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# Particle Removal and Antibacterial of Nanofiber Doped with Metal and Ions Prepared by Electrospinning

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## ABSTRACT

Due to the increase in human activities and the application of nanotechnology, people's exposure to nano and submicron particles is increasing. Conventional particle separation technologies, such as electrostatic precipitators, cyclone, wet washing, and filtration method, cannot work well. In addition, the fiber diameter and pore size of the traditional fiber filter material is too large, and the bulk density is difficult to control. Nanofiber membranes have a large surface area, small pore size and high porosity. The nanofiber membranes prepared by electrospinning technology are easy to intercept submicron and nanoparticles. Furthermore, the electrospinning technology is simple to operate, makes fibers of various materials easy, and is convenient for assembly and replacement. The most common bacteria are Escherichia coli, which harms the ecological environment and human health. Therefore, this study used bacteria and particles as the target pollutants and controlled by prepared nanostructured materials doped with metals and ions. In order to improve the treatment efficiency of particles and bacteria, this study added various metals and ions to nylon 6 nanofibers and explored the antibacterial and filterability of artificial fibrils. Different operation parameters, such as types of metal, types of ion, metal and concentration, and surface velocity, were also investigated to prepare various fibers to make the best performance fiber. In addition, the best-operating conditions could be obtained through a filtration test. Different salts, metals and metal ions with different concentrations, three metal oxides ($TiO_2$, $CeO_2$, and ZnO) and three ions ($Ag^+$, $K^+$, and $Na^+$) were used to test the filtration performance of various particle sizes for the best metal ion concentration, best filtration and bacterial removal performance. The experimental results show that the filtration efficiency of the composite fiber can reach 99%, and the composite fiber sprayed with a self-made antibacterial liquid has the best antibacterial ability.

**Keywords:** Electrospinning, Metal ion, Nylon 6, PM removal, Antibacterial

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## 1 INTRODUCTION

The vigorous development of modern industry has driven the economic and social living standards but also caused air pollution and endangered human health. In recent years, people's requirements for environmental awareness have gradually increased. Therefore, technology development and environmental impact reduction will become an issue worthy of in-depth discussion (Lin, 2018). Air pollution can cause stroke, lung cancer, chronic obstructive pulmonary disease, and cardiovascular disease (Yoon *et al.*, 2008; Yildiz and Bradford, 2013; Wang and Pan, 2015; Kim *et al.*, 2020; Zhao *et al.*, 2020). In addition to its profound impact on human health, particulate pollutants can also affect the living environment, affecting atmospheric visibility (Lv *et al.*, 2019; Bardouki *et al.*, 2003; Horton *et al.*, 2014). In addition, bioaerosols, including airborne viruses, bacteria and pollen, are widely present in the air and can cause various diseases, including allergies, when inhaled (Victor *et al.*, 2021). Most bacteria come from human and animal activities. The most common bacteria are Escherichia coli, which harm the ecological environment and human health. Therefore, this study uses bacteria and particles as the target pollutants.

It is crucial to make electrospinning filter materials with high performance, and good particle filtering effect since many fine particles in life are hazardous to the human body. The diameter of the fibers constituting the filter can be divided into traditional fiber filter and nanofiber. Traditional fiber filter materials (such as glass fiber, meltblown fiber and spunbond fiber) have too large fiber size and pore size diameter, and the bulk density is difficult to control (Hung *et al.*, 2011). Electrospinning technology has a simple operation method and can prepare ultra-fine fibers, which have the advantages of large surface area, small pore size, and high porosity (Xu *et al.*, 2016; Liu *et al.*, 2015a). It can effectively solve the filtration problem of ultra-fine particles (Chen, 2021; Wang *et al.*, 2018; Zhang *et al.*, 2017). In addition, in this study metal nanoparticles and ions added to nylon 6 fiber to enhance its filtration and antibacterial performance and promoted the filterability of nylon 6 membrane, N6 (Livraghi *et al.*, 2010; Horie and Iwahashi, 2014), to fine particles (particle size less than 1 µm). This study aims to prepare fibers containing metal ions by electrospinning and explore their effect on fine particles.

## 2 MATERIALS AND METHODS

### 2.1 Experimental Design

The experimental methods include the preparation of electrospun fiber materials, analysis of fiber materials characteristics, measurement of surface voltage, measurement of penetration ratio, calculation of single fiber efficiency and filtration quality. The test conditions are shown in Table 1.

### 2.2 Fiber Material Selection

N6 used in this study was purchased from Sigma Aldrich company in the United States. The chemical is of analytical grade and does not need to be purified. Due to its low melting point and accessible price, the mechanical properties of N6 can be improved by adding various modifiers. The use of N6 can achieve a translucent effect. It also has high mechanical strength, high tensile and compressive strength, excellent wear resistance, good electrical insulation, and short solidification time, although it has the disadvantages of moisture adsorption and no temperature resistance.

### 2.3 Selection of Spinning Solvent

The solvent used in this study was analytical grade formic acid ($CH_2O_2$), purchased from Acros Company in the United States, and no further purification was required. For optimal filtration quality, the diameter of fibers produced by electrospinning is significant. The fiber diameter varies with the polymer/solvent solution used for electrospinning. Three main solution properties may drive this difference in fiber diameter, including solution viscosity, solvent permittivity, and solution conductivity. Higher viscosity solutions generally produce thicker fibers, while higher conductivity and solvent permittivity result in smaller fiber diameters. In order to make a filter membrane with a smaller fiber diameter, formic acid was used as the spinning solvent because of its higher electrical conductivity and higher solvent dielectric constant.

**Table 1.** Experimental parameter table.

| Parameter | Illustration |
|---|---|
| Type of metal oxide | $TiO_2$, ZnO, $CeO_2$ |
| Types of ions | $Ag^+$, $K^+$, and $Na^+$ |
| Metal and ion content (%) | 0.5, 1.0, 2.0, 4.0 |
| Voltage (KV) | 20, 25, 30, 35 |
| Speed (rpm) | 50, 100, 150, 200, 250 rpm |
| Spinning tine (hr) | 0.5, 1.0, 2.0 |

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## 2.4 Preparation of Composite Materials

In this study, spinning polymer solutions and random fiber gaskets containing metal ions have been prepared. Polyethylene terephthalate (PET) and N6 spunbond were placed on the drum collector as the substrate, and the fibers produced by electrospinning were directly spun on the PET substrate to prepare fibers with sufficient mechanical strength and facilitate the test in the experiment before being placed in the drying box for storage. During the filtration test, a layer of PET substrate is superimposed to form a sandwich structure. The schematic diagram of its composite material is shown in Fig. 1.

## 2.5 Composite Material Preparation

In this study, 20 wt% N6 and formic acid were prepared. After the magnet was stirred for 6 hours, different concentrations of metals and ions were added. After the magnet was stirred and mixed evenly, the spinning precursor solution was completed. In addition, PET was used as the substrate, the voltage was controlled at 20–35 kV, the drum collector was set at 50–250 rpm, the feeding rate was 0.1 mL $hr^{-1}$, and the distance from the metal needle to the collector was 15 cm to prepare nano fibers. Finally, the composite fiber was sprayed with antibacterial liquid to complete the composite material preparation. During the filtration test, a layer of PET substrate was superimposed to form a sandwich structure. Electrospinning setup with modified nanofibers is shown in Fig. 2.

## 2.6 Filter Performance Test System

The filter membrane was fixed with a stainless steel clip, and then the particles produced by the particle generation system were introduced. The wide range particle size spectrometer (WPS, 1000xp, MSP Inc.) was used to measure the particle number concentration in the range of

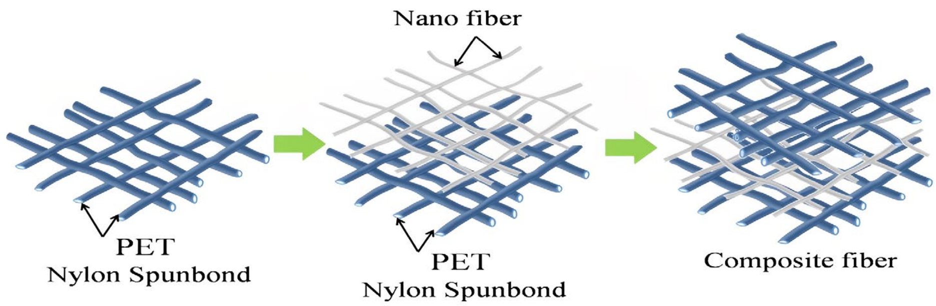


**Fig. 1.** Schematic diagram of composite material structure.

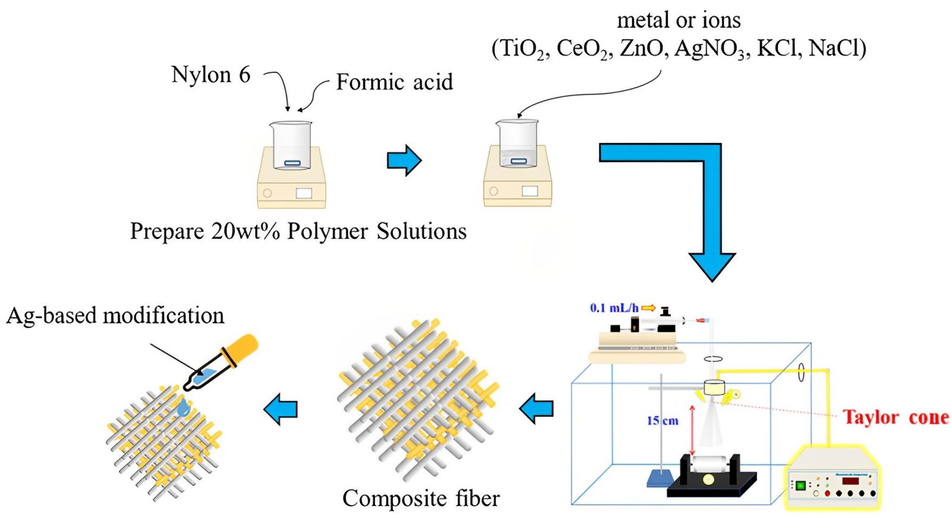


**Fig. 2.** Electrospinning setup with modified nanofibers.

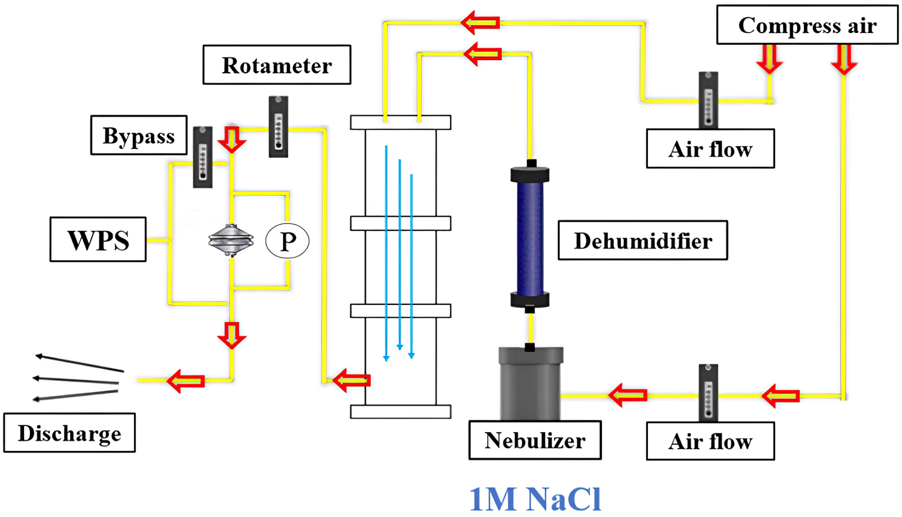


**Fig. 3.** Filtration performance homemade test set-up.

10–500 nm before and after filtration. At the same time, the pressure drop of the filter membrane was measured by using a differential pressure gauge connected to the inlet and outlet of airflow in test tunnel (Fig. 3). The filter membrane area is about 16 $cm^2$. Under the same experimental conditions, each filter membrane is subjected to three repeated filtration tests, and each test will be cycled for three times. Finally, the average values and corresponding standard deviations are taken to represent the filtration performance of samples.

## 2.7 Calculation of Filtration Quality

The filtration quality parameter can directly reflect the filtration performance of the filter membrane. The calculation formula of penetration ratio is shown in Eq. (1). In addition to the penetration ratio, the air resistance of the filter membrane, which is the pressure loss, must also be considered. Reducing the penetration ratio can easily lead to a sudden increase in pressure loss, and affect the increase in energy loss. The filter quality (Quality factor, $Q_f$) is used as the performance index of the filter material, which can more appropriately evaluate the quality of the filter material, as shown in Eq. (2), with considering the penetration ratio and pressure drop at the same time.

$$P = \frac{C_{out}}{C_{in}} \times 100\% \tag{1}$$

$P$: penetration ratio (%)
$C_{out}$: particle number concentration in exhaust gas
$C_{in}$: particle number concentration in intake air

$$Q_f = \frac{-\ln(P)}{\Delta P} \tag{2}$$

$Q_f$: Filtration quality of filter material
$\Delta p$: Pressure drop of filter membrane

## 2.8 Antibacterial Zone Test

The antibacterial zone test method is to assess the drug diffused in the agar plate to inhibit the growth of the surrounding bacteria and form a transparent circle. The bacteriostatic zone method has the advantages of convenient operation, low cost, accurate and reliable results, and is widely used (Liu *et al.*, 2015b). Activation of bacteria and preparation of bacterial solution: inoculate the frozen bacteria to the plate medium by scribing, and culture at 37°C for 24 hours. Select single colony and inoculate it to 100 mL liquid culture medium, culture it overnight in a shaking table at 37°C at 200 r min$^{-1}$, and reserve the bacterial solution. This study adopts the coating plate method. First pour about 20 mL of plate culture medium into the sterilized plate, set it horizontally, inoculate 0.1 mL of bacterial solution, and use it for standby after uniform coating. When the content of bacteriostatic agent is constant, the size of bacteriostatic circle has a great relationship with the cell concentration. If the cell concentration is too high, it will antagonize the effect of bacteriostatic agent, resulting in a small bacteriostatic zone.

# 3 RESULTS AND DISCUSSION

## 3.1 Characteristic Analysis Results

### 3.1.1 SEM image

In this study, the morphology and average diameter of fibers spun from N6-0.5% $AgNO_3$, 0.5% ZnO and 0.5% $TiO_2$ were observed by SEM. The tested fibers were spun at 35 kV and 100rpm. The surface of the three kinds of metal ion-loaded fibers was smooth and flat. Metal nanoparticles could not be clearly observed in the SEM images since the diameter of the metal ions was too small and was covered by the fibers (Juuti *et al.*, 2019). Figs. 4(a) and 4(b) show that 0.5% $AgNO_3$ and 0.5% ZnO produce fibers with smaller pores. The average fiber diameter of the filter material is 134 and 203 nm, respectively. The reason for the smallest diameter of the fibers containing $AgNO_3$ may be attributed to the higher conductivity of its spinning precursor solution (Canalli Bortolassi *et al.*, 2019). From Fig. 4(c), it can be seen that the fibers of 0.5% $TiO_2$ are few and not dense, and the average fiber diameter of the filter material is 157 nm. In addition, the average diameter of the fibers spun with 0.5% NaCl and KCl is about 200 nm.

### 3.1.2 Elemental mapping image

In this study, EDS mapping analysis was carried out to ensure that metals and metal ions were successfully mixed with N6. The tested fibers were spun with fibers containing 0.5 wt% metal ions at 35 kV and 100 rpm. As shown in Fig. 5, it can be seen that Ag, Zn and Ti elements can be detected on the filter materials spun with N6-0.5 wt% $AgNO_3$, N6-0.5 wt% ZnO and N6-0.5 wt% $TiO_2$, which confirm that the metal ions are indeed successfully mixed with the spinning solution.

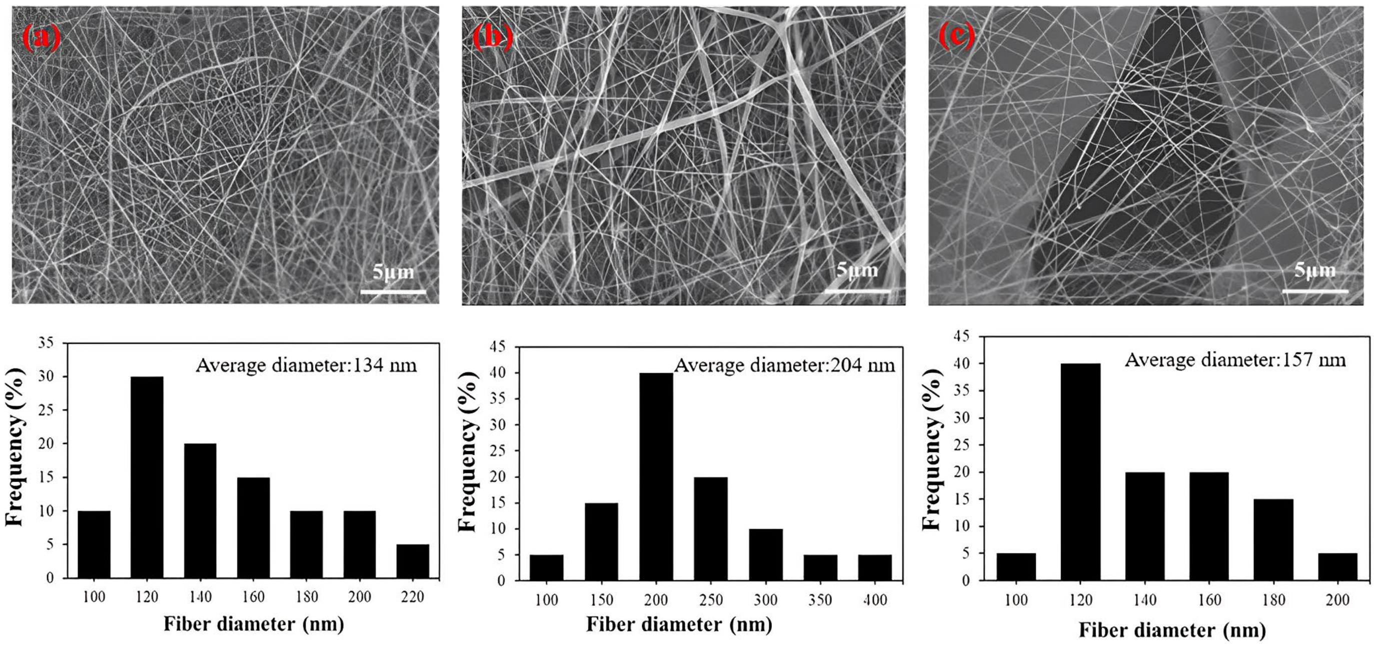


**Fig. 4.** SEM image (a) N6-0.5%$AgNO_3$, (b) N6-0.5%ZnO, and (c) N6-0.5%$TiO_2$.

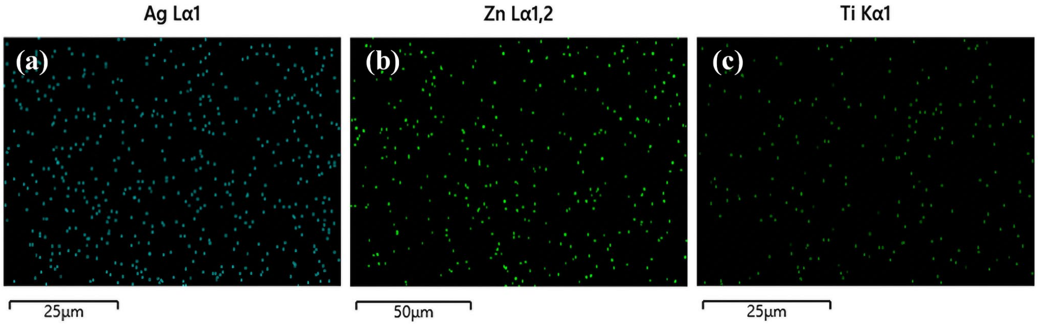


**Fig. 5.** EDS mapping images (a) Ag distribution of 0.5 wt% $AgNO_3$, (b) Zn distribution of 0.5 wt% ZnO, and (c) Ti distribution of 0.5 wt% $TiO_2$.

## 3.2 Performance Evaluation

In this study, the effects of different parameters on the filtration quality of electrospinning were carried out by using different metal ions, concentrations of metal ions, different voltages, collector speed and spinning time. Then, the fibers prepared by each parameter were used for filtration tests, and the penetration ratio, pressure loss and filtration quality of fiber filter materials were calculated.

### 3.2.1 Filter performance test at different voltage and speed

In order to evaluate the influence of different voltage and rotation speed on the filtration performance of electrospun filter materials, the penetration rate of filter materials prepared at different voltage (20, 25, 30, and 35 kV) and different rotation speed (50, 100, 150, 200, and 250 rpm) was tested. It was found that the average filtration quality of the filter material with a rotation speed of 100 rpm was the best. Fig. 6(a) shows the test results of the penetration ratio of filter materials prepared at different voltages. It can be seen that the particle penetration rate is low, and the fiber filtration quality is improved due to the enhanced electrostatic repulsion force and the finer fiber diameter. When the voltage is 20 kV, it can be observed that the filtration efficiency of the fiber is poor for particles with small particle size, which may be caused by the excessive pore size of the fiber, causing small particles to penetrate the fiber. The Filtration efficiency can reach nearly 100% when the voltage is 35 kV and rotation speed is 100 rpm with filter quality of 0.109 $Pa^{-1}$, as shown in Fig. 6(b). The main reason is that the fibers on the collector are relatively evenly distributed at this rotational speed, the filtering effect increases and the pressure loss of the filter material is relatively small.

### 3.2.2 Filtration performance test of different metal oxides and ions

In this study, 0.5 wt% of metal ions was used to evaluate the effect of the filter material on the penetration ratio and filtration efficiency. Electrospinning was performed using the optimized parameters of the experiment at voltage of 35 kV and rotation speed of 100 rpm. From Fig. 7, it can be seen that the average penetration ratio of KCl fibers is the highest (up to 5.8%) due to larger fiber diameter with adding KCl. In contrast, the average penetration ratio of $AgNO_3$ fibers is the lowest (only 0.2%) since the diameter of $AgNO_3$ fibers is relatively small (only about 134 nm), and the pores of the formed fibers are small.

### 3.2.3 Filtration performance test for different metal ion concentrations

The nano fiber membrane filtration efficiency (up to 99.8%) added with $AgNO_3$ is higher than the other fibers added with NaCl and KCl. As shown in Fig. 8(a), the average penetration ratio of fibers with 0.5, 1.0, 1.5, and 2.0wt% $AgNO_3$ added were 0.2, 1.0, 0.5, and 3.0%, respectively. The fiber containing 0.5 wt% $AgNO_3$ had the lowest penetration ratio since appropriate amount of $AgNO_3$ can make it spin out more uniform fibers. In addition, it may cause the solution viscosity to be too high when the concentration of $AgNO_3$ is high, which affects the formation of spinning fibers. It can be seen from Fig. 8(b) that the fiber with the highest filtration quality is the fiber added with 0.5 wt% $AgNO_3$ (about 0.082 $Pa^{-1}$). It means it can improve the uniformity of the fiber with adding appropriate amount of $AgNO_3$ for reduction of fiber pressure loss.

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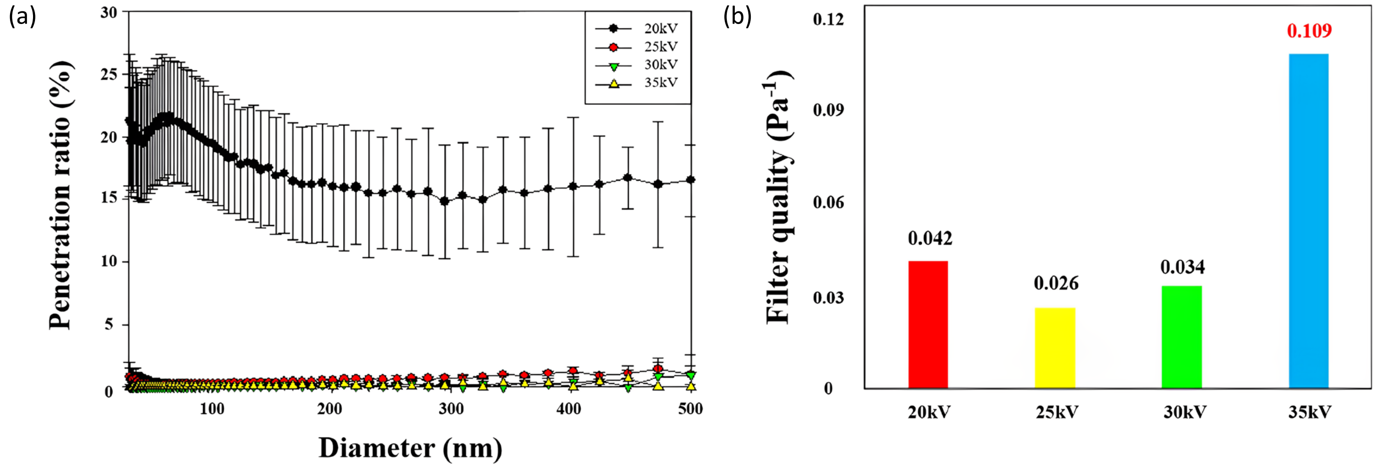


**Fig. 6.** Performance of fiber (a) penetration ratio and (b) filtration quality of filter material spun with different voltages @100 rpm.

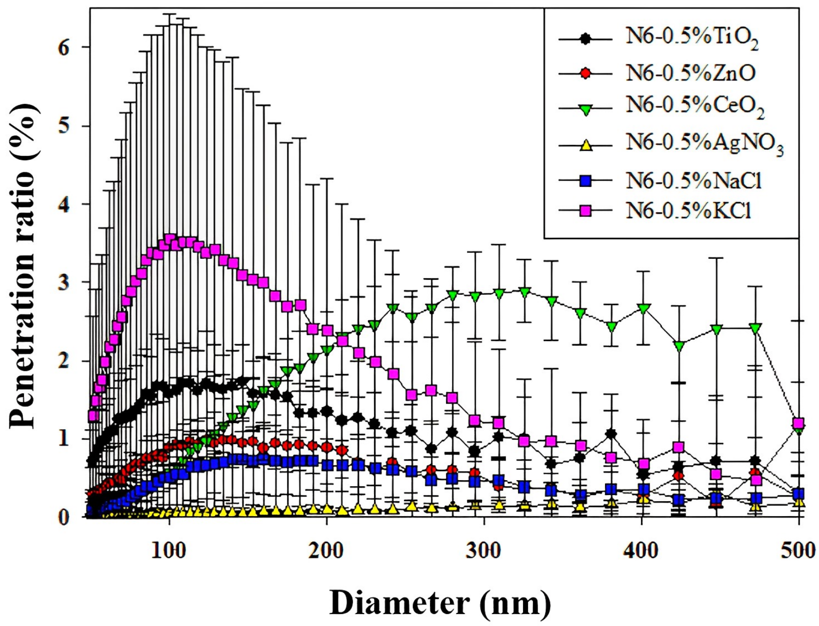


**Fig. 7.** Penetration ratio of different metal oxides and metal ions.

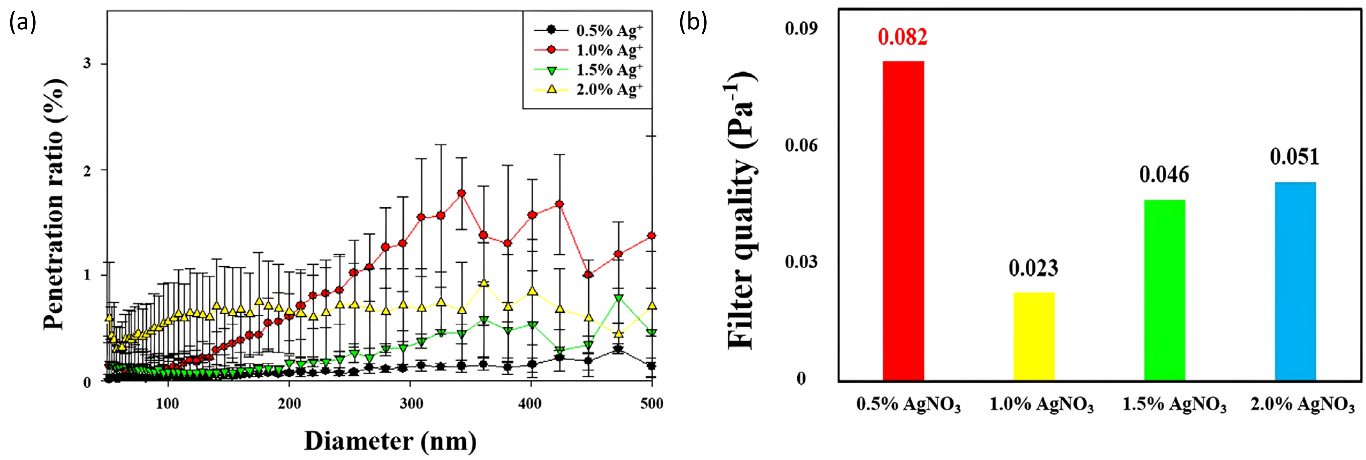


**Fig. 8.** The effect of addition $AgNO_3$ (a) penetration ratio and (b) filtration quality.

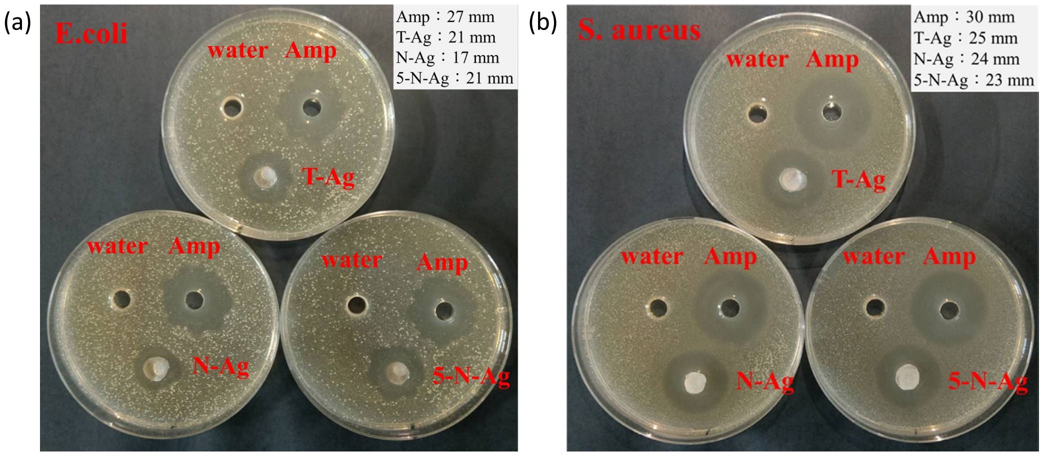


**Fig. 9.** Effects of different silver ions on antibacterial performance (a) *E. coli* and (b) *S. aureus* bacteria.

### 3.2.4 Antibacterial effect test

In this study, fibers containing 0.5 wt% $AgNO_3$ were prepared under optimal operating parameters (voltage of 35 kV, rotation speed of 100 rpm), and the fibers were sprayed with different silver solutions for antibacterial inhibition zone test. The fibers sprayed with different silver solutions have inhibitory effects on Escherichia coli and Staphylococcus aureus as shown in Fig. 9. The control group is penicillin (Amp), and the fibers sprayed with homemade triangular nanosilver solution (T-Ag). The antibacterial effect of the fiber is second after comparing with the results of Amp, and the size of the inhibition zone is 21 and 25 mm, respectively. In addition, the antibacterial effect is higher than that of the nanosilver solution with five times silver content (Zhu *et al.*, 2019). The antibacterial activity mechanism of fibers sprayed with silver containing solution is dominated from the binding of AgNPs to negatively charged bacterial cell walls and resulting in structural changes that degrade bacterial cell walls for inhibiting bacterial growth (Victor *et al.*, 2021).

## 4 CONCLUSIONS

In this study, electrospinning for preparing fibers was performed using solutions containing different metals and ions under different operating parameters. The optimal operating parameters were voltage of 35 kV and rotation speed of 100 rpm for preparing nanometer fibers with good filtration and antibacterial properties. Both the SEM and EDS mappings confirm that the ions are successfully mixed with the electrospun fibers uniformly. In addition, the filtration efficiency increases with the increase of the voltage because the fiber diameter is decreased with increasing voltage. The submicro and nano particle removal efficiency can be obtained as 99.8% with 0.5 wt% $AgNO_3$ sprayed with triangle silver for higher filtration quality of 0.082 $Pa^{-1}$ and lower pressure drop. Furthermore, the filter material sprayed with T-Ag has the best antibacterial performance for *E. coli* and *S. aureus* growth inhibition from the inhibition zone test.

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## CREDIT AUTHOR STATEMENT

The synthesis of the material that is utilized in this research was performed by Y.C. Jiang and Y.C. Cheng. The different filtration test with respect to the different parameters in probation was conducted by Y.C. Jiang. Characterization experiments done to the nanofilter was performed by Y.C. Cheng. Conceptualization of the research was aided by C.T. Chang and A.R. Caparanga. Data analysis and manuscript write up was accomplished by C.T. Chang and R.H.T. Cayron. Moreover, this research was made possible throuh the cooperative works of all the authors.

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