FEDERAL UNIVERSITY OF SÃO CARLOS
CENTER FOR EXACT SCIENCES AND TECHNOLOGY
POSTGRADUATE PROGRAM IN CHEMICAL ENGINEERING

Development of Filter Media from Cellulose Acetate Polymer Containing Silver Nitrate (AgNO3)

Ali Zamanikia

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“Science is the light that illuminates the path of human progress, leading us towards a future full of possibility and wonder.”

(Hafez)

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ABSTRACT

This master's thesis provides a thorough analysis of the creation of nanofiber media with enhanced antibacterial characteristics that are integrated with cellulose acetate polymer and silver nitrate (AgNO3). The study focuses on the synthesis and characterization of electrospun nanofibers, employing a range of analytical techniques, including Fourier Transform Infrared Spectroscopy (FT-IR), filter efficiency, and filter permeability. The primary objective of this research is to understand the influence of varying silver nitrate concentrations (3%, 5%, and 10%) on the properties of cellulose acetate nanofibers. Scanning Electron Microscopy (SEM) analysis reveals that the nanofiber diameter is affected by the addition of silver nitrate, with preliminary findings indicating that higher concentrations result in decreased nanofiber diameter compared to lower concentrations or pure cellulose acetate nanofibers. This observation suggests the potential for tuning nanofiber properties to achieve specific filtration and antibacterial performance. Furthermore, the study investigates the chemical composition and bonding modifications within the cellulose acetate nanofibers. FT-IR spectroscopy reveals spectral changes associated with the incorporation of silver nitrate at different concentrations, indicating possible modifications in chemical composition and bonding. These modifications are suggestive of interactions between silver nitrate and the cellulose acetate polymer, which alter the molecular structure and functional groups within the nanofibers. Also, the permeability and efficiency results provide valuable insights into the impact of silver nitrate concentration on the filter's performance. The findings of this research offer valuable insights into the development of filter media with tailored antibacterial properties, highlighting the potential for optimizing nanofiber diameter and chemical composition through the controlled addition of silver nitrate.

Keywords: Cellulose Acetate, Silver Nitrate, Antibacterial Properties, Fourier Transform Infrared Spectroscopy, Filtration.

RESUMO

Esta dissertação de mestrado fornece uma análise aprofundada da criação de meios de nanofibras com características antibacterianas aprimoradas que são integrados com polímero de acetato de celulose e nitrato de prata (AgNO3). O estudo centra-se na síntese e caracterização de nanofibras eletrofiadas, empregando uma gama de técnicas analíticas, incluindo Espectroscopia no Infravermelho por Transformada de Fourier (FT-IR), eficiência de filtro, e permeabilidade de filtro. O objetivo principal desta pesquisa é compreender a influência de diferentes concentrações de nitrato de prata (3%, 5% e 10%) nas propriedades das nanofibras de acetato de celulose. A análise de microscopia eletrônica de varredura (MEV) revela que o diâmetro da nanofibra é afetado pela adição de nitrato de prata, com descobertas preliminares indicando que concentrações mais altas resultam na diminuição do diâmetro da nanofibra em comparação com concentrações mais baixas ou nanofibras de acetato de celulose pura. Esta observação sugere o potencial de ajuste das propriedades das nanofibras para obter filtragem específica e desempenho antibacteriano. Além disso, o estudo investiga a composição química e as modificações de ligação nas nanofibras de acetato de celulose. A espectroscopia FT-IR revela alterações espectrais associadas à incorporação de nitrato de prata em diferentes concentrações, indicando possíveis modificações na composição química e na ligação. Essas modificações sugerem interações entre o nitrato de prata e o polímero de acetato de celulose, que alteram a estrutura molecular e os grupos funcionais dentro das nanofibras. Além disso, os resultados de permeabilidade e eficiência fornecem informações valiosas sobre o impacto da concentração de nitrato de prata no desempenho do filtro. As descobertas desta pesquisa oferecem informações valiosas sobre o desenvolvimento de meios filtrantes com propriedades antibacterianas personalizadas, destacando o potencial para otimizar o diâmetro e a composição química das nanofibras por meio da adição controlada de nitrato de prata.

Palavras-chave: Acetato de Celulose, Nitrato de Prata, Propriedades Antibacterianas, Espectroscopia Infravermelha com Transformada de Fourier, Filtração.

LIST OF FIGURES

LIST OF TABLES

SUMMARY

1. Introduction

A global epidemic has recently been brought on by brought on by aerosols carrying pathogenic microorganisms, such as bacteria and fungus, or even viruses, and other diseases that are transported by different types of particulate matter. Examples include the end of 2019 cases of COVID-19, MERS in 2012, and SARS in 2003. Technology for removing and protecting pollutants has become more significant for both industry and the general public (Wu, Li, Zhong, Wang, & Yang, 2023).

The requirement for high-performance filtering equipment that falls under the category of clean technologies in industries is significant, and the necessity to create and assess high-performance filter media is growing every day (Jackiewicz, Podgórski, Gradoń, & Michalski, 2013). Among all the equipment for separating fine particles from the gas stream, fiber filters are widely used (Wang & Otani, 2013). These filters are reasonably priced and simple to use. It is feasible to remove very small (nanometer) particles with great efficiency and very little pressure drop by utilizing fiber filters, particularly the non-woven variety (Ding & Yu, 2014). These filters' lifespan, pressure drop, and filtering efficiency are typically used to gauge how well they operate because they all heavily rely on the structure of the filter.

The nanofiltering technique is highly regarded as an effective way to eliminate pollution and microorganisms in the current state of environmental contamination, particularly air pollution (Beni & Jabbari, 2022). The creation of novel nano filter media that can effectively clean air and protect against microbial threats is a key aspect. The small diameter, high aspect ratio, and great specific surface area of nanofibers set them apart (Lyu C. , et al., 2021). Therefore, the ability to create nanofibers from a variety of polymers, as well as the addition of functional molecules and chemical groups, gives them the ideal filtering qualities. This allows for the removal of airborne pollutants and pathogens. Among the preparation techniques the electrospinning is one of the common technologies to prepare nanofibers, which has the advantages of a simple device and straightforward operation and can be utilized with a variety of materials (Fazeli, Fazeli, Nuge, Abdoli, & Shokooh, 2022).

As the basis for these filtration systems, cellulose acetate, a common and biodegradable polymer, offers enormous promise. Cellulose acetate is mostly utilized in the creation of products with diverse surfaces, such as films, fibers, and membranes

(Mishra, et al., 2019; Gomes, Pires, Mateus, & Cruz, 2022). Many recent studies have focused on the application of electrospun cellulose acetate nanofibers as wound dressings and carriers of antibacterial drugs (Çallioglu, 2023; Lei, et al., 2022; Wsoo, Shahir, Bohari, & Razak, 2020).

Furthermore, the antimicrobial and antibacterial properties of silver nanoparticles and silver nitrate make them promising candidates for incorporation into Antibacterial nanofiber media application (Mosselhy, El-Aziz, Hanna, Ahmed, & Feng, 2015; Kim, et al., 2007; Jang H. K., 2008). Therefore, when cellulose acetate coupled with silver nanoparticles, which is well known for having potent antibacterial properties, this composite material can effectively handle problems related to both filtration and disinfection (Majumder, Matin, & Arafat, 2020).

The overall goal of the dissertation is to use cellulose acetate polymer and silver nitrate (AgNO3) to create, produce, and assess nanofilter media with improved antibacterial qualities. It aims to understand how the combination of materials and structure influences antibacterial properties through detailed scientific investigation and experiments. This research focuses on developing advanced filter media, with the potential to benefit environmental/public health efforts. Therefore, this project includes an introduction explaining the history, problem description, objectives, significance, and motivation of the research. A complete analysis of the literature that includes antibacterial agents, cellulose acetate nanofiber filters, the integration of silver nanoparticles, and pertinent earlier investigations follows. The methodology section outlines the nanofiber synthesis, by characterization Fourier Transform Infrared Spectroscopy (FT-IR). Subsequently, the results and discussion section present the main findings obtained from characterizations. Following the references, a summary of the main conclusions, contributions, consequences, and research directions is provided.

2. Literature review

In this chapter, various critical aspects related to air filtration and nanofibers are explored. The subsections delve into specific areas, starting with an examination of the role of nanofibers in air filtration (2.1). Following this, the focus shifts to cellulose acetate polymer as a filter material, discussing its characteristics and suitability for filtration applications (2.2). The subsection on the antibacterial properties of silver and silver nitrate (AgNO3) (2.3) explores the use of silver-based compounds for enhancing the antimicrobial functionality of filters. The subsequent sections explore collection mechanisms (2.4), filter permeability (2.5), and porosity (2.6), addressing key parameters influencing the effectiveness of filters. Filter performance (2.7) is then discussed, emphasizing the broader considerations in evaluating filtration efficiency. The importance of antimicrobial properties for filtration applications is highlighted (2.8), leading to an exploration of nanofiber fabrication through the electrospinning process and the crucial parameters involved. The final subsection synthesizes the diverse elements discussed, specifically focusing on the integration of cellulose acetate nanofibers with silver for enhanced antibacterial properties in filtration applications.

2.1. Air filtration and nanofibers

Air pollution has significantly increased recently as a result of the growth of manufacturing and urbanization. Fine particulate matter (PM) is the main cause of worldwide haze, which has become a major environmental concern (Zhang, et al., 2017). These airborne fine particle pollutants, notably PM2.5 and PM1, have been linked to a number of health problems (Zhu, et al., 2018), including cancer, fibrosis, and chronic lung illnesses, according to several exposure studies (Givehchi & Tan, 2016). In addition, a variety of substances found in airborne particle pollution, such as bacteria, fungi, spores, pollen, and allergens, have the ability to cause allergic reactions and respiratory illnesses (Chuanfang, 2012; Zhu M. , et al., 2017). PM0.3 specifically refers to solid particles or small droplets that may disperse over large distances and have a diameter of 0.3 μm or less. These particles have the ability to enter the bloodstream, penetrate the bronchial system, and carry a wide variety of germs and viruses (Wang & Otani, 2013).

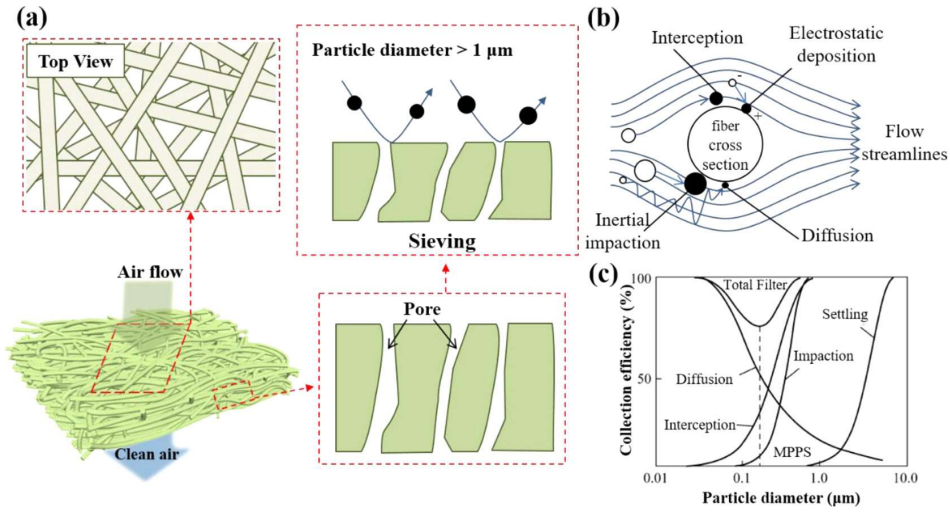
One important way viral diseases like COVID-19 can spread is by airborne transmission (Peron, et al., 2020). Currently, there are practical ways to improve the

declining air quality, such as lowering particle emissions and using passive filtration (Lv, et al., 2018). One effective method of shielding humans from dangerous particles is filtration. To create improved Air Filtration Materials (AFMs), researchers are experimenting with various techniques (Kadam & Padhye, 2018).

In addressing these challenges, researchers are focusing on developing filtration materials with effective particle capture, as conventional fiber filters frequently struggle to efficiently remove tiny particles due to their restricted strength, inconsistent fiber sizes, and wider holes. Furthermore, the air is filled with numerous microorganisms that are inaccessible to typical air filters, in addition to solid particles. Illnesses are spread more easily when bacteria, fungus, spores, and other disease-causing agents are carried by microscopic droplets, aerosols, and particles (Stanford & Chen, 2019). Thankfully, the large surface area, tunable fiber size, porosity, and linked pore structure of electrospun nanofibrous membranes make them excellent filters. As a result, the development of electrospun air filters is receiving more attention. (Lu, et al., 2021).

There are two stages to the air filtering process: steady state and unstable state. The qualities of the filter media, the characteristics of the particles, and the airflow velocity all play a role in the steady state pressure drop and filtration effectiveness throughout the Air Filtration Materials (AFMs). In contrast, as particles build up on the membrane over time, the unstable state causes variations in airflow resistance and filtration effectiveness (Lu, et al., 2021). It is difficult to fully explain this unstable filtering process theoretically due to its complexity. However, the deposition of particles in the air does not appreciably change the thickness of electrospun nanofibrous membranes during filtering since airborne particle concentrations are usually kept low. It follows that stable filtration conditions are expected for the electrospun fiber air filter membrane. Figure1a shows the general composition of an electrospun membrane as well as the particulate matter (PM) removal procedure. As seen in Figure1b, classical filtration theory states that in the steady state, fibers collect airborne particles through mechanisms such electrostatic deposition, Brownian diffusion, inertial impaction, and (Wei, Chun-Shun, & Chao, 2006).

Figure1 Techniques for extracting tiny particles from the air using (a) a single nanofiber and (b) a porous membrane. (c) A single fiber's filtering efficiency using various filtration techniques.



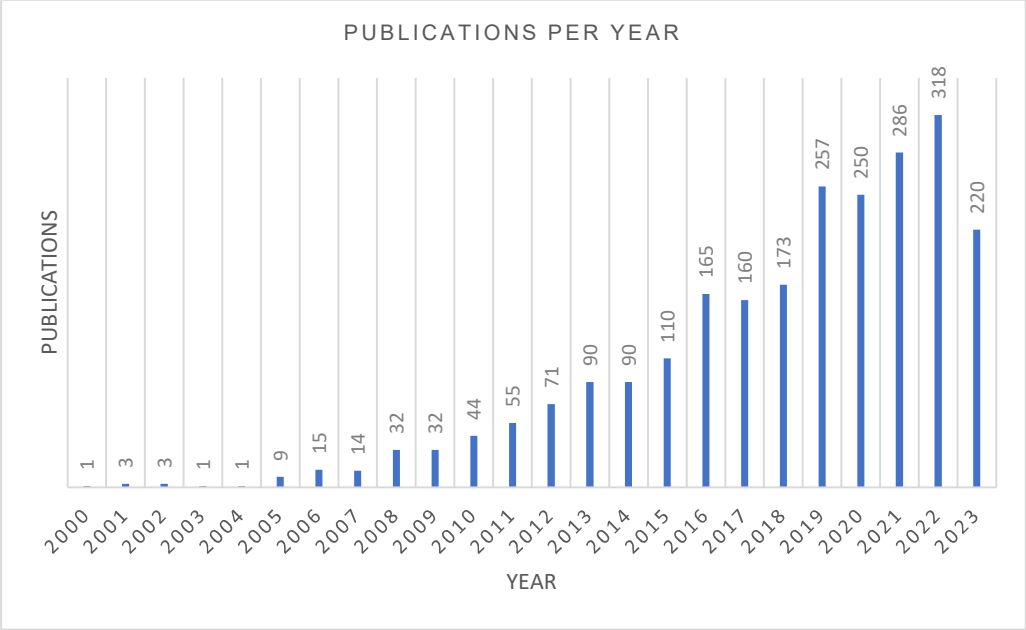
Source: (Lu, et al., 2021)

The changing state of this field's research by searching **'Air filtration and nanofibers'** keyword is depicted in the Figure2. It shows the annual trend of publications in nanofiber-based filtration technologies, giving an illustration of the field's growing importance and level of interest over time. This analysis provides a better understanding of the historical background and developing themes in the field of nanofibers and filtration by looking at the trajectory of scientific output. The graph provides insights into the past, present, and possible future paths for this important field of research.

The number of articles increased gradually from 1 in 2000 to 32 in 2008, but stayed relatively low throughout the years 2000 and 2008. The genuine study and publication boom began in 2010, when the annual number of articles almost doubled. There were 44 articles in 2010, and by 2021, there were 286. This increase was steady. In 2016, there was a notable increase in the quantity of articles, which more than doubled to 165 from the previous year. The trend remained upward in the ensuing years, peaking at 318 articles in 2022. The number of articles dropped to 220 in 2023. This could be because of a number of things, such as funding availability, research trends, or the way external events—like the COVID-19 pandemic—affect research production. With a significant increase in research and publications during the early 2010s, a peak in 2022, and a little fall in 2023, this trend suggests an increasing interest in nanofibers and

filtration technology overall. Despite potential annual variations in publishing rates, the topic appears to be a center of scientific investigation and innovation.

Figure2 Graph of scientific papers published per year in the period 2000-2023 regarding nanofibers used in filtration. From Web of Science

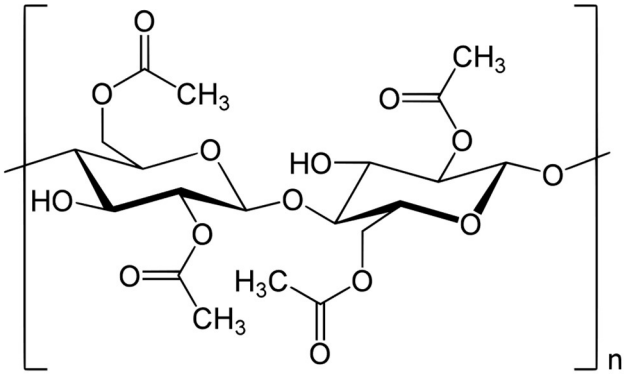


Source: Personal archive

2.2. Cellulose acetate polymer as a filter material

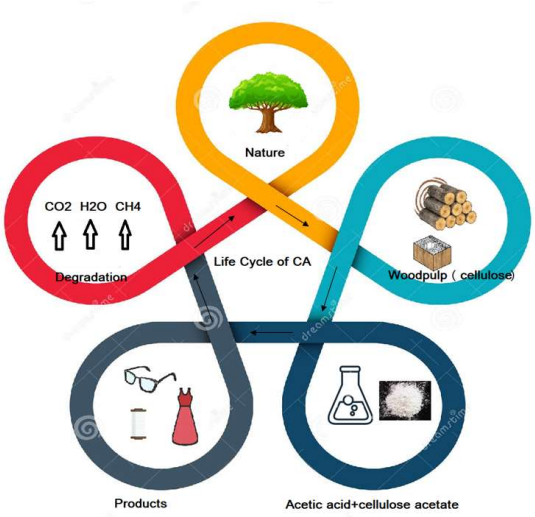
Cellulose acetate is a biodegradable polymer derived from cellulose; a naturally abundant biopolymer found in plant cell walls (Sarkar, Upadhyay, Pandey, & Saha, 2023). The chemical structure of cellulose acetate is shown in Figure3. The life cycle of cellulose acetate involves several stages, from its production to its disposal. It includes raw material sourcing, acetylation, production of cellulose acetate products, use and consumption, end of life options, environmental impact and sustainability considerations, as shown in Figure4.

Figure3 Chemical structure of cellulose acetate



Source: *Ribba, Cimadoro, & Goyanes, 2017*

Figure4 Life cycle of cellulose acetate



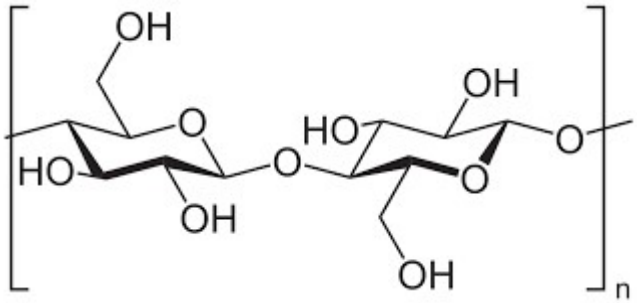
Source: Personal archive

It has gained significant attention in various industries due to its favorable properties, including good film-forming capabilities, excellent mechanical strength, chemical resistance, and biocompatibility (Vatanpour, et al., 2022). These properties make cellulose acetate an attractive material for a wide range of applications, including textiles, films, coatings, and, relevant to this study, filter media (Konwarh & Misra, 2013).

In the following the structure and properties of cellulose acetate are explained. Cellulose is a linear polysaccharide composed of repeating glucose units interconnected β-1, 4-glycosidic bonds. This arrangement forms long, fibrillar chains with both

crystalline and amorphous regions (Sasikanth, Meganathan, logo, Seshachalam, & Gopi, 2023), Figure5.

Figure5 Cellulose structure



Source: *Dey & Abhijit, 2021*

The hydroxyl groups (-OH) of cellulose are partially or fully replaced by acetyl groups (-OCOCH3) through esterification. This modification reduces the polarity of the polymer, enhancing its solubility in organic solvents and improving its processability (Doelker, 2005).

The outstanding film-forming capacity of cellulose acetate is one of its distinguishing qualities. Cellulose acetate can provide solutions that are simple to cast into thin films when it is dissolved in specific solvents, such as acetone or an acetone and water mixture. In order to create coatings with a variety of thicknesses, these films can be deposited onto a variety of surfaces. The ability of cellulose acetate to form films makes it helpful in coating applications including paints, varnishes, and lacquers. These coatings improve aesthetics, protect surfaces from the elements, and give surfaces a smooth finish (Vatanpour, et al., 2022).

In a variety of organic solvents, including acetone, dichloromethane, and ethyl acetate, cellulose acetate shows good solubility. Through solution casting or other deposition methods, this solubility enables the creation of thin films. Flexibility combined with adequate tensile strength characterize the good mechanical qualities of cellulose acetate sheets. This qualifies it for applications that call for strong and resilient materials (Hon, 2017).

The term "biodegradability" describes a substance's capacity to degrade over time into its natural constituents under the influence of microbes, enzymes, and environmental conditions (Alshehrei, 2017). The natural polymer cellulose, which is present in the plant cell walls, is the source of cellulose acetate. A substance that preserves many of the characteristics of cellulose while being more easily degradable is created when cellulose is chemically changed by acetylation in the process of creating cellulose acetate. Comparing cellulose acetate to synthetic polymers like polyethylene or polypropylene, cellulose acetate is known to biodegrade quite quickly. Enzymatic hydrolysis, which involves the breakdown of the acetyl groups linked to the cellulose backbone, is the method used to degrade it. Acetic acid and cellulose fragments are produced as a result of this process, which can subsequently be further digested by microbes or incorporated into the natural carbon cycle. The degree and rate of biodegradation are influenced by things including the environment, microbial activity, and the particular composition of cellulose acetate (Sofi, Akram, Shabir, Vasita, & Sheikh, 2021).

The term "biocompatibility" describes a material's capacity to interact safely with living tissues or organisms (Ghasemi-Mobarakeh, Kolahreez, & Williams, 2019). Due to its natural origin and similarity to cellulose, a substance that is well tolerated by humans, cellulose acetate demonstrates a high level of biocompatibility. This characteristic makes cellulose acetate appropriate for a range of pharmaceutical and medical uses. Because cellulose acetate is often well tolerated by biological systems and has a low toxicity, it can be used in biomedical applications including drug delivery systems and scaffolds for tissue engineering (Foster, 2015).

Cellulose acetate is frequently utilized in the medical industry to create medical fabrics, wound dressings, surgical drapes, and drug delivery devices. When in touch with human tissues or body fluids, its biocompatibility guarantees that there will be few negative reactions. Additionally, the porous structure of cellulose acetate can be modified to regulate the release of medication or other therapeutic agents, increasing the material's potential for precise and controlled drug administration (Teixeira, Paiva, & Felgueiras, 2020).

Due to its biodegradability, cellulose acetate is regarded as being environmentally friendly. Microorganisms are able to degrade it, which helps to prevent waste from building up (Patel, et al., 2011).

Moreover, the processing of cellulose acetate can result in filter media with regulated porosity, permitting fluid flow while successfully removing particles and bacteria (Kaiser & Grass, 2017).

In addition, cellulose acetate has a fibrous structure that can offer a large surface area for particle collection, making it useful for filtration applications (Si, et al., 2016).

Due to its properties, such as biodegradability and mechanical strength, cellulose acetate was chosen as the foundation polymer for producing filter media with antibacterial characteristics. It is a good material for filtration systems because to its biodegradability, mechanical strength, and film-forming characteristics. Also, Silver nitrate (AgNO3) can potentially exhibit enhanced antibacterial characteristics when combined with cellulose acetate, making the resulting nanofilter a desirable option for preventing bacterial development and maintaining the security and effectiveness of filtration procedures. The synthesis, production, and evaluation of filter media in this study are all based on an understanding of the properties of cellulose acetate polymer.

2.3. Antibacterial properties of silver and silver nitrate (AgNO3)

For over a century, silver has been recognized as an antibacterial substance. Silver plates were utilized by the Romans and Egyptians in antiquity to promote ulcer healing and wound healing (McGillicuddy, et al., 2017). Paracelsus employed silver nitrate and even took silver orally to cure wounds (Alexander, 2009). However, it wasn't until John Higginbottom proposed in 1847 that silver nitrate may delay inflammation to reduce problems and heal erysipelas that they discovered how quickly silver nitrate could heal wounds (Higginbottom, 1847). Since β-hemolytic Streptococci of group A produce erysipelas, this phenomenon showed that silver nitrate has antibacterial potential (Inghammar & Linder, 2014).

Later, in 1849, it was discovered that silver nitrate was effective in treating laryngeal ulcers (Bedingfield, 1849). In a study, *Staphylococcus* aureus was treated with 0.5 to 2% silver nitrate solution to demonstrate the antibacterial capabilities of silver (Shi, Wei, Wang, Zhang, & He, 2019). Protargol, largin, albargin, argonin, and argentamine are some of the silver compounds that exhibit antibacterial properties concurrently and may be able to inhibit Neisseria gonorrhoeae, which is known to induce conjunctival infections. Although 0.5% silver nitrate solution did not inhibit epidermal proliferation,

it did kill *Escherichia coli* (E. coli), *Pseudomonas aeruginosa*, and *Staphylococcus aureus*, which provided direct evidence of the solution's safety and effectiveness in the clinic (Sun, 2019).

But since antibiotics first appeared, silver has gradually been supplanted by them. In order to gauge the antibacterial activity of antibiotics like penicillin, silver was subsequently considered a positive control. The high antibacterial activity of antibiotics led to an increase in dependence on them. Antibiotic-resistant bacteria, including methicillin-resistant *S. aureus* (MRSA), penicillin-resistant *S. pneumoniae* (PRP), and vancomycin-resistant *Enterococcus* (VRE), have been observed in clinical settings due to antibiotic abuse that has arisen in recent years as a result of this dependence (Sun, 2019).

Thus, silver's antibacterial properties have garnered attention once more due to the dire circumstances surrounding bacterial medication resistance. Since nanotechnology has advanced, nanomaterials have shown great promise in a variety of industries, such as biomedical devices, cosmetics, medicine, renewable energy, and environmental remediation (Tran & Le, 2013). Consequently, the use of metal medications such as AgNPs and nanodrugs is being seriously explored for the treatment of drug-resistant microorganisms. A mixture of silver and nanomaterials has been found to have potent antibacterial action. AgNP impregnation into medicinal polymers was found by Furno et al. to produce durable antibacterial action, particularly against drug-resistant bacteria. AgNP complexes have been shown by Aymonier et al. (2002) to exhibit strong antibacterial activity, which is altered by amphoteric hyperbranched macromolecules (Sun, 2019).

Because of their broad-spectrum antibacterial properties, AgNPs are now widely used in the biomedical area. For instance, AgNPs are doped in orthodontic appliance baseplates to prevent bacteria that cause cavities from multiplying (Ghorbanzadeh & Bahador, 2015). AgNPs have so far been shown to be effective against a wide range of bacterial species, including *Salmonella typhus, Enterococcus faecalis, Pseudomonas aeruginosa, Escherichia coli, Bacillus subtilis, Vibrio cholerae, Klebsiella sp., Listeria sp.,* and *Acinetobacter sp.* The Activity of AgNPs against sensitive bacteria are listed in Table 1. Antibacterial medications like ampicillin (Amp), vancomycin, linezolid, nisin, sulfonamides, and quorum-sensing inhibitors are also attached as ligands to the surface of AgNPs due to their unique properties, which may strengthen their effects against bacteria that are resistant to antibiotics (Sun, 2019).

These findings suggest that AgNPs may increase antibiotics' antibacterial efficacy. Moreover, superbugs are more susceptible to AgNPs, which is an intriguing phenomenon (Shi, Wei, Wang, Zhang, & He, 2019).

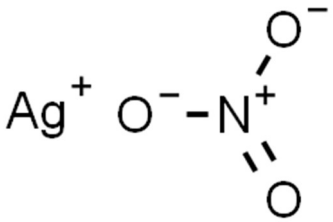
Table 1 AgNPs activity against delicate microorganisms

			Bacteria species	**Zone of inhibition/MIC**	**Size of AgNPs**
Kirby-Bauer disc diffusion method	G+	1	*S.aureus* ATCC 6538	9.1mm	7nm
	G-	2	*P.aeruginosa* ATCC9027	9.1mm	7nm
		3	*E.coli* MTCC433	6mm	66.7-73.9nm
Microdilution method	G+	4	*S.aureus* ATCC 6538	3 μg/ml	8nm
		5	*Staphylococcus aureus* CCM 3953	3.38	26nm
		6	*Enterococcus faecalis* CCM 4224	6.75 μg/ml	26nm
		7	*Pseudomonas aeruginosa* CCM 3955	3.38 μg/ml	26nm
		8	*B. subtilis* MTCC 441	30-50 μg/ml	5-20nm
	G-	9	*P. aeruginosa* ATCC 9027	2.5 μg/ml	8nm
		10	*Escherichia coli* CCM 3954	1.69 μg/ml	26nm

Source: Personal archive

To create filter media with improved antibacterial properties, it is essential to comprehend the antibacterial mechanisms and efficacy of silver nitrate. The chemical structure of silver nitrate is shown in Figure6.

Figure6 Chemical structure of silver nitrate



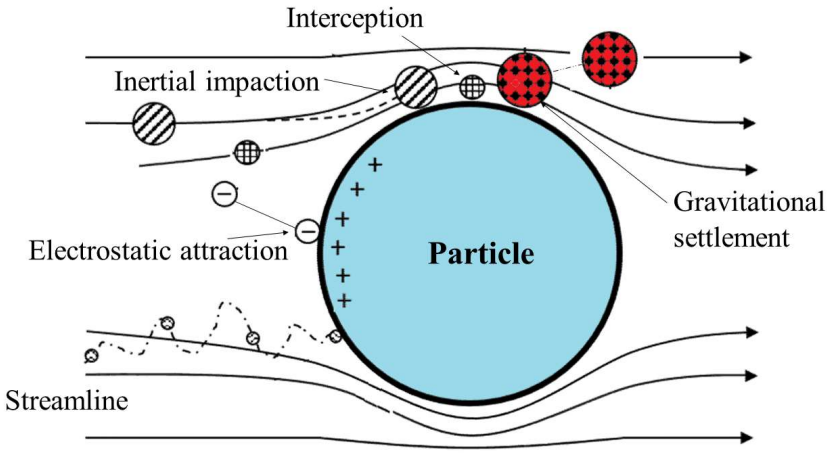
Source: Personal archive

The synthesis, manufacture, and assessment of filter media in terms of their antibacterial properties and possible applications in the field of filtration and antimicrobial techniques are all influenced by our understanding of the antibacterial mechanisms and efficacy of silver nitrate.

2.4. Filtration's Collection mechanisms

Several processes are used in classical filtration theory to catch particles throughout the filtration process. Direct interception, inertial impaction, diffusional effect (Brownian diffusion), gravitational effect, and electrostatic (or electrophoretic) effect are some of the mechanisms that are represented in Figure7. The size of the particles to be filtered, the gas flow velocity, the characteristics of the fluid to be filtered, and the diameter of the fibers that make up the filter medium are some of the variables that determine the prevalence of one or more of these mechanisms. Filtration efficiency is also highly dependent on the distribution of these fibers, which can be successfully regulated by methods such as electrospinning (Oliveira & Guerra, 2021).

Figure7 Filtration's collection mechanisms



Source: Personal archive

The interactions that take place between the gas molecules and the particles that are present in a fluid are known to have a significant impact on the movement of those

particles. Equation represents the dimensionless Knudsen number, which controls this interaction:

$$K_n = \frac{2\lambda}{d_p} \quad (1)$$

where d_p is the particle diameter and λ is the mean free path of the molecules in the gas.

The size of the particle affects the behavior of the aerosol and the laws governing its properties, and this dimensionlessness characterizes the behavior of the particle with respect to the gas. The following formula can be used to find the molecules' mean free path (Barros & Aguiar, 2016):

$$\lambda = 2{,}15 * 10^{-4}.\mu.P.T^{0,5} \quad (2)$$

where T is the gas's absolute temperature, μ is the air's viscosity, and P is the pressure. Particles with reduced diameters, meaning their sizes are equivalent to the mean free path of molecules ($K_n >> 1$), perceive the gaseous medium as a cluster of particles rather than a continuous mass. If, however, the particle is larger than the mean free path ($K_n << 1$), it will see the gas as a continuous medium. When the fluid's particles are tiny and the continuous flow hypothesis is applicable, the Cunningham slip factor (F_s) can be used to adjust for the impact of molecular interactions.

$$F_s = 1 + K_n\left[1{,}246 + 0{,}42 * e^{\left(\frac{-0{,}87}{K_n}\right)}\right] \quad (3)$$

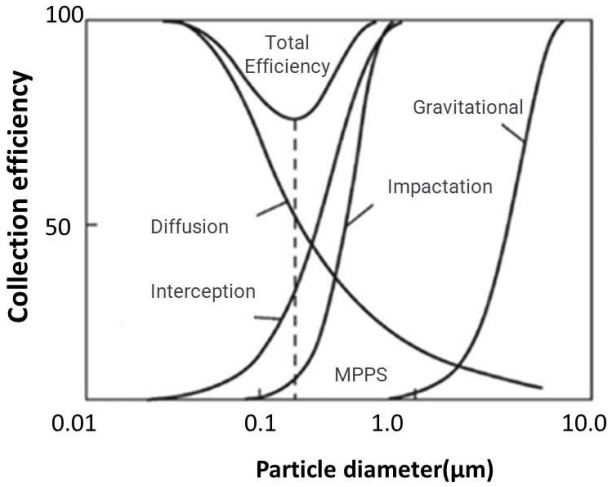
For particles with sizes near the mean free path (λ), the Cunningham slip factor accounts for the non-continuity of the medium. It is therefore easier to give an explanation of each collecting mechanism—which are idealized versions of the physical processes that encourage particle-fiber contact—after these ideas defining the interaction between the gas and the constituent particles are laid out (Barros & Aguiar, 2016).

The following topics will provide descriptions of the five collecting mechanisms along with their corresponding equations. The literature contains information on formulae for calculating fiber processes for particle capture and single fiber efficiency (Liu K. W., 1982).

2.5. Global efficiency

In traditional evaluations, a collector's total collection efficiency is typically calculated as the total of their separate efficiencies. However, it is crucial to acknowledge that this viewpoint might oversimplify the complex interactions between different mechanisms, since the effectiveness of one mechanism can greatly influence another (Lee, Liu, & Liu, 2007; Wang & Otani, 2013). Figure8 provides a more detailed view of this relationship by showing how particle size is closely related to the prevailing processes and, in turn, to the overall efficiency of collection. This empirical description considers a filter with the following specifications: filtration speed set to 0.1 m/s, collector diameter of 2.0 μm, porosity of 0.95%, and thickness of 1 mm (Li, Wang, Zhang, & Wei, 2014). The Figure8 offers important insights into the dynamic nature of particle collection mechanisms in this particular experimental scenario by visualizing the complex relationships and correlations among various parameters.

Figure8 Interaction of collection mechanisms and resulting efficiency



Source: *Li, Wang, Zhang, & Wei, 2014*

It is evident from the examination of the curves in Figure8 that there are areas where more than one collection mechanism predominates. These areas are known as zones of minimal efficiency because they have more particle penetration through the filtering medium. Figure8 illustrates that the inertial and direct interception processes are more active for particles bigger than 1.0 μm, but the diffusional mechanism is more active for particles smaller than 0.1 μm. The previously described region of minimal efficiency

can be observed in the region corresponding to particle sizes between 0.1 and 1.0 μm. The graph's total efficiency curve illustrates the decrease in overall collection efficiency that occurs in this region as a result of increased particle penetration into the filter. Filtration efficiency decreases with increasing diameter (0.1 to 0.4 μm) because the particles are too big for an efficient diffusion effect yet too small to have a major effect on the diffusion mechanisms capture (Lv, et al., 2018).

The use of nanofibers in air filtration applications is justified by the theoretical curves' ability to demonstrate that a reduction in fiber size increases collection effectiveness across all particle size ranges, porosity, and air velocity variations (Balgis, et al., 2015). Moreover, it has been noted that the collection effectiveness in the range of nanometric particles is reduced by an increase in porosity, which is defined as an increase in voids over the thickness of the filtering medium (Bortolassi, et al., 2019). In order to achieve both high collection efficiency and low energy consumption through minimal pressure drop, filter porosity must be adjusted during the filter media's manufacturing process. Lastly, it is important to remember that rising air velocity reduces collection efficiency, which is closely linked to a reduction in the diffusion mechanism's effectiveness for this range of particle sizes (Huang, et al., 2017).

It is crucial to emphasize that a single fiber's characteristics have a significant impact on a fibrous filter's collecting efficiency (Adanur & Jayswal, 2022). The concentration of particles in the air stream at the intake and output can be used to express the experimental filtering efficiency. The following equation can be used to express the experimental filtering efficiency (η):

$$\eta = \frac{G_1 - G_2}{G_1} = \frac{Q\,(N_1 - N_2)}{N_1 Q} = 1 - \frac{N_2}{N_1} \qquad (4)$$

where N_1 and N_2 represent the concentration of particles in the inlet and exit flows ($mg.m^{-3}$), Q represents the volumetric flow ($m^3.h^{-1}$), and G_1 and G_2 indicate the number of particles in the air flow ($mg.h^{-1}$). Based on the mathematical equations describing each mechanism, there exist mathematical models that try to forecast efficiency. As previously indicated, these equations (including their sum) are available at Hinds (Hinds, 2012). Consequently, the overall efficiency is the product of the efficiencies for every mechanism, corresponding to the interplay of all process kinds and primarily dependent on the diameter of the fiber, filtration speed, and particle size, the latter of which is

primarily controlled by the electrospinning procedure, as previously noted (Zhu M. , et al., 2017).

Since the filter media creates flow resistance, the pressure drop is measured close to the front and back of the filter. The filtration efficiency and pressure drop are integrated by the quality factor (QF) in the following way.

$$QF = \frac{-\ln(1 - \eta)}{\Delta P} \qquad (5)$$

where, ΔP is the pressure drop (Han, Kim, & Ko, 2021).

2.6. Filter permeability

The Darcy equation can be used to determine permeability. This is shown in the equation and is used in the case of exclusively viscous flows:

$$\frac{\Delta P}{L} = \frac{\mu v_s}{k_l} \qquad (6)$$

where, ΔP is the pressure drop, L is the thickness of the filtering medium, μ represents the viscosity of the fluid, v_s the surface velocity of the aerosol and k_l is the permeability constant of the filtering medium (Xiao, et al., 2019).

2.7. Porosity

Porosity can be estimated by the Ergun equation (1952), which assesses the proportion of voids existing between fibers. However, experimental methods for determining porosity are also available (Bortolassi A. C., et al., 2019). The Ergun equation, a fundamental tool in fluid dynamics, provides a quantitative framework for understanding the flow of fluids through packed beds, which, in this context, corresponds to the interstitial spaces between fibers. By utilizing this equation, we were able to mathematically express the porosity of the material under consideration, taking into account factors such as fiber arrangement and packing density. This approach enables a rigorous evaluation of the void fraction, contributing to a comprehensive characterization of the porous structure. The application of the Ergun equation in this context underscores the integration of established fluid dynamics principles to elucidate the structural

properties of the material, thereby enhancing the precision and reliability of our porosity assessment.

$$\frac{\Delta P}{L} = \frac{(150(1-\varepsilon)^2 \mu v_s)}{\varepsilon^3 d_f^2} + \frac{(1{,}75(1-\varepsilon)\rho_g v_s^2}{\varepsilon^3 d_f} \quad (7)$$

where ρ_g is the gas density, μ is the gas viscosity, ε is the porosity, v_s is the superficial filtration velocity, d_f is the fiber diameter and L is the thickness of the filtering medium (Almeida, et al., 2020). Therefore, the filtration resistance (ΔP) is expressed by the equation:

$$\Delta P = \frac{2 * C' v^2 L \rho_g}{\pi d_f^2} \ (Pa) \quad (8)$$

where C′ is the experimentally determined resistance coefficient, ν the filtration speed (m/s), L is the thickness of the filtration layer (m), ρ_g is the gas density (kg.m-3) and d_f is the fiber diameter (m) (Qin & Wang, 1285-1290).

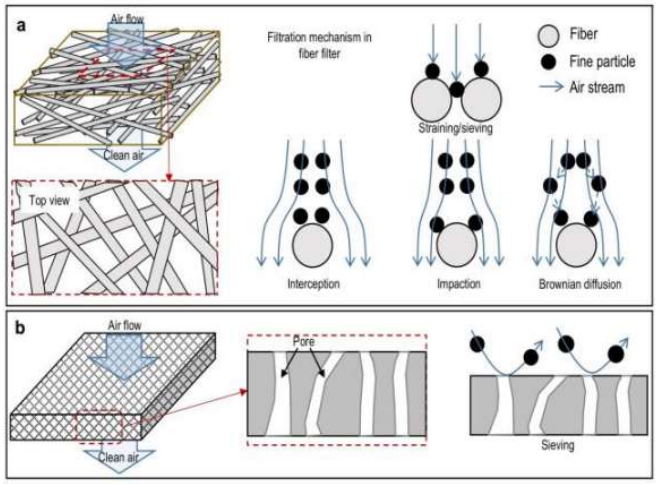
2.8. Importance of antimicrobial properties for a filtration

As one of the environmental issues, air pollution can have a major negative impact on people's health because of the deteriorating quality of the air. A number of activities, including fuel use, transportation, industry, and forest fires, are linked to the degradation of air quality (Fugiel, Burchart-Korol, Czaplicka-Kolarz, & Smoliński, 2017) . Airborne particles, such as tiny particles and bioaerosols, are a type of pollution that have drawn more attention recently due to their ease of mobility. The range in size of bioaerosols is from submicron (< 0.01 μm) to bigger than 100 μm (Smith, et al., 2009). They are kinetically stable and able to hang suspended in the atmosphere for extended periods of time due to their size (Kang, et al., 2016).

A filter media made of different materials, such as carbon, glass fiber, or polymer, is used in air filtering. There are various kinds of air filters, including porous polymeric membrane, nanofiber membrane, and non-woven fiber filter. High efficiency filters are classified as H, or high efficiency particulate air (HEPA), and U, or ultra-low particulate air (ULPA), in accordance with the EN1822 standard (Noor, Kowal, Tiernan, Soulimane, & Tofail, 2016). While ULPA can remove at least 99.999% of 0.1 μm particles, HEPA can only remove 99.97% of 0.3 μm particles. These days, membrane technology is crucial

to many different industrial processes (Komaladew, Khoiruddin, Surata, Subagia, & Wenten, 2018). This technology's benefits led to a sharp rise in the use of membrane applications. Membranes usually need less energy, are less expensive, and have a smaller environmental footprint. Furthermore, the membrane's remarkable separation efficiency can be attributed to its molecular level filtration capability, which is applicable in air purification applications. A membrane used in air filtration typically comprises of non-woven nanofiber or porous material that collects and holds onto particles throughout its thickness or depth (see Figure9). Another name for the most recent membrane is a nanofiber membrane.

Figure9 The mechanism of (a) fiber, nanofiber membrane, or nanofiber filter, and (b) porous membrane for removing fine particles from air.



Source: Komaladew, Khoiruddin, Surata, Subagia, & Wenten, 2018

The fiber diameter of a nanofiber membrane is typically less than 0.1 µm. Melt fibrillation, gas jet, and self-assembly techniques are available for producing nanofiber membranes (Ramakrishna, et al., 2006). Currently, researchers are using electro-spinning because of several limitations with those methods (Komaladewi, Khoiruddin, Surata, Subagia, & Wenten, 2018; W. S. Khan, 2013). Numerous benefits were demonstrated by the nanofiber membrane, including high pore density, high packing density, high permeability, ease of surface functionalization, and superior mechanical properties (Balamurugan, Sundarrajan, & Ramakrishna, 2011).

Numerous studies have altered nanofiber membranes utilizing a range of materials and modifier types (Table 2). Nanofiber membranes have been altered, for instance by

adding nanonets, to enhance the separation property. By adding an extra nanofilter (99.9995%), Liu et al. (Liu, Zhang, Wang, Yu, & Ding, 2015) showed that the nanonets could increase the mechanical strength and particle removal rate of the nanofiber membrane.

Another method of modification was to add a porous nanobead to the fiber media (Komaladew, Khoiruddin, Surata, Subagia, & Wenten, 2018). The beads were said to be able to increase filtration efficiency.

Table 2 Material, modifier, and performance of nanofiber membranes

Material	Modifier	Particulate size (µm)	Flow rate (L/min)	Filtration efficiency (%)	Pressure drop (Pa)
PA-56	Nanofiber/nets	0.3-0.5	30	99.996	100
PAN	Silica Nanofibrous SiO2	0.3-0.5	85	99.99	117
PEI	Nanoparticles Boehmite	0.3	32	95	75
PEI	Nanoparticles BaTiO3	0.3	32	85	58
PI	Carbon woven	0.3-2.5	40	93	462.6
PSF	TiO2	0.3-0.5	30	99.9	40

Source: Komaladew, Khoiruddin, Surata, Subagia, & Wenten, 2018

Bioaerosols, which can cause a variety of health issues for humans if inhaled, can be found in an air stream. Bacteria, viruses, fungus, yeast, fungal spores, pollen, and their fragments (containing a variety of antigens) are the constituents of bioaerosols. People are now spending a higher amount of time indoors, which increases the risk of health issues related to indoor air circulation. Bioaerosols are eliminated using a variety of antimicrobial technologies, including heating and UV light, air ion emission, ozone, and so on. Due to their affordability and ease of use, air filters are now frequently utilized to collect bioaerosols. Bioaerosols can be captured by an air filter, but they also have the potential to develop, persist, and re-suspended into an air stream, posing a threat of becoming a secondary source of pollution. As a result, a lot of research has gone into

creating air filters with antimicrobial qualities by adding antimicrobial chemicals (Table 3), (Komaladew, Khoiruddin, Surata, Subagia, & Wenten, 2018).

Numerous research has reported on the effectiveness of air filters that contain organic and inorganic antibacterial agents (Table 3), (Komaladew, Khoiruddin, Surata, Subagia, & Wenten, 2018). Researchers have recently begun to employ extracts from natural items as antibacterial agents. Natural compounds have a high level of antibacterial activity and are thought to be less harmful than inorganic antimicrobial agents.

Table 3 Material, preparation method, and performances of antimicrobial air filter

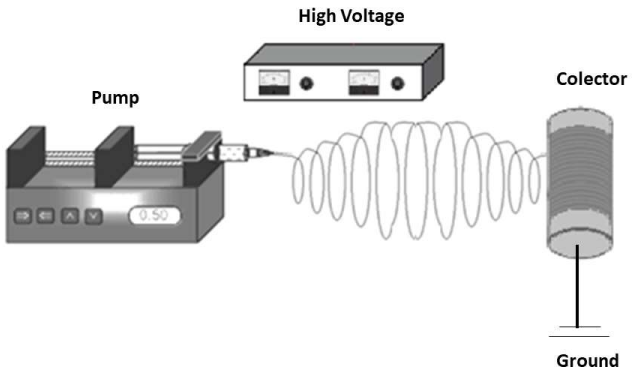
Filter type	**Filter material**	**Antimicrobial agent**	**Preparation method**	**Remarks**
High efficiency air filter media (HEPA)	Fiber glass	ε-Polylysine and Natamycin	Dip-coating	*Staphylococcus aureus*: Filtration efficiency=99.998%, antibacterial efficiency=99.996%
Fiber filter	Polyurethane	Ag/CNTs	Nebulization/aerosol process	*E. Coli* filtraction efficiency=95%, relative microbial viability<0.2%
Fiber filter	Polyurethane	*Euscaphis japonica* methanolic extract powder	Nebulization/aerosol process	*M.luteus*: antimicrobial efficiency=82%,filteration efficiency=95%
Fiber filter	Polyethylene terephthalate (fiber diameter=1.5μm)	Propolins	Nebulization/aerosol process	Penetration levels of bioaerosols=1.4-2%, Inactivation rates=54.4-75.5%
Fiber filter	Polyurethane	Nanoparticles of *Sophora flavescens* ethanolic extract	Electro-spraying	*S. epidermidis:* Relative microbial viability=0%
Polyester air filter	Polyester	PMA-capped silver nanoparticles	Layer-by-layer coating technique	*S. aureus*: reduction rate=92.2%
Nanofiber filter(Fiber diameter=300-400nm)	Sericin/Poly Vinyl Alcohol (PVA)/Clay	Sericin	Electro-spinning	*E. Coli*: Antibacterial activity= 98.3%
Fiber filter	Polyethylene terephthalate (PET)	*Grapefruit seed extract* (*GSE*)	Spray coating	*S. aureus*: inactivation rate=>98%

Source: Komaladew, Khoiruddin, Surata, Subagia, & Wenten, 2018

2.9. Nanofiber fabrication: electrospinning processes

Electrospinning is one of the most flexible ways to create nanofibers. The technique is simple to use and inexpensive. It applies high voltage between a metallic collector and the tip of a needle (Figure10). A pump regulates the flow of the injection of the polymer solution or melt material held within the syringe. The electric field causes the solution drop to stretch and distort as it leaves the needle's tip, creating a Taylor cone. The electric field and gravity work together to draw the viscoelastic jet that is formed when the cone elongates away from the tip. The interaction between the applied electric field and the electric charge carried by a jet produces the tensile force. Surface tension, the viscous flow, and the viscoelasticity of the liquid in the jet all contribute to the transmission of this force. The solvent evaporates enroute to the collector, where it solidifies the fibers (Medeiros, Lima, Almeida, Guerra, & Aguiar, 2022).

Figure10 Strategy for electrospinning



Source: Personal archive

Melt processing is the proper term for the method that uses molten polymer; it is commonly referred to as molten polymer electrospinning. Compared to electrospinning from a polymer solution, this approach is more difficult since the experimental setup needs to take into account the qualities of melting in addition to certain process features

such melting at high temperatures, high viscosity of the melted polymer, and low conductivity. There are variations in the procedure for getting fibers, despite the similarities in the operations themselves (Qin, Qu, Kaschta, & Schubert, 2018). In solution electrospinning, the spinning is caused by the solvent evaporating and the applied electric field; in melt processing, however, the temperature gradient takes on this role (Tapia, Tenorio López, Martínez Estrada, & Guerrero Sánchez, 2019).

That being said, melt processing holds great promise and has many benefits, including the ability to avoid using hazardous organic solvents for post-processing, which is particularly useful in biomedical applications where the residue may be harmful. Second, a larger yield can be obtained since certain polymers, like polyethylene and polypropylene, lack appropriate solvents at ambient temperature and there won't be any mass loss from solvent evaporation. It is frequently quite difficult to find a solvent that solubilizes both polymers in multicomponent systems, like nanocomposites (Lyu C. , et al., 2021).

In order to ensure a steady and controlled feeding rate during the electrospinning process, the solution is maintained in a syringe that is connected to a pump. The droplet at the syringe tip elongates and takes on a conical shape known as a Taylor cone when it is subjected to a strong electrostatic field (Mercante, Scagion, Migliorini, Mattoso, & Correa, 2017). The charged solution jet is ejected onto the collector when the electric field reaches a threshold magnitude where the electrical repulsive force surpasses the surface tension force (Li & Xia, 2004). The electric field can govern this jet's trajectory once it has been charged. The solvent evaporates as the jet moves through the atmosphere, causing the charged polymer fiber to fall at random onto the collector (Robert & Nallathambi, 2020). Applications of this technology have also reported using alternating current (AC) power supplies in place of the more common direct current (DC) power supply for the high voltage source. In addition, various nanofibers may be made by varying the type of needle and its configuration, and the metal collector's movement and form can be changed to regulate the fiber's orientation (Mercante, Scagion, Migliorini, Mattoso, & Correa, 2017). Even though electrospinning is a very simple process, a number of factors linked to the kind of solution, processing, and ambient conditions can have a major impact on the creation and structure of the fiber (Jeanne, Tian, Wang, Tian, & Liao, 2020).

One might emphasize the polymer's concentration, molecular mass, volatility in the solvent, electrical conductivity of the solution, and surface tension in relation to the solution. The electric field's strength, the flow rate, and the distance between the needle tip and the collector can all be found in the processing parameters. It's also important to take into account the environmental factors, such humidity and temperature, that affect how fibers form (M Zhu, 2017). Thus, it can be said that this set of variables directly influences the electrospinning procedure, which in turn influences the properties of the resultant material (Huang, Zhang, Kotaki, & Ramakrishna, 2003).

Many polymeric and inorganic materials have been electrospun up to this point, and it is known that these materials can exhibit a variety of morphologies, including porous surfaces (Jeanne, Tian, Wang, Tian, & Liao, 2020), shell and shell or "core-shell" structures (Mercante, Scagion, Migliorini, Mattoso, & Correa, 2017).

Moreover, electrospinning is a well-liked method for creating nanofiber-based filter media with improved mechanical characteristics and surface area. Researchers have added antibacterial components to electrospun nanofiber mats, including silver nanoparticles, quaternary ammonium compounds, and natural antimicrobial extracts. Electrospun nanofiber filters are potential options for air and water filtration applications since they have shown higher filtration efficiency and excellent prevention of bacterial growth (Ul-Islam, et al., 2016). Research on the comprehensive investigation of electrospun nanofiber membranes for air filtration was done by Bortolassi, et al. [2019]. The research encompassed the optimization of nanofiber manufacturing parameters, including solution concentration, collector to needle distance, flow rate, voltage, and duration, to develop high-quality silver/polyacrylonitrile (Ag/PAN) nanofiber membranes. These membranes were characterized using various analytical techniques to understand their structural and chemical properties. Importantly, the study assessed the filtration performance of these nanofiber membranes, demonstrating their exceptional efficiency in capturing nanoparticles (ranging from 9 to 300 nanometers) from the air while maintaining low pressure drops. Additionally, the research highlighted the nanofibers' remarkable antibacterial properties, showcasing their ability to inhibit the growth of *E. coli* bacteria. This multidisciplinary approach addresses the critical challenges of both efficient nanoparticle removal and antibacterial action, offering promising advancements in air filtration technology for improved air quality control. Also, the design and characterize PAN nanofiber membranes containing different

nanoparticles to enhance their air filtration capabilities and antibacterial properties was studied by Bortolassi et al. Bortolassi, et al. [2019], ultimately improving their suitability for air purification and bacterial removal applications.

Moreover, research on characterization and assessment of various filter media's effectiveness in eliminating nanoparticles was done by (Bortolassi, Guerra, & Aguiar, 2017). Another research on electrospun nanofibers made of biodegradable cellulose acetate (CA) nanofibers and cationic surfactant cetylpyridinium bromide (CPB) for effective airborne nanoparticle retention was studied by (Daniela Sanches de Almeida, 2020).

2.9.1. Important parameters in electrospinning process

Electrospun nanofibers are very desirable for both scientific and industrial applications because of their large surface areas per unit volume, small diameters, and long lengths. From a technological perspective, it is crucial to forecast the electrospun fiber morphology because it is the primary indicator of the quality of the electrospun fiber. It is also recognized that the electrospinning settings determine the fiber shape. Research has been conducted to examine the relationship between electrospinning parameters and electrospun fiber morphology, but thus far no significant progress has been made (Nurwaha, Han, & Wang, 2013).

Numerous factors can affect how the resulting fibers are shaped during the electrospinning process. Three primary groups of parameters can be identified (Khajavi & Abbasipour, 2017):

(1) solution parameters, such as concentration, viscosity, surface tension, and conductivity of the polymer solution;
(2) process parameters, such as applied electrostatic potential, collection distance, and feed rate;
(3) ambient parameters, such as temperature, relative humidity (RH), and surrounding air velocity in the spinning chamber.

An overview of the basic relationships between the morphology of the electrospun nanofibers and processing factors (such as solution characteristics, process parameters, and ambient conditions), which are listed in Table 4, is given in order to produce nanofibers with desired functions and a controlled structure.

Table 4 Classifications of the electrospinning process variables

Solution parameters	Process parameters	Ambient parameters
Material selection	Electromagnetic fields	Humidity
Solvent selection	Spinning distance	Temperature
Concentration	Solution flow rate	Atmosphere
Viscosity	Spinneret morphology	Air movement
Dielectric constant	Collector morphology	
Conductivity		
Surface tension		
Elasticity		

Source: Lennox, 2011

In addition, it is possible to make the general observations listed below (Lennox, 2011):

- The electric potential needs to be high enough to overcome the solution's surface tension.
- The fiber will be thinner the longer it takes the polymer jet to solidify and the farther it travels before solidification. This is because the substance can only flow while it has any fluidity, and the droplet must extend across the distance the fiber is moving (elongation of a fixed volume).
- To produce discernible fibers rather than an interlocking web, the jet must have solidified by the time it reaches the collector. This means that a combination of the time-in-flight, which is related to the spinneret–collector separation, and the evaporation rate of the solvent must, in combination, allow the polymer to solidify before hitting the collector.

2.9.1.1. Solution parameters

Numerous widely used polymer–solvent combinations have been studied, and the solution parameters are extensively reported in the literature. The need to create fibers from a certain material frequently dictates the choice of solution; therefore, the solution

must be designed with the following characteristics in mind: viscosity, conductivity, surface tension, and elasticity (Lennox, 2011).

Many techniques can be used to modify the properties of the solution, such as adjusting the concentration, selecting a different solvent, changing the polymer's molecular weight, and adding additives.

Concentration: During the electrospinning process, the concentration of a polymer solution has a significant impact on the fiber creation. Because of variations in surface tension and viscosity, solution concentration influences the development of fibers. The following factors need to be taken into account in order to calculate a solution's concentration (Khajavi & Abbasipour, 2017):

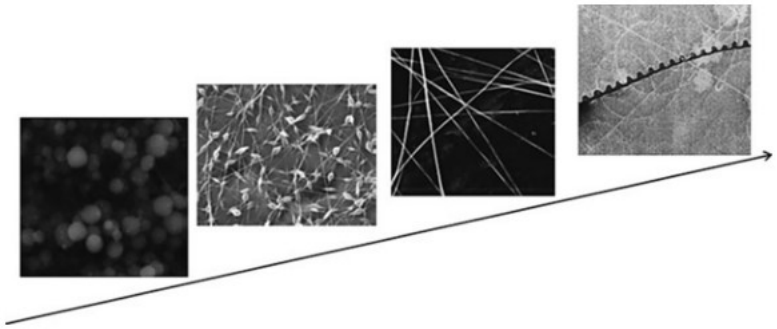
A) Surface tension is the primary influence on fiber shape at very low concentrations. Therefore, if the concentration of the polymer solution is low, the spinnability, also known as "solution viscosity," will be low. Currently, the electrospray process rather than the electrospinning process occurs due to the solution's high surface tension and low viscosity, and drops rather than fibers form (Figure11).

B) The electrospun nanofibers will be unstable due to the somewhat greater concentration (medium concentration), resulting in a mixture of beads and fibers. Beads become more spindle-like and have bigger diameters and longer average distances between them when the concentration of the solution increases (Figure11).

C) Bead-free nanofibers are produced when the concentration is raised to a high concentration because the viscoelastic force becomes dominant and overcomes the surface tension. The talks have made it clear that the concentration of the polymer solution affects both the structure and shape of the fibers as well as their ability to spin (Figure11).

As the concentration of the solution increases, the fiber diameter usually increases as well. Furthermore, the solution concentration can be changed to modify the viscosity of the solution. The impact of concentration on several polymers, such as silk, poly-Dlactic acid, polyethylene oxide (PEO), and polyacrylonitrile (PAN), was examined. With a narrow pore-size distribution, the fiber diameter rose as the concentration of the polystyrene (PS) solution increased (Khajavi & Abbasipour, 2017).

Figure11 Pictures captured by scanning electron microscope (SEM) showing how the products changed in concentration during the electrospinning process, ranging from low to high.



Source: Khajavi & Abbasipour, 2017

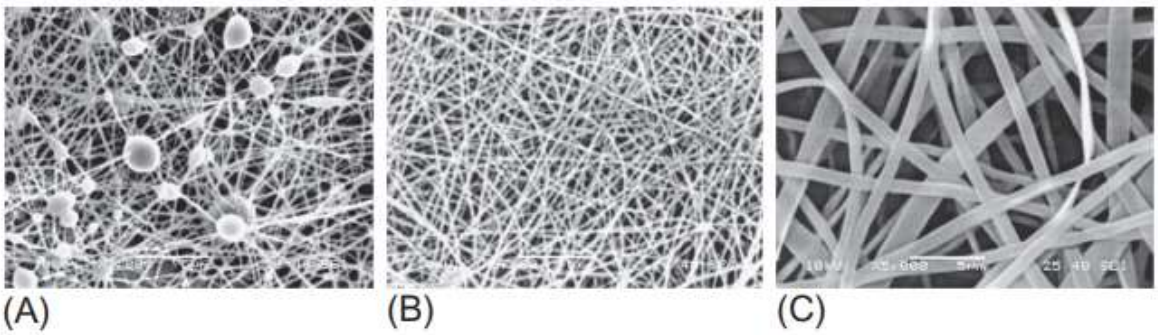
Molecular weight: The morphology of electrospun fibers is significantly influenced by the molecular weight of the polymer. The entanglement of polymer chains in liquids is influenced by molecular weight. Furthermore, the Mark–Houwink–Sakurada equation represents the molecular weight (M) and inherent viscosity [η] of a linear polymer:

$$[\eta] = KM^a \quad (9)$$

Where the constants "K" and "a" depend on the polymer type, solvent, and temperature.

When a polymer's molecular weight tends to decrease at a steady concentration, beads start to form. However, raising the molecular weight will result in smooth fibers. Furthermore, fibers with a micro-ribbon morphology will be produced as the molecular weight increases (Figure12). Even at low concentrations, it has been noted that high molecular weight has a tendency to generate micro ribbons. However, if sufficient intermolecular interactions do place, the molecular weight is not thought to be required for electrospinning (Khajavi & Abbasipour, 2017)

Figure12 Microscopy images showing the variation in the molar mass of cellulose acetate and its influence on the morphology of the nanofibers. (A) 9000–10,000 g/mol, (B) 13,000–23,000 g/mol, and (C) 31,000–50,000 g/mol.

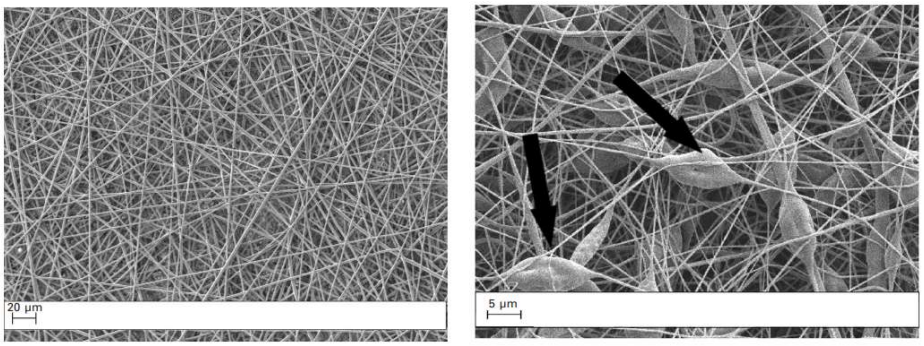


Source: Khajavi & Abbasipour, 2017

Surface tension: Surface tension can play a significant role on its own since the electric field must be able to overcome surface tension in order to form the solution jet. By altering the substance utilized or including surfactants in the mixture, surface tension can be adjusted. It is also crucial for beading along the fiber since high surface tension solutions encourage the growth of beads. Compared to a continuous fiber, beads have a higher volume to surface area ratio. Additionally, the formation of beads reduces the surface energy of the material because less surface is formed. The general goal of electrospinning is to make fibers with a very low volume to surface area ratio, and this works against that goal. Droplets will be produced through a process called electrospraying if the surface tension or viscosity are too high or low, respectively, preventing the formation of fibers (Lennox, 2011).

Volatility of solvent: The type of solvent utilized in the creation of fibers can significantly affect the fibers' characteristics. Because more volatile solvents evaporate more quickly, less spinning distance will be needed. Variations in solvent composition can lead to solutions containing polymers with different viscosities, which can impact the ultimate shape of the fibers. Additionally, it has been demonstrated (Figure 13) that, under the same process conditions, beading in poly-e-caprolactone (PCL) may be eliminated and the mechanical characteristics of the materials produced can be changed by substituting hexafluoroisopropanol (HFIP) for acetone as the solvent (Lennox, 2011).

Figure13 Effect of solvents on the morphology of nanofibers. a) PCL fibers spun at a voltage of 25 kV across a distance of 20 cm in a 10% w/v solution with HFIP. b) PCL showing beading spun from a 10% w/v solution with acetone over 20 cm using a 25 kV potential.



(a) (b)

Source*: Lennox, 2011*

2.9.1.2. Processing parameters

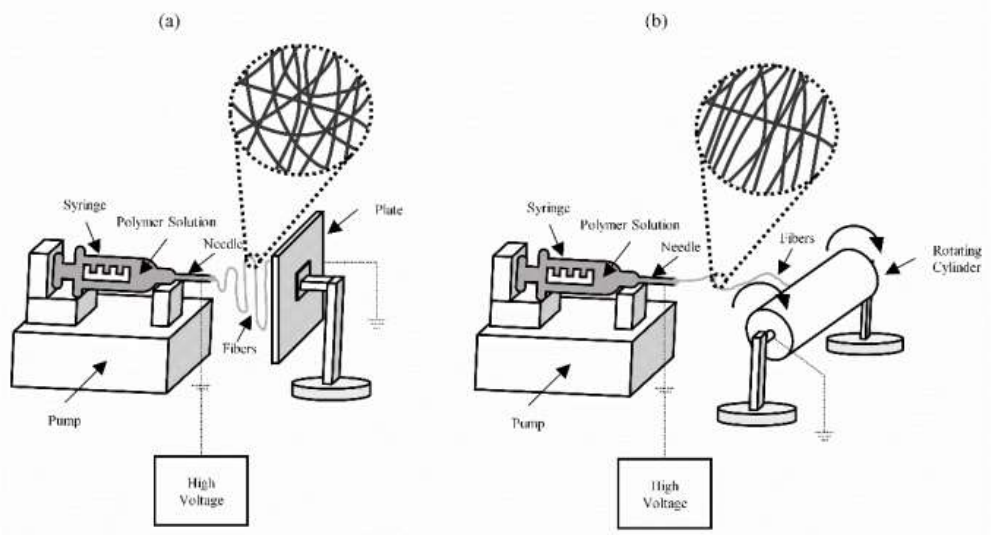
Voltage: An important factor in the electrospinning process is the applied voltage. The viscosity and feeding rate of polymer solutions, together with the initial droplet shape, have all been seen to be impacted. A voltage greater than the critical voltage causes the charged jets to shoot out of the Taylor cone. An electrically charged jet is propelled from a droplet of a polymer solution when a voltage is applied because the liquid's surface becomes charged. This electric force overcomes the surface tension. The polymer solution's original cone form at the spinneret's tip is determined by the electric force. As a result, altering the voltage can influence the morphologies of electrospun fibers (Khajavi & Abbasipour, 2017). Furthermore, the diameter of electrospun fibers is influenced by the applied voltage. According to a number of studies, the decreased fiber diameter was brought about by an increase in the electrostatic repulsive force on the charged jet at higher voltages (Khajavi & Abbasipour, 2017).

Flow rate: One more crucial element influencing the shape of electrospun nanofibers is the polymer solution's flow rate. When the flow rate is low, the polymer solution will have ample opportunity to polarize. Bead fibers with thick diameters occur if it is particularly high (due to low stretching force and quick drying time). For instance, research is done on how flow rate affects the size and shape of polystyrene fibers. When the flow rate exceeded 0.1 mL/min, the authors noticed the production of beads. Furthermore, when the flow rate grew, so did the fibers' diameter and pore size. Yuan et al. (2004) looked into how the flow rate affected the Polysulfone Fiber morphologies in

a 20% PSF/DMAC solution (solution containing Polysulfone (PSF) dissolved in Dimethylacetamide (DMAC)) at 10 kV (Khajavi & Abbasipour, 2017).

Collectors: The kind of collector utilized in the electrospinning process is a crucial factor. The nanofibers in this technique are gathered on a collector, which acts as a conductive substrate. Aluminum flat plates, as shown in Figure14(a) are typically employed as collectors; however, because of the challenges associated with transferring the collected fibers and the requirement for aligned fibers for a variety of applications, research has begun on other collectors, including spinning rods and drums (Figure14b) (Medeiros, Lima, Almeida, Guerra, & Aguiar, 2022).

Figure14 Electrospinning setups and collector types: (a) flat plate; (b) drum collectors.



Source: *Medeiros, Lima, Almeida, Guerra, & Aguiar, 2022*

Tip to collector distance: The morphology of the fibers that are produced is largely determined by the distance between the spinneret and the collector. Jet elongation and thinning only occur when the jet is in flight and remains a fluid, as was previously mentioned. The reason for this elongation is due to a combination of net pull towards the collector and charge repulsion between the ions in the solution. The jet may whip around due to bending instabilities, lengthening its flight distance. Polymer fibers are formed during flight as the solvent evaporates from the surface and the polymer solution hardens. Consequently, if the polymer is not yet solid, increasing the spinning distance will

lengthen the time it takes for thinning to occur and lower the average fiber diameter. It is important to specify the solvent, temperature, humidity, and composition of the atmosphere so that the jet freezes immediately before it hits the collector in order to guarantee that elongation occurs over the entire distance. In addition, considering opposing factors like air resistance and localized charge concentrations, the electric field must be strong enough to accelerate the polymer jet throughout the whole distance. The field strength is impacted by the spinning distance as well. The field diminishes in an inverse square connection with increasing distance (Lennox, 2011).

2.9.1.3. Ambient conditions

Temperature and humidity are examples of environmental factors that can have an impact on the morphologies and diameters of fibers. For instance, a study on an increasing the temperature to produce thinner fibers was carried. An investigation on the the electrospun silk fiber shape at 25, 50, and 75 degrees Celsius was done by researchers. At 25°C, circular morphology was seen, but flat fibers were formed at 50 and 75°C. The resultant fibers at various temperatures fell within the same diameter range. An increase in the electrospinning temperature to 50°C resulted in a small modification of the morphological structure from a circular cross-section to a ribbon-like shape (Nurwaha, Han, & Wang, 2013).

Low humidity causes complete dryness and faster solvent evaporation. Conversely, because of the neutralization of the charges on the electrospinning jet and the tiny stretching pressures, high humidity will cause the fibers to become thicker (Nurwaha, Han, & Wang, 2013).

2.10. Cellulose acetate nanofibers containing silver for antibacterial Properties

Cellulose acetate nanofibers containing silver are of great interest for their potential antibacterial properties. Silver nanoparticles, known for their wide-ranging applications in medicine, electronics, optics, and antibacterial purposes, are particularly suited for integration into cellulose acetate nanofibers (Jatoi, Ogasawara, Kim, & Ni, 2020). People find it valuable for its unique properties, and that's why it's extensively applied in various fields. Silver has a long history of usage in the treatment of bacterial infections, wounds, burns, and cuts due to its antibacterial qualities. Methicillin-resistant

Staphylococcus aureus (MRSA) and other harmful microbial species, as well as fungi, viruses, and bacteria, can be protected against by silver (Duan, et al., 2019). It also works well against cancerous cells. Reactive oxygen species (ROS) or the production of silver ions, or both, have the combined impact of dissolving bacterial cell walls, dimerizing their DNA, and obstructing respiratory chains, all of which contribute to the microorganism's demise. The bacterial inhibition efficacy may gradually decline as a result of silver depletion following excess release. Alternative strategies, like immobilizing the silver nanoparticles on various organic or inorganic substrates, are being explored to get around the negative side effects and achieve bactericidal activity over an extended period of time (Jatoi, Ogasawara, Kim, & Ni, 2020).

The characteristics of cellulose acetate (CA) nanofibers make them ideal for creating an Ag nanofiber composite with antibacterial capabilities. This biopolymer's hydrophilicity, biodegradability, biocompatibility, and moisture-retaining qualities make it very appropriate for antibacterial applications (Zhijiang, Yi, Haizheng, Jia, & Liu, 2016).

There are several researches in the field of cellulose acetate nanofibers containing silver and silver nitrate for antibacterial properties. Research presented a novel approach to produce polymer nanofibers containing silver nanoparticles on their surface (Son, Youk, & Park, 2006). By electrospinning cellulose acetate (CA) nanofibers from solutions with a small amount of silver nitrate (AgNO3) and subsequently exposing them to UV light at 245 nm, the researchers observed the predominant formation of silver nanoparticles on the nanofiber surface. Over a 240-minute period, the number and size of these silver nanoparticles increased. The UV irradiation facilitated the diffusion and aggregation of silver ions (Ag+) and clusters on the CA nanofiber surface. The resulting silver nanoparticles, with an average size of 21 nm, exhibited robust antimicrobial activity. This study introduces a unique method for creating polymer nanofibers with antimicrobial properties by utilizing UV-induced silver nanoparticle generation (Son, Youk, & Park, 2006).

One research is presented to investigate the development of a practical method for creating antimicrobial ultrafine fibers embedded with silver nanoparticles. The process involves direct electrospinning of a solution containing cellulose acetate (CA) and small amounts of silver nitrate, followed by a photoreduction step. The silver nanoparticles within the resulting ultrafine CA fibers are stabilized through interactions with carbonyl oxygen atoms present in the CA. The study demonstrates that these ultrafine CA fibers,

incorporating silver nanoparticles, exhibit potent antimicrobial activity. In summary, the research explores a method to produce highly effective antimicrobial fibers by incorporating silver nanoparticles through a combination of electrospinning and photoreduction techniques (Son, Youk, Lee, & Park, 2004).

Abdul Wahab et al. (Jatoi, Ogasawara, Kim, & Ni, 2020) developed a composite nanofiber material, specifically cellulose acetate/carbon nanotube/silver nanoparticles (CA/CNT/Ag), for antibacterial applications. The researchers aimed to address potential harmful effects associated with silver exposure, such as argyria and argyrosis. To achieve this, silver nanoparticles were anchored onto carbon nanotubes (CNTs) and embedded within the cellulose acetate (CA) matrix. The resulting nanofiber composite was expected to minimize direct contact between silver nanoparticles and human cells, thereby reducing the risk of silver leaching. The study employed various analytical techniques, including scanning electron microscopy (SEM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), transmission electron microscopy (TEM), and antibacterial assays, to characterize the synthesized material. The research aimed to confirm the effectiveness of the CA/CNT/Ag composite nanofibers in inhibiting bacterial growth, suggesting their potential as safer materials for antibacterial applications.

In addition, Muhammad Qamar Khan et al. (Khan, et al., 2020) studied antibacterial wound dressings through the surface modification of nanofibers made from cellulose acetate (CA). The authors aimed to create wound dressings with antibacterial properties without changing the mechanical characteristics of the foundational materials or the nanofiber web. They achieved this by using a straightforward method to functionalize the surface of CA nanofibers and then synthesizing silver sulfadiazine (SSD) nanoparticles on the modified surface. The study involved various characterization techniques such as scanning electron microscopy, transmission electron microscopy, FT-IR, XPS, XRD, water contact angle measurements, and antibacterial agar disc diffusion tests. The results suggested that the developed CA/SSD nanofiber composite could be effectively used as antibacterial wound dressings, particularly against gram-negative bacteria like *E. Coli*, as SSD demonstrated significant potential in combating E. Coli bacteria.

Ki et al. (Jang, Yu, Lee, Kang, & Park, 2014) investigated and compare the antimicrobial properties of cellulose acetate (CA) nanofibers containing silver ions (Ag ions) and/or silver nanoparticles (Ag NPs) against two types of bacteria: Gram-negative *Escherichia coli* and Gram-positive *Staphylococcus aureus*. The researchers created

different types of nanofibers through electrospinning and UV irradiation processes, varying the concentration of silver nitrate (AgNO3) and the deacetylation of UV-irradiated CA nanofibers. The study aimed to assess the effectiveness of these nanofibrous matrices in inhibiting bacterial growth, with a focus on understanding the kinetics of antimicrobial activity against *E. coli* over a 30-minute period. The results would provide insights into the potential use of these nanofibers for applications requiring antimicrobial properties, particularly in the context of wound dressings or other medical materials.

The main differential of this research from the previous studies presented in the literature review is the novel approach in integrating silver nitrate (AgNO3) into cellulose acetate nanofibers for enhancing antibacterial properties. While existing studies have explored the use of silver nanoparticles in various polymer matrices, the use of silver nitrate offers distinct advantages, including potentially improved control over the release of silver ions. This research also focuses on a comprehensive investigation, including detailed synthesis and characterization of the electrospun nanofibers. Additionally, it employs a range of analytical techniques such as Fourier Transform Infrared Spectroscopy (FT-IR), filter efficiency measurements, and filter permeability analysis. These aspects differentiate this study by providing a thorough understanding of the developed nanofiber media and its potential for applications in filtration systems with enhanced antibacterial capabilities.

3. Objectives

This master's thesis pursues two primary objectives: a general objective, which will be elucidated in the following section, and specific objectives.

3.1. General objective

The main objective is to make a type of antibacterial material by adding silver nitrate (AgNO3) to filters made from cellulose acetate polymer. This will improve their ability to fight against bacteria, leading to better hygiene and safety in different uses.

3.2. Specific objectives

Among the specific objectives are:

- Develop filter media using cellulose acetate polymer and incorporate silver nitrate (AgNO3);
- Investigate the antibacterial properties of the developed filter media;
- Evaluate the potential applications like in air filtration systems, particularly in environments where airborne bacteria pose health risks, such as hospitals, laboratories, and industrial settings and benefits of the developed filter media;

4. Materials and methods

In this section, a detailed account will be provided of the materials, devices, and methods employed in the experimental tests for the development of filter media from cellulose acetate polymer containing silver nitrate (AgNO3) with a focus on enhancing antibacterial properties. The description will encompass the reagents used in preparing the solution for producing the filter media, and the generation of particles. The experimental procedure, executed via the electrospinning method, will be detailed. Furthermore, the characterization of the filter media, and evaluation of filtering efficiency and filter permeability will be presented. This section aims to offer insight into the process of developing antibacterial filter media using cellulose acetate polymer containing silver nitrate (AgNO3).

4.1. Materials

The preparation of cellulose acetate nanofibers with varying concentrations of silver nitrate involved, to obtain the bactericidal effect of filter media, the use of specific substances and reagents. These materials included Cellulose Acetate (*Sigma Aldrich*), Dimethyl Sulfoxide (*Supelco*), Acetone (*Synth*, P.M 58,08), and Silver Nitrate ($AgNO_3$, Aldrich).

Solvents played a crucial role in the preparation process, serving to dissolve cellulose acetate and create the nanofiber spinning solution, as shown in Figures 15 and 16. Moreover, solvents also play a fundamental role in the electrospinning of fibers. In the context of electrospinning, several solvent parameters play a crucial role in the process. One of the most relevant parameters is the solution viscosity. The rate of solvent evaporation is directly related to the viscosity of the polymer solution. Rapid evaporation can lead to the formation of smaller diameter fibers, while slow evaporation may result in larger diameter fibers. Controlling the solvent's volatility helps in achieving the desired fiber morphology. Dimethyl Sulfoxide (DMSO) and acetone were selected as the appropriate solvents due to their effective ability to dissolve cellulose acetate.

Figure15 Cellulose acetate solution for electrospinning

Source: Personal archive

Figure16 Preparation of solution

Source: Personal archive

The preparation of four distinct solutions of cellulose acetate nanofibers, each with varying silver nitrate concentrations, followed a systematic procedure:

Cellulose Acetate Nanofibers without Silver Nitrate: To prepare a 5 mL solution, 600 mg of cellulose acetate was precisely weighed and placed in a container. Subsequently, 3 mL of Dimethyl Sulfoxide (DMSO) and 2 mL of acetone were added to the container.
Cellulose Acetate Nanofibers with 3% Silver Nitrate: To prepare a 5 mL solution, 600 mg of cellulose acetate was precisely weighed and placed in a container. Subsequently, 3 mL of Dimethyl Sulfoxide (DMSO) and 2 mL of acetone were added to the container. Finally, 12 mg of silver nitrate was weighed and incorporated into the solution. Vigorous

stirring was conducted for 8 hours until complete dissolution occurred, yielding a 3% silver nitrate solution.

Cellulose Acetate Nanofibers with 5% Silver Nitrate: In the case of a 5 mL solution, the procedure involved accurately weighing 600 mg of cellulose acetate, which was then placed in a container. Subsequently, 3 mL of Dimethyl Sulfoxide (DMSO) and 2 mL of acetone were added to the same container. Following this, 30 mg of silver nitrate was weighed and introduced into the solution. Thorough stirring ensued until all components dissolved, resulting in a 5% silver nitrate solution.

Cellulose Acetate Nanofibers with 10% Silver Nitrate: The preparation of a 5 mL solution commenced with the precise weighing of 600 mg of cellulose acetate and its placement in a container. To this, 3 mL of Dimethyl Sulfoxide (DMSO) and 2 mL of acetone were added. Subsequently, 60 mg of silver nitrate was weighed and added to the solution. A rigorous stirring process was carried out until complete dissolution was achieved, yielding a 10% silver nitrate solution.

4.2. Methods

The method section encompasses two core components: the electrospinning process and subsequent characterization techniques, Fourier Transform Infrared Spectroscopy (FT-IR). This comprehensive approach entails the synthesis of cellulose acetate nanofibers with varying silver nitrate concentrations, followed by a thorough analysis of their structural and chemical properties. Additionally, the filter efficiency and permeability tests are conducted to assess the practical applicability and antibacterial performance of these nanofibrous materials in filtration applications.

4.2.1. Production of filter media (Electrospinning process)

In this study, the electrospinning technique was employed to fabricate both pure cellulose acetate nanofibers and cellulose acetate nanofibers loaded with varying concentrations of silver nitrate. The filter media produced through the electrospinning process were developed in the Environmental Control Laboratory of the Department of Chemical Engineering at the Federal University of São Carlos, as shown in Figure17. The cylindrical collector, which is in constant motion during the experiment, it collected the fibers, since the fibers are produced by the electrospinning process. It is made up of a metal cylinder, which lets the fibers go straight to the collector, a tachometer, and an engine. As one of the most crucial parameters in the electrospinning process, the tachometer regulates the rotation speed. The collector's large size (20 cm in diameter)

allows for the collection of samples over a greater area, enabling tests and characterizations to be performed in triplicate. The pump (Pump 11 Elite, Harvard Apparatus) is depicted in Figure18 and plays a crucial role in maintaining a steady flow rate of the solution, a key variable that must be controlled during the electrospinning process. Special care must also be taken with the high voltage source, responsible for generating the electric field required to form the nanofibers. This voltage is achieved by connecting the needle to the positive terminal and the collector to the negative terminal, ensuring a consistent stream of liquid is expelled from the syringe once the voltage surpasses a critical point. As the solvent evaporates, nanofibers are electrospun and deposited onto the collector's surface. While it can be challenging to observe the formation of these extremely fine fibers during the experiment, they become more evident over time as they accumulate on the collector, forming layers thicker than the substrate. The substrate used in this work is a fiberglass fabric coated with PVC. The voltages employed in the studies range from 13 to 24 kV, as detailed in Table 5. The equipment utilized (HIPOT CC EH 6005C, Electro test) is capable of handling voltages ranging from 0 to 200 kV.Here, the solution-filled syringe is deposited and the solution is progressively released. The electrospinning setup and conditions for each sample were meticulously controlled to ensure reproducibility and precision.

Figure17 Electrospinning equipment

Source: Personal archive

Figure18 Pump used

Source: Personal archive

Several references were used for preparation the materials percentage and the condition of the tests. Tests were first conducted using the optimal settings as described in the literature (Han, Youk, Min, Kang, & Park, 2008; Rasool, Goethem, & Vankelecom, 2020; Kalwar & Shen, 2019). After preparation the solutions, new tests were carried out by changing some process variables and the percentages of materials to have a uniform solutions and better nanofibers. The parameters of the electrospinning technique used for these tests are presented in Table 5. In accordance with the results obtained regarding the thickness of the nanofiber sheets produced, it was found that they were suitable for filtration tests.

Table 5 Operating parameters for solution electrospinning

Sample	Composition	Voltage (kV)	Needle-to-collector distance (cm)	Flow Rate (ml/h)	Collector rotation speed (rpm)	Duration of electrospinning (min)
1	Pure Cellulose acetate	13	10	0.8	295.57	5;15;30
2	Cellulose acetate, 3% AgNo3	22	8	0.8	295.57	5; 15; 30
3	Cellulose acetate, 5% AgNo3	22	8	0.3	295.57	5;15;30
4	Cellulose acetate, 10 % AgNo3	24	10	0.3	295.57	5;15;30

Source: Personal archive

For the production of pure cellulose acetate nanofibers, a polymer solution was prepared using cellulose acetate (50,000 g/mol) dissolved in a 3:2 mixture of Dimethyl Sulfoxide (DMSO) and acetone, respectively. An applied voltage of 13 kV was used, with the needle-to-collector distance set at 10 cm, and a flow rate of 0.8 mL/h to achieve consistent nanofiber formation.

To create cellulose acetate nanofibers containing 3% silver nitrate, the same polymer solution mentioned earlier was utilized, but with the addition of 3% silver nitrate. The applied voltage was increased to 22 kV, the needle-to-collector distance reduced to 8 cm, while maintaining the flow rate at 0.8 mL/h.

In fact, the modification in electrospinning experimental conditions, involving an increase in the concentration of silver nitrate from 0% to 10%, was a strategy to enhance the antibacterial properties of cellulose acetate nanofibers. This adjustment aimed to optimize the antimicrobial efficacy of the filter media, as silver nitrate is well-established for its antibacterial properties. The decision to use a 3% silver nitrate concentration was based on the preliminary study of literatures (Han, Youk, Min, Kang, & Park, 2008; Rasool, Goethem, & Vankelecom, 2020; Kalwar & Shen, 2019). Changes in parameters such as an increased voltage to 22 kV, reduced needle-to-collector distance to 8 cm, and maintaining a flow rate of 0.8 mL/h were necessary to accommodate the modified solution properties, which this process was based on train and error approach. These adjustments ensured successful electrospinning of cellulose acetate nanofibers containing silver nitrate, facilitating the development of a filter media.

Similarly, for cellulose acetate nanofibers with 5% silver nitrate, the electrospinning conditions remained consistent with those of the 3% silver nitrate counterpart. The polymer solution included the 5% silver nitrate additive, and the electrospinning parameters were maintained at an applied voltage of 22 kV, a needle-to-collector distance of 8 cm, and a flow rate of 0.3 mL/h.

Lastly, cellulose acetate nanofibers loaded with 10% silver nitrate were produced using the same polymer solution as for pure cellulose acetate nanofibers but with the incorporation of 10% silver nitrate. The electrospinning conditions were an applied voltage of 24 kV, a needle-to-collector distance of 10 cm, and a flow rate of 0.3 mL/h. In fact, the higher voltage was necessary to overcome the increased resistance in the solution due to the elevated silver nitrate concentration, promoting the formation of well-dispersed

nanofibers. The extended needle-to-collector distance accommodated the changes in solution viscosity and ensured a stable electrospinning process. Additionally, the reduction in the flow rate to 0.3 mL/h was implemented to optimize the deposition of the silver nitrate-containing polymer solution, contributing to the overall effectiveness of the electrospinning process. These controlled electrospinning conditions ensured the precise fabrication of cellulose acetate nanofibers with varying silver nitrate concentrations for subsequent characterization and filter efficiency testing.

Figure19 Filter media after being produced

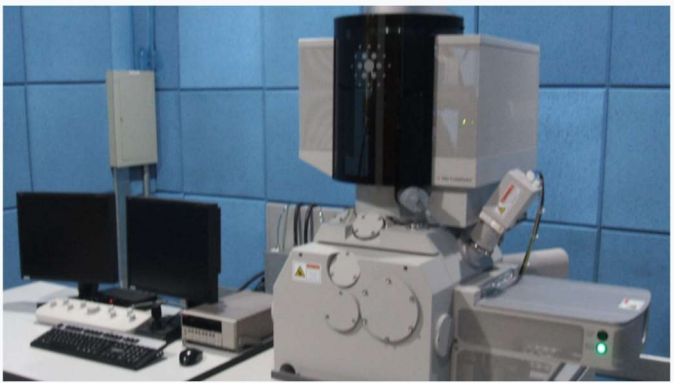
Source: Personal archive

4.2.2. Characterization and performance analysis of filter media

After creating the filtering media, its efficiency and characteristics were analyzed. Scanning electron microscopy (SEM) images, were employed for this purpose by the Magellan Scanning Electron Microscope as shown in Figure20. The FEI Magellan 400 L, located in the Structural Characterization Laboratory (LCE) in Sao Carlos, UFSCAR, is a state-of-the-art field emission gun scanning electron microscope (FEG-SEM) known for its exceptional sub-nanometer resolution. This advanced microscope is equipped with an Energy Dispersive X-ray Spectroscopy microanalysis system, facilitating precise chemical quantification and mapping. Additionally, it features scanning transmission electron microscopy (STEM) detectors, enabling the detailed analysis of thin samples.

Utilizing both manual and automated methods, these images were used to measure the filter medium's thickness, the maximum, minimum, and average pore sizes within the nanofiber layer, and the diameter of the nanofibers. This analysis was conducted through the Image Pro Plus 7 program and the ImageJ plug-in, enabling the determination of critical parameters.

Figure20 Magellan Scanning Electron Microscope (SEM)



Source: Personal archive

The resulting nanofiber samples were subjected to various characterization techniques to evaluate their morphological, structural, and chemical properties. These techniques are detailed in subsequent sections.

In the characterization process of nanofiber air filtration, a comprehensive method has been established to ensure accurate and detailed analysis. The steps outlined below detail the procedure for SEM image analysis and overlay with vertical sections, using Image J software. This method aims to provide a thorough examination of nanofiber morphology and structure.

The characterization method for nanofiber air filtration involves a systematic approach to ensure accurate analysis and insightful data. In the initial step of SEM imaging, a minimum of three SEM images is acquired, showcasing well-defined nanofibers devoid of breakages and agglomerations. The subsequent utilization of Image J enhances visualization and precision in measurements.

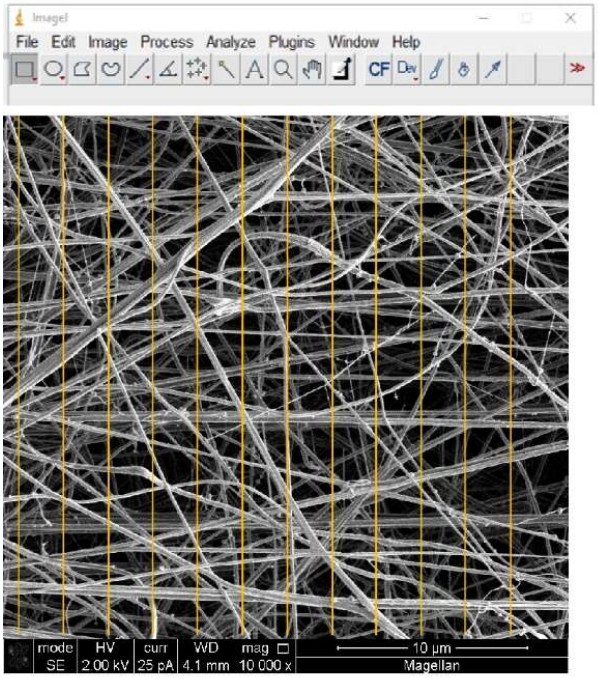
Moving to Step 2, the overlaying of SEM images with vertical sections is executed through a series of specific actions on the Image J platform. This includes uploading the SEM image, navigating to the "Advanced" option, selecting "Overlay image" uploading

vertical sections, adjusting these sections on the SEM image using the "Resize frame" option, applying the changes, and ultimately saving the overlaid image in the "Downloads" folder on the computer, (Figure21).

For calibration purposes, Image J is further employed. The initial step involves opening the SEM image containing yellow lines, which act as reference points for calibration. Subsequently, the calibration is carried out by using the "Magnifying" for zooming in, the scrolling tool for image navigation, and the "Line" tool for measuring the scale using the yellow lines. The "Set scale" option in the "Analyze" menu is then utilized to adjust the distance and unit according to the image caption.

The third step involves measurements, where the "Tools" and "ROI Manager" tables are opened, and clicking on "Measure" displays the measurements in a table named "Results." Finally, the fourth step focuses on saving the obtained measurements either in .xls format or by copying and pasting the data from the "Results" table for subsequent analysis and documentation. This methodical approach ensures a robust and comprehensive characterization process, contributing valuable insights to the overall understanding of nanofiber air filtration for various applications.

Figure21 Scanning Electron Microscopy (SEM) process



Source: Personal archive

4.2.3. Fourier Transform Infrared Spectroscopy (FT-IR)

An extensively utilized analytical method that offers useful insights into the chemical make up and molecular structure of materials is called Fourier Transform Infrared Spectroscopy (FT-IR). It functions on the premise that molecules absorb infrared light at particular wavelengths that are associated with the vibrational modes of their chemical bonds. FT-IR is an effective tool for characterizing materials since it allows one to identify functional groups and chemical bonds inside a sample by examining the resulting infrared spectrum (Burgula, et al., 2007). In this project Fourier transform infrared (FTIR) spectra were obtained between 4000 cm-1 to 400 cm-1 by Nicolet Summit FTIR equipment equipped with a diamond Attenuated Total Reflectance (ATR) unit.

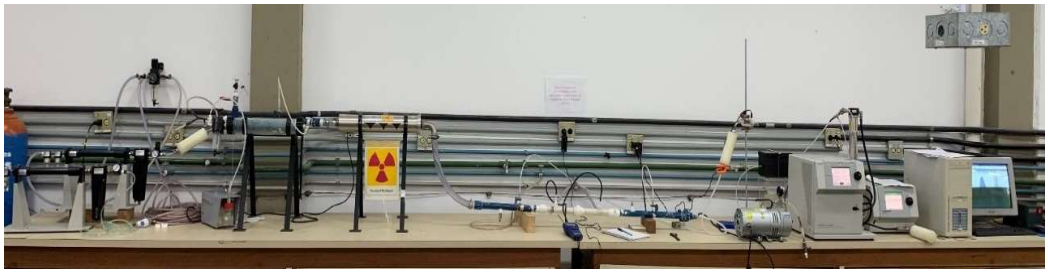
4.2.4. Collection efficiency, Permeability and medium quality factor filter

An experimental unit that was previously provided was used to test the permeability of the filter medium and the efficiency of collecting particles. To improve accuracy, the tests were run using three different batches of the same filter material. Pressure drop (obtained to evaluate permeability) and collection efficiency were used to construct the quality factor.

4.2.4.1. Particle collection efficiency

Following the comprehensive characterization of the filter media, a series of filtration tests were conducted using the experimental unit depicted in Figure22. These experiments were conducted within the facilities of LabCam/DEQ/UFSCar. The primary objective of these tests was to assess the performance of the filter media in terms of collection efficiency.

Figure22 Experimental testing unit for nanoparticles: SMPS



Source: Personal archive

The experimental setup involved exposing the filter media to a controlled particle generation environment. Particles were generated from a 0.1 g/L NaCl solution (Chemis) under a pressure of 1.7×10^4 Pa. The filtration tests were conducted at a consistent speed of 4.7 cm/s, maintaining a standardized filtration area of 5.3 cm^2 throughout the experiment.

To quantify the collection efficiency, it was imperative to determine the particle concentration both before and after passing through the filter media. The Electrical Mobility Particle Analyzer was employed for this purpose. The analyzer was exposed to constant particle generation for a duration of one hour. Subsequently, concentration values were averaged before and after filtration. Equation 4 was then applied to calculate the collection efficiency of the filter medium.

These experiments were conducted under controlled conditions in the Environmental Control Laboratory of the Department of Chemical Engineering at UFSCar, as illustrated in Figure22. The controlled environment ensured the accuracy and reproducibility of the experimental results, providing valuable insights into the filtration performance of the characterized filter media under real-world conditions. The methodology employed in these tests contributes significantly to the overall evaluation of the filter media's effectiveness in particle capture and underscores the practical applicability of the research conducted.

4.2.4.2. Permeability of the filter media

The determination of permeability was a meticulous process involving the measurement of pressure drop at specific volumetric flow values, carefully selected to cover a range from 200 to 2032 cm^3/min. This approach allowed for a comprehensive examination of the filter medium's behavior across varying flow conditions. The aerosol's surface velocity, a critical parameter indicative of filtration efficiency, was subsequently calculated by considering the volumetric flow rate and the established filtration area.

Precision in measurement was ensured through the utilization of a digital manometer, which accurately gauged the pressure drop at each of the pre-established flow values. The filtration area, set at 5.21 cm^2, served as a consistent reference, contributing to the reliability of the data. The assumed viscosity of the fluid, at 1.74×10^{-5} Pa s.

To standardize the data and facilitate meaningful comparisons, corrections were applied for ambient temperature and pressure. Subsequently, the measured values were averaged, providing a representative overview of the filter medium's performance. A graphical representation was then generated, depicting the relationship between pressure drop and the thickness of the filter medium as a function of surface velocity.

5. Results and discussion

In this session, the results obtained from the methodologies according to the subtopics defined in the experimental procedures will be presented and discussed.

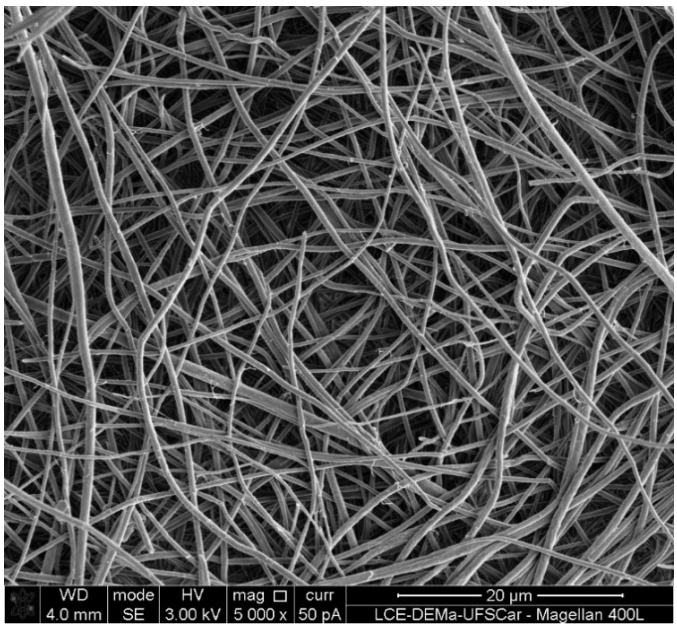
5.1. Result of Scanning Electron Microscopy (SEM)

In the Scanning Electron Microscopy (SEM) analysis, we explored the morphological characteristics of cellulose acetate nanofibers under different conditions, specifically cellulose acetate nanofibers (CA), cellulose acetate nanofibers with 3% silver nitrate (CA-3%$AgNO_3$), cellulose acetate nanofibers with 5% silver nitrate (CA-5%$AgNO_3$), and cellulose acetate nanofibers with 10% silver nitrate (CA-10%$AgNO_3$). SEM figures were obtained to assess the nanofiber diameter distributions and surface topography for each case, shedding light on the impact of varying silver nitrate concentrations.

5.1.1. Cellulose acetate nanofibers

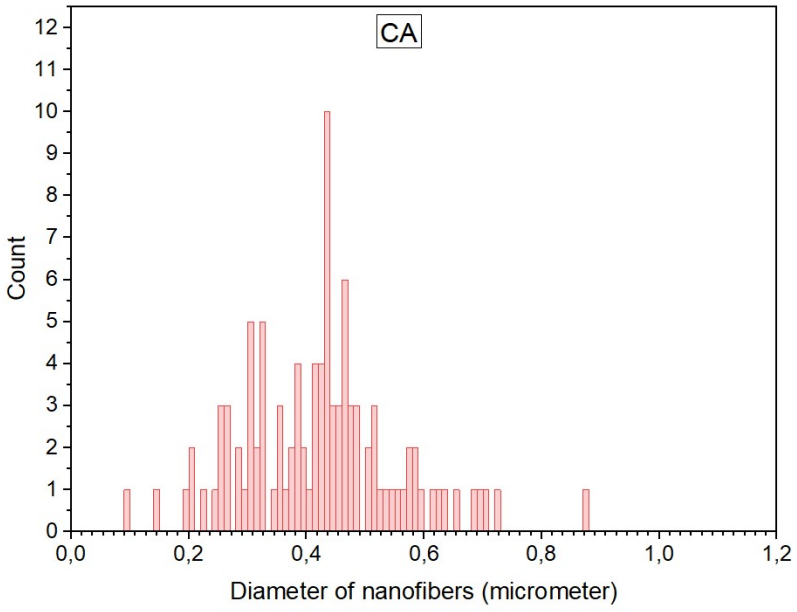
In Figures 23 and 24, are observed the morphology of pure cellulose acetate nanofibers. The SEM image displayed a distribution of nanofiber diameters.

Figure23 SEM image of pure cellulose acetate nanofibers



Source: Personal archive

Figure24 Fiber diameter distribution of pure cellulose acetate nanofibers

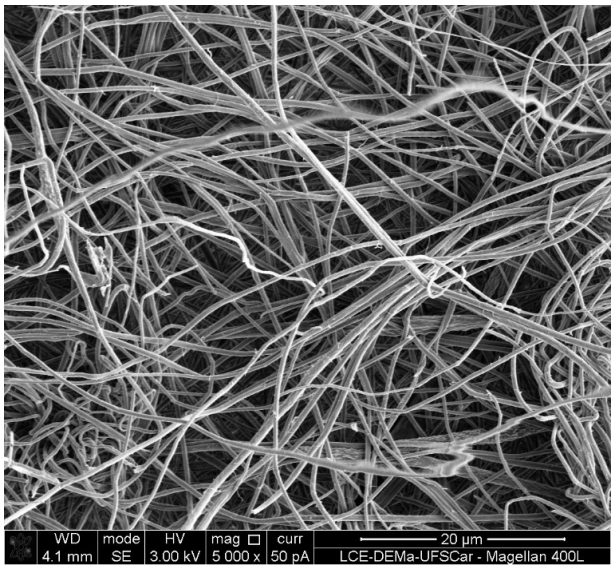


Source: Personal archive

5.1.2. Cellulose acetate nanofibers with 3% silver nitrate

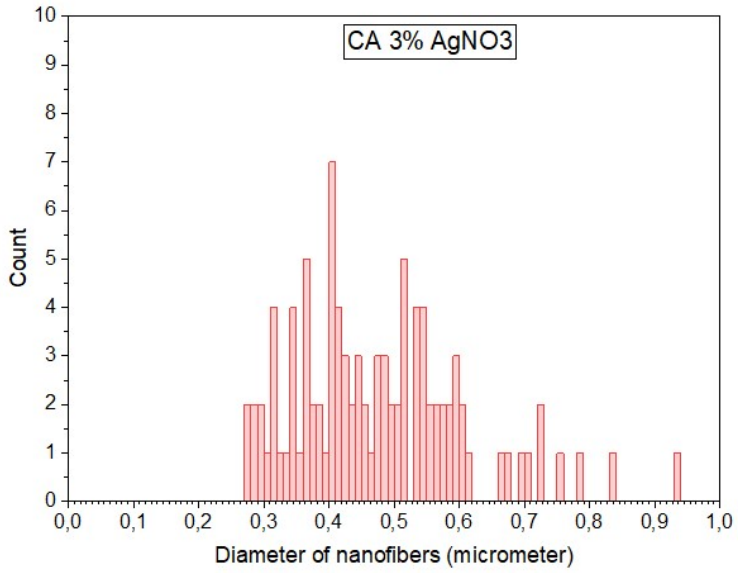
Figure25 and 26 illustrated the nanofibers prepared with 3% silver nitrate. The SEM image shown another distribution of nanofiber diameters, potentially demonstrating a more pronounced impact on morphology compared to the pure cellulose acetate. This variation in nanofiber diameter distribution was indicative of the influence of silver nitrate on the electrospinning process.

Figure25 SEM image of cellulose acetate nanofibers with 3% silver nitrate



Source: Personal archive

Figure26 Fiber diameter distribution of cellulose acetate nanofibers with 3% silver nitrate

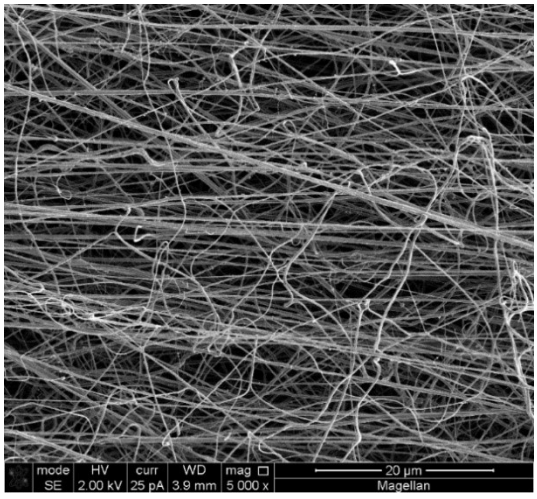


Source: Personal archive

5.1.3. Cellulose acetate nanofibers with 5% silver nitrate

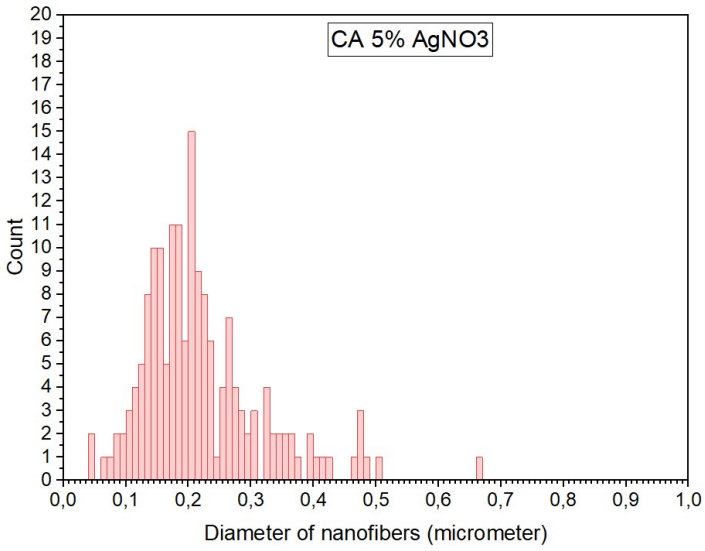
Figure27 and 28 illustrated the nanofibers prepared with 5% silver nitrate. The SEM image shown another distribution of nanofiber diameters, potentially demonstrating a more pronounced impact on morphology compared to the lower 3% silver nitrate concentration. This variation in nanofiber diameter distribution was indicative of the influence of silver nitrate on the electrospinning process.

Figure27 SEM image of cellulose acetate nanofibers with 5% silver nitrate



Source: Personal archive

Figure28 Fiber diameter distribution of cellulose acetate nanofibers with 5% silver nitrate

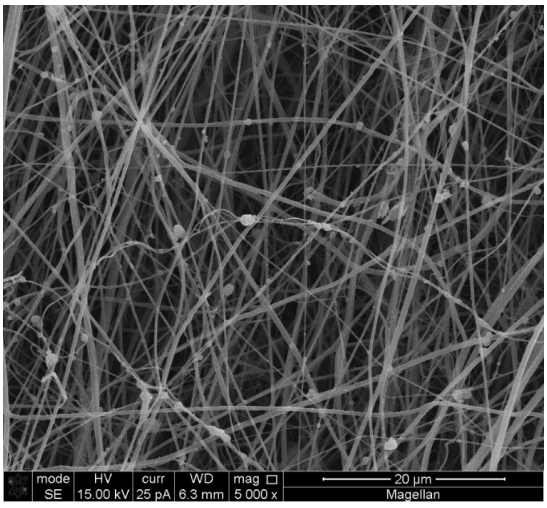


Source: Personal archive

5.1.4. Cellulose acetate nanofibers with 10% silver nitrate

Finally, Figures 29 and 30 shown the nanofibers synthesized with the highest concentration of silver nitrate, namely 10%. The SEM image highlighted a distinct distribution of nanofiber diameters, potentially indicating a more significant effect on morphology compared to lower silver nitrate concentrations (3% and 5%). The presence of 10% silver nitrate appeared to have a substantial influence on nanofiber diameter, further emphasizing the importance of concentration in modulating the nanofiber structure.

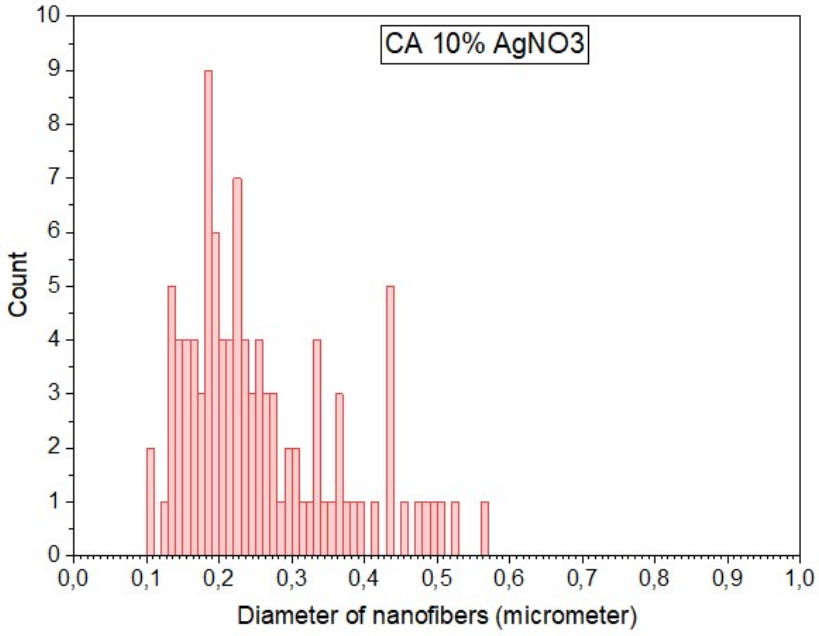
Figure29 SEM image of cellulose acetate nanofibers with 10% silver nitrate



Source: Personal archive

The presence of beads in the cellulose acetate nanofibers containing 10% silver nitrate, as depicted in Figure29, can be attributed to the challenges associated with the electrospinning process under the specific experimental conditions employed. The elevated concentration of silver nitrate introduces changes in solution properties, such as increased viscosity and conductivity, which can disrupt the uniformity of the electrospinning jet. The higher viscosity may lead causing irregularities in the nanofiber deposition and resulting in the observed bead formation. Additionally, the electrostatic forces acting on the polymer solution may become less balanced with the increased concentration of silver nitrate, contributing to the formation of beads. To address this issue, further optimization of electrospinning parameters, such as voltage, needle-to-collector distance, and flow rate, may be necessary to achieve a more stable electrospinning process and minimize bead formation in the cellulose acetate nanofibers containing 10% silver nitrate.

Figure30 Fiber diameter distribution of cellulose acetate nanofibers with 10% silver nitrate



Source: Personal archive

In short, when we looked at pictures (SEM figures) of different cases, such as pure cellulose acetate nanofibers and ones with different amounts of silver nitrate, we could clearly see that silver nitrate had a noticeable effect on how the nanofibers looked. These figures revealed distinct diameter distributions, with higher silver nitrate concentrations appearing to correlate with finer nanofibers. This morphological information is crucial for understanding the potential benefits of controlling nanofiber diameter in applications

such as enhanced filter properties and antibacterial performance, and it warrants further investigation and quantitative analysis for a comprehensive understanding of the observed trends.

The literature review revealed that silver nitrate is often incorporated into nanofiber matrices for its antibacterial properties (Sedlarik, Galya, Sedlarikova, Valasek, & Saha, 2010; Thangavelu, Adil, & Arshad, 2021). Our SEM results supported this trend, demonstrating alterations in nanofiber morphology with increasing silver nitrate concentration. The results also highlighted the importance of careful consideration in adjusting silver nitrate levels, as excessively high concentrations may lead to undesirable structural changes. The SEM images indicated distinctive variations in nanofiber characteristics at different silver nitrate percentages.

Moreover, the SEM analysis allowed us to identify potential areas for optimization in the electrospinning process. The literature emphasized the significance of controlling parameters such as solution viscosity and electrospinning conditions for achieving uniform nanofiber structures (Behroozi, Al-Shaeli, & Vatanpour, 2023; Yun, et al., 2010).

Our SEM findings corroborated these considerations, illustrating the impact of varying silver nitrate concentrations on nanofiber morphology. In fact, the addition of silver nitrate and changes in experimental conditions can indeed influence the diameter of the nanofibers. Several factors may contribute to variations in fiber diameter when incorporating silver nitrate into cellulose acetate nanofibers and modifying experimental parameters during electrospinning. The presence of silver nitrate can alter the solution properties, such as viscosity and conductivity. Changes in these properties can affect the stretching and elongation of the polymer solution during electrospinning, leading to variations in fiber diameter. In addition, higher concentrations of silver nitrate may result in increased solution viscosity. This change in viscosity can impact the electrospinning process, influencing the diameter of the nanofibers. The interaction between the polymer and silver nitrate molecules can also affect the solution's rheological behavior. Also, variations in electrospinning parameters, including voltage, needle-to-collector distance, and flow rate, directly influence the morphology of the nanofibers. Adjustments in these parameters, made to accommodate changes in the polymer solution, can impact the diameter and uniformity of the resulting nanofibers. Moreover, the rate at which the solvent evaporates during electrospinning is critical. Changes in silver nitrate concentration and experimental conditions can affect the solvent evaporation rate, influencing the solidification and diameter of the nanofibers.

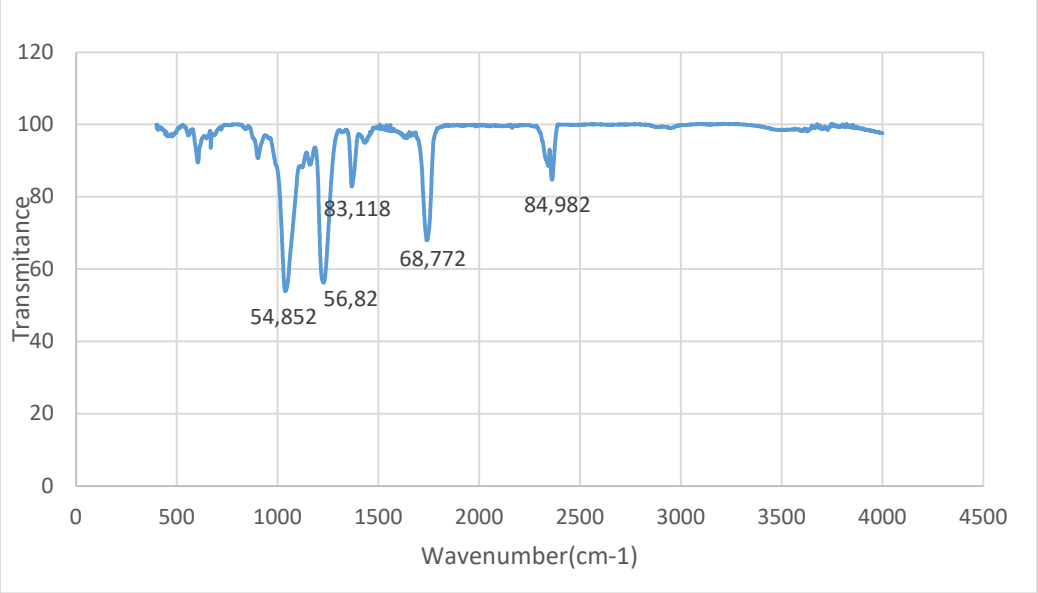
5.2. Results of Fourier Transform Infrared Spectroscopy (FT-IR)

The Fourier Transform Infrared Spectroscopy (FTIR) analysis was conducted on four distinct sample types: Cellulose Acetate Nanofibers (CA), Cellulose Acetate Nanofibers with 3% Silver Nitrate (CA-3%$AgNO_3$), Cellulose Acetate Nanofibers with 5% Silver Nitrate (CA-5%$AgNO_3$), and Cellulose Acetate Nanofibers with 10% Silver Nitrate (CA-10%$AgNO_3$).

FTIR spectra were obtained to examine the chemical composition and functional groups within each sample, providing critical insights into the impact of varying silver nitrate concentrations on the cellulose acetate nanofibers.

In Figure31, the FTIR spectrum of pure cellulose acetate nanofibers (CA) revealed characteristic peaks associated with cellulose acetate. Peaks corresponding to functional groups such as ester carbonyl (C=O) and ether (C-O-C) bonds were clearly observed, confirming the composition of the nanofibers.

Figure31 FTIR of pure cellulose acetate

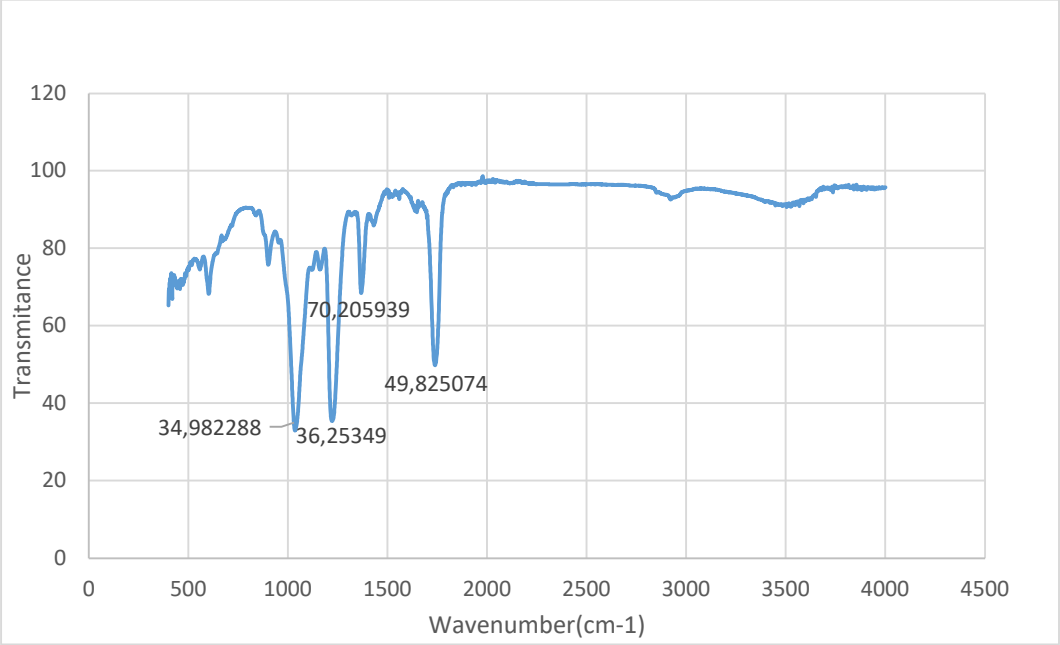


Source: Personal archive

Figure33 displays the FTIR spectrum of nanofibers prepared with 5% silver nitrate (CA-5%$AgNO_3$). Similar to the 3% silver nitrate case, Figure32, this spectrum exhibits

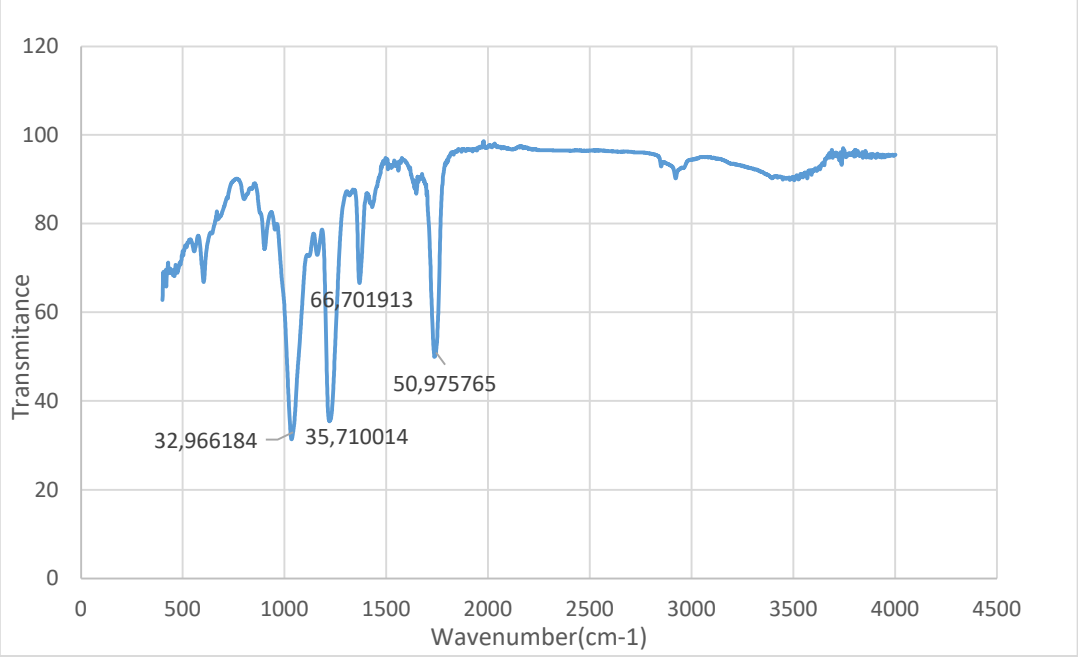
characteristic peaks of cellulose acetate alongside potential shifts or alterations indicative of silver nitrate interactions. The presence of silver nitrate at this concentration appears to have a more pronounced effect on the FTIR spectrum compared to lower concentrations.

Figure32 FTIR of cellulose acetate with 3% silver nitrate



Source: Personal archive

Figure33 FTIR of cellulose acetate with 5% silver nitrate

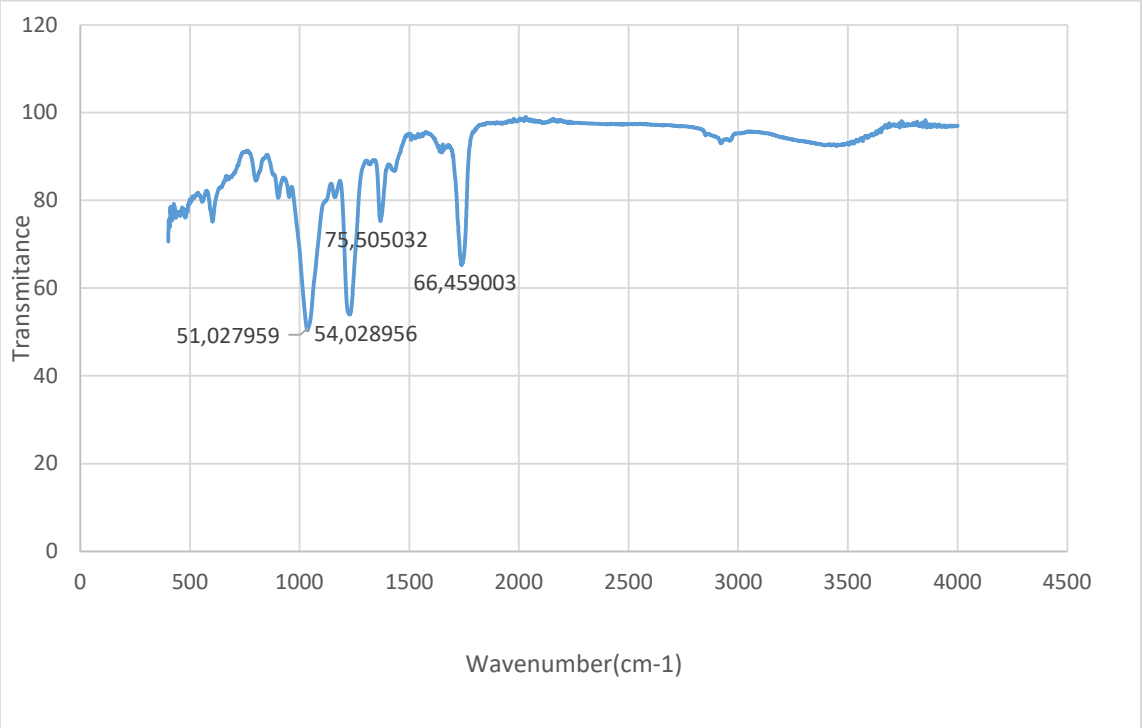


Source: Personal archive

Figure34 demonstrates the FTIR spectrum of nanofibers fabricated with the highest concentration of silver nitrate, 10% (CA-10%$AgNO_3$). In this spectrum, we observed significant changes in peak positions and intensities compared to pure cellulose acetate, indicating substantial interactions between silver ions and the polymer's functional groups.

The FTIR analysis of these samples underscores the potential chemical modifications induced by varying silver nitrate concentrations. The observed shifts in peaks and changes in peak intensities suggest complex interactions between cellulose acetate and silver nitrate. These alterations in chemical composition may have implications for the nanofibers' properties, including their antibacterial functionality. Further detailed spectral analysis, peak assignment, and chemical characterization would be essential to precisely understand the nature of these interactions and their influence on the cellulose acetate nanofibers' properties and performance in applications such as filter media.

Figure34 FTIR of cellulose acetate with 10% silver nitrate



Source: Personal archive

There are several key aspects to consider:

1. Overall Spectrum: the major peaks are shown in the plots. The spectrum typically covers a range of wavenumbers, from 400 to 4000 per cm (cm-1).
2. Functional Groups of Cellulose Acetate: Cellulose Acetate is known to exhibit characteristic peaks related to its functional groups. The key peaks associated with cellulose acetate, such as:
    1. Carbonyl stretch (C=O) around 1350-1250 cm-1
    2. O-H stretching vibration around 2500-2000 cm-1
    3. C-H stretching and bending vibrations in the range of 2000-1500 cm-1
    4. C-O stretching vibrations around 1200-1000 cm-1
3. Changes with Silver Nitrate Addition: Now, analyze the FTIR plots for the different concentrations of Silver Nitrate (3%, 5%, and 10%) added to Cellulose Acetate Nanofibers and compare them with the reference spectrum of pure Cellulose Acetate.
    1. Identify shifts in peak positions: A shift in wavenumber indicates a change in the bond strength or chemical environment.
    2. Changes in peak intensities: An increase or decrease in intensity suggests a change in the concentration or interaction of the corresponding functional group.
    3. Appearance of new peaks: These new peaks may indicate the formation of new chemical bonds or the presence of additional functional groups resulting from the interaction between cellulose acetate and silver nitrate.

5.3. Results of Filter permeability testing

The results of the filter permeability testing at different silver nitrate concentrations, specifically 1%, 3%, and 5%, reveal a nuanced relationship between silver nitrate content and permeability. The permeability values were found to be 2.02E-10 m^2, 1.05E-13 m^2, and 2.39E-12 m^2, respectively. Interestingly, an initial decrease in

permeability with the increase in silver nitrate percentage was observed, followed by a subsequent increase. It is worth mentioning that, the differences in concentrations are due to the different formulations we tested, and characterizations must have been carried out for each percentage tested. We were able to produce nanofibers at other concentrations (0%, 3%, 5% and 10%) but not enough for FTIR, permeability and efficiency analyses, so new concentrations (1%, 3% and 5%) were necessary to produce membranes for the permeability tests.

Table 6 Filter permeability results

Filters	m^2
Filter 1% Silver Nitrate	**2,02E-10**
Filter 3% Silver Nitrate	**1,05E-13**
Filter 5% Silver Nitrate	**2,39E-12**

Source: Personal archive

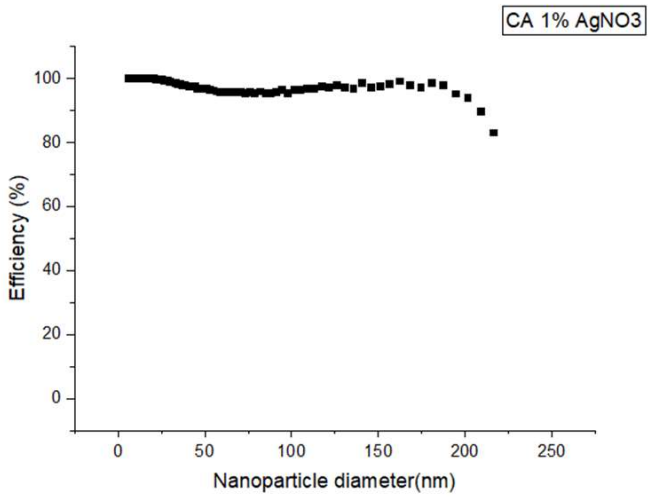
In conclusion, the permeability results provide valuable insights into the impact of silver nitrate concentration on the filter's performance. The initial decrease and subsequent increase in permeability highlight the need for a meticulous balance in nanoparticle incorporation. This delicate interplay between nanoparticle concentration and structural properties has been observed in related studies and emphasizes the importance of optimizing silver nitrate content for achieving desired filter characteristics.

5.4. Results of Filter efficiency testing

The results of the filter efficiency testing at different silver nitrate concentrations (1%, 3%, and 5%) reveal an intriguing pattern where the efficiency initially decreases and then increases with the rise in silver nitrate content. The recorded efficiency values were 98.0466%, 90.9513%, and 99.3358%, respectively.

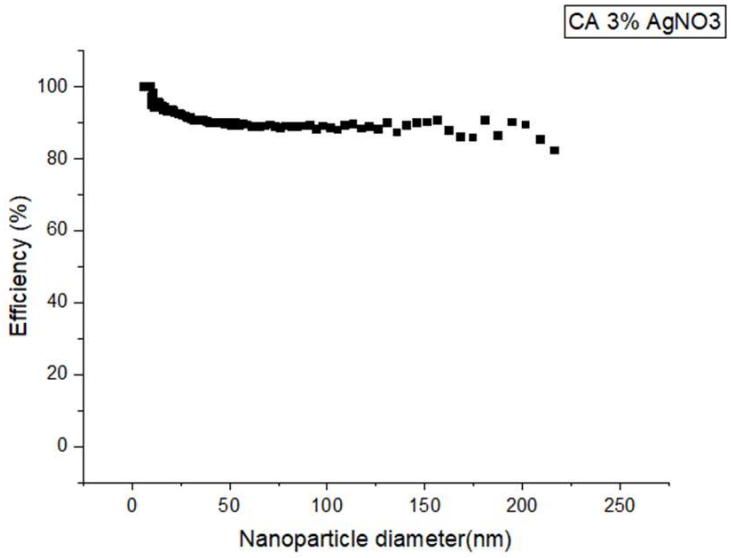
In conclusion, the efficiency results highlight the intricate interplay between silver nitrate concentration and filter performance. The initial decrease and subsequent increase in efficiency emphasize the need for precise control over silver nitrate content to achieve the desired balance between antibacterial functionality and filtration efficiency.

Figure35 Efficiency of filter with 1%Silver Nitrate



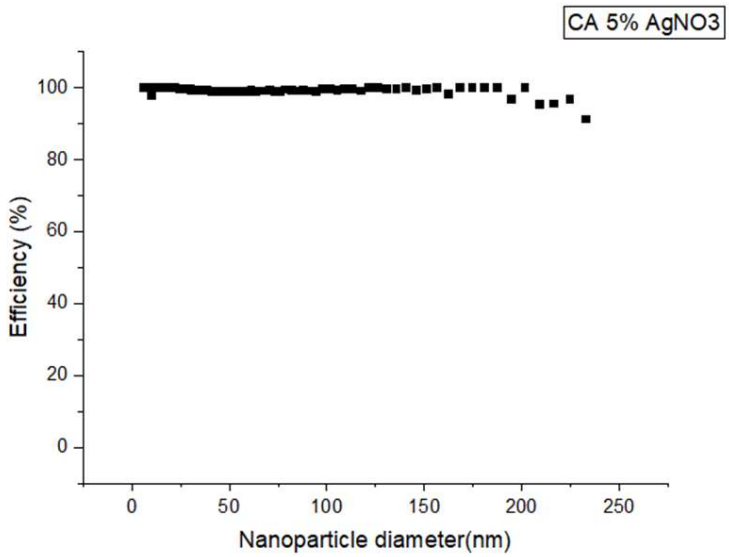
Source: Personal archive

Figure36 Efficiency of filter with 3% Silver Nitrate



Source: Personal archive

Figure37 Efficiency of filter with 5% Silver Nitrate



Source: Personal archive

Table 7 Filter global efficiency percentage

Filters	%
Filter 1% Silver Nitrate	**98,0466**
Filter 3% Silver Nitrate	**90,9513**
Filter 5% Silver Nitrate	**99,3358**

Source: Personal archive

The results show that a notable relationship between the concentration of silver nitrate (AgNO3) and the permeability of the cellulose acetate nanofiber filter media. A non-linear trend in permeability as the percentage of silver nitrate is varied.

1% to 3% Silver Nitrate Concentration: concentration of silver nitrate is increased from 1% to 3%, a clear decrease in permeability. This reduction in permeability is due a higher concentration of silver nitrate particles in the nanofiber matrix leads to the

formation of more interconnected structures, which can restrict the flow of liquids or gases through the filter media.

3% to 5% Silver Nitrate Concentration: Surprisingly, as the concentration of silver nitrate is further increased from 3% to 5%, there is a reversal in the trend, and permeability starts to increase. This increase in permeability could be attributed to various factors.

Efficiency of the filter media exhibits a similar non-linear relationship with silver nitrate concentration. As silver nitrate percentage increases, there is an initial decrease in efficiency followed by an increase.

Decrease in Efficiency (1% to 3%): might be attributed to the formation of a denser structure that hinders the flow of contaminants but also limits their interaction with the silver ions.

Increase in Efficiency (3% to 5%): higher concentration of silver nitrate particles may enhance the antibacterial properties of the filter media. This can lead to improved filtration performance, as a greater proportion of contaminants is captured and eliminated.

Complex relationship between silver nitrate concentration, permeability, and efficiency in cellulose acetate nanofiber filter media.

6. Conclusions and suggestions

The study into the impact of silver nitrate concentration on cellulose acetate nanofiber properties has yielded significant insights. Analysis of scanning electron microscopy (SEM) images demonstrated that the addition of silver nitrate at varying concentrations (3%, 5%, and 10%) indeed exerts an influence on the nanofiber diameter. Notably, initial observations indicate that higher silver nitrate concentrations tend to correlate with a reduction in nanofiber diameter in comparison to both lower concentrations and pure cellulose acetate nanofibers.

Furthermore, the investigation extended to the assessment of chemical composition and bonding modifications within the cellulose acetate nanofibers. The initial analysis of Fourier Transform Infrared (FT-IR) spectra provided valuable insights. It revealed that the addition of silver nitrate at different concentrations can induce changes in the spectra, signifying potential modifications in the chemical composition and bonding within the nanofibers.

Moreover, the filter permeability testing and filter efficiency testing provided valuable insights into the performance characteristics of cellulose acetate nanofiber filters at different silver nitrate concentrations. The numerical results from permeability testing demonstrated a nuanced trend, showcasing a decrease in permeability from 2.02E-10 m^2 (1% silver nitrate) to 1.05E-13 m^2 (3% silver nitrate) followed by an increase to 2.39E-12 m^2 (5% silver nitrate).

In parallel, the results from filter efficiency testing revealed numerical values of 98.0466% (1% silver nitrate), 90.9513% (3% silver nitrate), and 99.3358% (5% silver nitrate). The observed trend of an initial decrease in efficiency followed by an increase emphasizes the delicate balance required to optimize filter performance.

The results highlight the importance of precisely controlling the amount of silver nitrate in cellulose acetate nanofiber. The findings offer clear data on how changes in silver nitrate levels affect the structure and function of nanofiber filters.

In considering potential future projects for the ongoing research on the development of antibacterial filter media from electrospun cellulose acetate nanofibers, several key areas of exploration can be identified:

1. Optimization of Electrospinning Parameters: Investigating the effects of various electrospinning parameters (e.g., voltage, flow rate, collector distance) on the

morphology, diameter, and alignment of cellulose acetate nanofibers to optimize their antibacterial properties.

2. Incorporation of Nanoparticles: Studying the incorporation of antibacterial nanoparticles (e.g., silver nanoparticles, zinc oxide nanoparticles) into cellulose acetate nanofibers to enhance their antibacterial efficacy while maintaining their structural integrity.
3. Comprehensive Thermal Analysis: Conducting in-depth Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) experiments to understand the thermal stability and degradation behavior of the filter media under various conditions, aiding in performance optimization.

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