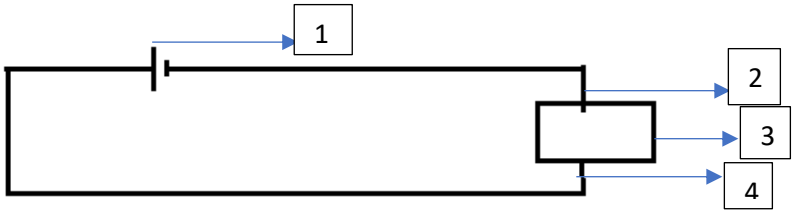


Figure 11: Setup used for Galvanisation

The setup used for the process of galvanisation is represented as shown above in Figure 11. The following are the parameters of the setup used:

- Cathode: copper
- Dc voltage: 5.5Volts
- Current: 0.41Amp
- Current source used: Statron type 2229



1: DC voltage source

2: Cathode

3: Galvanisation setup

4: Anode

Figure 12: Electric circuit

4.2 Experiment 2:

In this experiment using the spunblown process antimicrobial material was produced. The spunblown process is a relatively new process and is a technology which bridges the gap between the melt blowing and spun bonding processes.

4.2.1 Materials Used:

Granulate PP with 1200 MFI was used as basic spun-blown material. was utilised in the spun blown process. The compound at low concentrations is said to achieve a high degree of performance, it also is stable when exposed to light and shows no discolouration with age [212].

4.2.1.1 Composition:

- Resin/ Carrier – LLDPE
- Antimicrobial- 25% Silver-based [212]
- MCX 12009

4.2.1.2 Technical data:

- Recommended let down – 2 to 6%
- FDA application – 2 to 4%
- Pellet size – 60pellets/gram
- Supplied form – Pellets
- Application – extrusion/film/injection moulding
- Heat stability (additive) - 500°C
- Packaging – Gaylord, bag or drum [212].

4.2.2 Method of preparation:

The samples were prepared with 1,2,3 and 4% concentration of active silver by weight.

4.2.3 Device used:

The spun blown machine was utilised here to produce the samples, the general schematic of the spun blown machine is given in Figure7.

5.0 Results and Discussions:

The samples tested have 1 to 4% by weight of the additivities and the following variation of samples were used:

- Control sample (pure polypropylene)
- 1,2,3 and 4 with 2% active silver additives

5.1 Variation in strength:

The mechanical properties of the material with the antibacterial/antimicrobial additives is investigated here and the results are given Table 6 given below:

Table 6: Variation in strength

Concentration of Ag [%]	**MD**		**CD**	
	F [N/50mm]	**Δl [mm, %]**	**F [N/50mm]**	**Δl [mm, %]**
Control	11,43	23,14	8,41	73,11
1%	11,26	29,88	8,18	69,64
2%	9,57	27,28	7,27	56,32
3%	6,43	13,19	6,59	11,88
4%	9,57	20,83	5,92	50,44

It can be seen from the results that the strength of the substrate reduces with the increase in the percentage of the additives used.

5.2 Variation in areal weight:

Table 7 given below shows the variation in weight when the substate has the additives.

Table7: Variation in areal weight

Concentation of Ag [%]	**Control sample**	**1%**	**2%**	**3%**	**4%**
GSM [$g.m^{-2}$]	23,96	23,08	24,22	23,32	23,98

From the results obtained it can be seen that the weight of the samples when compared with the control sample do not exhibit any increase in weight with the increase in the percentage of the additive.

5.3 Fiber Diameter:

5.3.1 Due to additive:

The variation in the diameter of the fibers used is given in Table 8 below

Table 8: Variation in Diameter

Concentration of Ag [%]	**Diameter [µm]**	**St. Deviation [µm]**	**Minimum [µm]**	**Maximum [µm]**
Control	2,11	0,82	0,93	5,63
1%	2,29	1,64	0,56	10,35
2%	2,25	1,61	0,53	10,1
3%	2,81	2,48	0,44	11,98
4%	2,18	1,59	0,59	9,73

It can be seen that there isn't much of a change in the diameter of the fibers expect in the case of 3% Ag. The range of the diameter of the fibers increase with the increase in percentage of additive used a minimum of less than 0,5 µm to maximum of up to 12 µm can be obtained.

5.3.2 Optimization of filtration efficiency

Variation of suction:

The suction force on the collector was varied 10,20,30 and 50% and the variation in the fiber diameter is given in Table 9 below.

Table 9: Variation of Diameter depending on suction intensity:

Suction intensity [%]	**Diameter [µm]**	**Minimum [µm]**	**Maximum [µm]**
10 %	2,17	0,66	8,82
20%	2,27	0,48	9,11
30%	2,66	0,69	10,16
50%	2,16	0,49	12,72

Table 10 gives the efficiency of filtration and Figure 13 gives the fractional filtration efficiency of the samples tested.

Table 10: Filtration efficiency: before optimization

Sample:	**Δp_0 95 (Pa)**	**Δp_0 30 (Pa)**	**Δp_0 160 (Pa)**	**E (0,6 µm)**	**BFE (%)**	**Estimated class (EN 149)**	**Estimated class (EN 14683)**
1%	145,0	41,0	245,0	87,87	100,00	FFP1	Type II
2%	75,0	22,0	130,0	69,70	100,00	no	Type II
3%	35,0	14,0	62,0	30,67	98,00	no	Type II
4%	92,0	28,0	160,0	63,48	100,00	no	Type II
5%	162,0	46,0	274,0	89,87	100,00	FFP1	high pressure

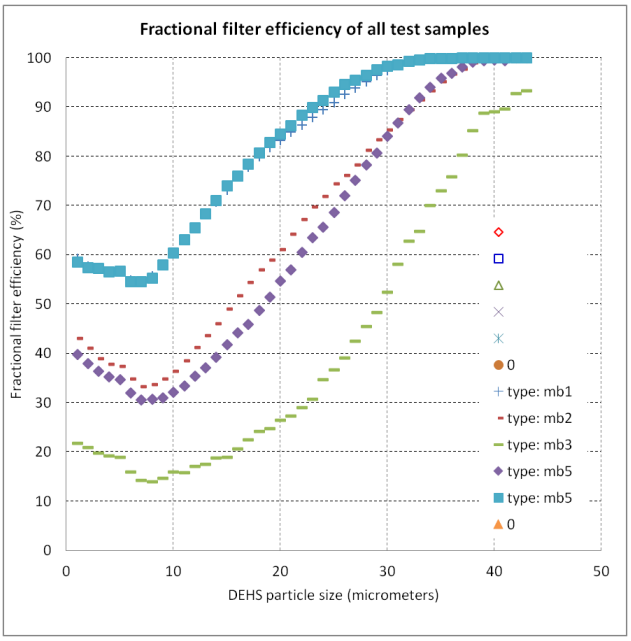


Figure 13: Fractional filter efficiency

After optimisation: the following table gives the results after optimisation.

Table 11: Filtration efficiency: After optimization

Test Results							
Samples	Δp_0 95 (Pa)	Δp_0 30 (Pa)	Δp_0 160 (Pa)	$E_{0,6}$ (%)	BFE (%)	Classification according to EN 149	Classification according to EN 14683
Sample 1 with 10%	151,0	44,0	262,0	94,68	100,00	FFP2	Type II
Sample 1 with 10%	155,0	45,0	266,0	94,54	100,00	FFP2	Type II
Sample 1 with 10%	147,0	43,0	250,0	94,25	100,00	FFP2	Type II
Sample 2 with 20%	170,0	52,0	286,0	96,74	100,00	FFP2	

Sample 2 with 20%	166,0	51,0	283,0	97,41	100,00	FFP2	
Sample 2 with 20%	162,0	49,0	273,0	97,57	100,00	FFP2	

Comparing with table 10 we can see that the optimal suction intensity could be at 20% .

6.0 Conclussions and recommendations:

Here put some results what was observed and so on.....

- The force of suction used to create the vacuum at the collector can be altered. With the a gradual decrease in the intensity of suction, the 20% suction intensity seems to be an ideal value.
- The force at which the air is blown out from the nozzle on a spun blown machine can be altered.
- The drum-collector-distance (DCD) can be varied.
- The temperature of the melt utilised can be altered.
- The temperature of the air being blown out from the nozzle can be varied.
- The MFI of the polymer can varied.

7.0 References:

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