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Research article

Fabrication of Composite Carbon Nanofibers with Silver Particles for High-quality Membranes for Antimicrobial Water Filtration

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Abstract

In this study, carbon nanofiber (CNF) composites containing silver particles at varying silver-to-carbon ratios of 0%, 10%, 20%, and 40% (denoted as CNF, CNF@Ag-10, CNF@Ag-20, and CNF@Ag-40, respectively) were fabricated using the electrospinning technique. Polyacrylonitrile (PAN) solutions, with silver nitrate ($AgNO_3$) as a precursor at concentrations of 10%, 20%, and 40%, were dissolved in dimethylformamide (DMF) to produce the fibers. These fibers were subsequently calcined to form carbon nanocomposites embedded with silver nanoparticles. The resulting fibers, with average diameters ranging from 527 to 750 nm, incorporated silver nanoparticles measuring between 6 and 35 nm. The membranes derived from these fibers exhibited filtration rates of 7.9 to 14.3 cm^3/min and effectively inhibited *Escherichia coli*, *Salmonella*, and *Enterobacter*, achieving microbial reductions of 59.46% to 98.23%. The antimicrobial performance of the CNF@Ag composites was found to increase with higher silver doping concentration.

Keywords: carbon nanocomposite fibers; electrospinning; membrane filtration; microbial inhibition

1. Introduction

Water is an essential resource for sustaining life, especially when it comes to safe drinking water that must be free from contaminants, including heavy metals and pathogenic microorganisms (Hasan et al., 2020). According to the World Health Organization (WHO), approximately 780 million people globally lack access to clean drinking water, a problem exacerbated by rapid population growth and increasing water demand (Pal et al., 2018). The imbalance between water supply and demand due to industrial growth and population increase has led to a looming water crisis. The growing demand for clean water, compounded by industrial pollution and population growth, necessitates the exploration of

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alternative water sources and more effective purification technologies. Membrane filtration has emerged as one of the most promising methods for water treatment and is particularly important in desalination and wastewater recycling (Subramani & Jacangelo, 2015; Yusuf et al., 2020). However, biofouling caused by the accumulation of microorganisms on membrane surfaces remains a significant challenge, increasing operational costs and reducing efficiency (Flemming, 2011). Therefore, developing membranes that can resist biofouling while maintaining high water flux and contaminant removal is imperative.

Electrospinning is a versatile and widely used technique to fabricate nanofibers with diameters ranging from tens of nanometers to a few micrometers. It involves applying a high-voltage electric field to a polymer solution or melt, which causes a charged jet to be ejected from a needle (Bhardwaj & Kundu, 2010). As the jet travels through the electric field, it elongates, and the solvent evaporates (in the case of polymer solutions), leaving behind solid nanofibers that are collected on a grounded substrate. Electrospun nanofibers possess a wide range of applications due to their unique properties, including a high surface-area-to-volume ratio, tunable pore sizes, and excellent structural versatility (Ding et al., 2018). These characteristics make them highly effective in various fields. In water filtration and desalination, electrospun membranes serve as ideal candidates for removing contaminants, offering enhanced filtration efficiency through customizable pore structures (Sanaeepur et al., 2022). Similarly, in tissue engineering, the nanofibers mimic the extracellular matrix, providing scaffolds that promote cell growth and tissue regeneration. In drug delivery systems, the high porosity and large surface area of electrospun nanofibers enable controlled and sustained release of drugs, improving therapeutic outcomes (Shahriar et al., 2019). Additionally, electrospun fibers are highly responsive to environmental changes, making them valuable in sensor applications, where their sensitivity can be harnessed to detect gases, chemicals, or biological markers. The versatility of electrospun nanofibers underscores their potential to revolutionize a variety of industries, from water treatment to biomedicine and environmental sensing (Kenry & Lim, 2017).

Recent advances in nanotechnology have opened new avenues in water treatment, particularly through the use of carbon nanofibers (CNFs). CNFs exhibit unique electrical, thermal, and mechanical properties, making them highly effective for the removal of contaminants such as heavy metals, organic compounds, and microorganisms (Sayyed et al., 2021). Furthermore, the incorporation of silver nanoparticles (AgNPs) into CNF membranes offers a powerful antimicrobial capability (He et al., 2022). Silver is a well-known antibacterial agent, effective in disrupting cellular processes and inhibiting biofilm formation (Markowska et al., 2013).

In this study, we developed a novel anti-microbial membrane for desalination pretreatment by adding silver nanoparticles. The membrane was synthesized without the use of binders, relying on silver particles as the binding agent for the CNFs. By adjusting the silver content (0%, 10%, 20%, and 40% by weight), we examined how the silver level affected the membrane's water permeate flux, porosity, and antibacterial properties, particularly its effectiveness against *Escherichia coli, Salmonella,* and *Enterobacter*. Our findings indicate that the membranes with silver loading demonstrated superior performance, removing 98.23% of the tested bacteria. This novel membrane holds great potential for real-world applications in water desalination and treatment, offering a robust solution to the persistent problem of biofouling.

2. Materials and Methods

2.1 Materials

Nanofibrous membranes were fabricated using the electrospinning technique. Polyacrylonitrile (PAN) was used as the polymer precursor, and silver nitrate ($AgNO_3$) was added to produce silver nanoparticle-embedded carbon nanofibers. The following stages describe the membrane preparation procedure and the raw materials used in this study. Carbon nanofibers (CNFs) used in this study were obtained polyacrylonitrile (PAN), Mw 150,000, Sigma-Aldrich, USA. Silver nitrate ($AgNO_3$, reagent grade, ≥99% purity) was purchased from Sigma Aldrich and used as the silver source. N, N-Dimethylformamide (DMF) (Sigma-Aldrich, USA) was used as the solvent for CNF impregnation.

2.2 Preparation of carbon nanofiber composite (CNF@Ag) membranes

PAN (4 g) was dissolved in 40 mL of N, N-Dimethylformamide (DMF) by stirring continuously on a hot plate at 50°C for 2 h until a homogeneous polymer solution was obtained. For the silver-incorporated samples, silver nitrate ($AgNO_3$) was dissolved separately in 5 mL of DMF, and the solution was gradually added to the PAN-DMF mixture at different concentrations (0%, 10%, 20%, and 40% w/w of silver to PAN) under continuous stirring for 4 h. The prepared solutions were loaded into a 10 mL syringe equipped with a stainless-steel needle (22 gauge). Electrospinning was carried out under a voltage of 13 kV with a needle-to-collector distance of 18 cm. The solution flow rate was maintained at 0.60 mL/h using a syringe pump. The nanofibers were collected on a rotating aluminum drum wrapped in foil to form non-woven fibrous mats. The collected fibers were subjected to calcination in a tube furnace to convert the PAN into carbon-based nanofibers and to embed the silver nanoparticles within the fiber structure. Initially, the fibers were stabilized at 100°C for 2 h in an argon atmosphere, which was followed by carbonization at 500°C for 2 h, and finally at 900°C for 2 h in a CO_2 environment. The CO_2 environment was introduced to enhance porosity by creating nanopores in the carbon structure. The resulting carbon-silver nanocomposite fibers were denoted as CNF@Ag-X (where X refers to the silver concentration of 0, 10, 20, or 40%).

2.3 Characterization techniques

The surface morphology of the CNF and CNF@Ag fibers was examined using scanning electron microscopy (SEM, JEOL JSM 7800F). Transmission electron microscopy (TEM, FEI/TECNAI G2 20 S) was used to observe the internal structure of the fibers and the distribution of silver nanoparticles within the carbon matrix. X-ray diffraction (XRD, Bruker D2 X-ray diffractometer) analysis was employed to determine the crystalline structure of the carbon nanofibers and silver nanoparticles. Raman spectroscopy (RAMAN, JOBIN YVON HORIBA T64000) was used to investigate the structural characteristics of the carbon fibers. The specific surface area and porosity of samples were analyzed using Brunauer–Emmett–Teller (BET, BELSOFP-miniII, JAPAN) surface area analysis. The antimicrobial properties of the membranes were assessed by testing their ability to inhibit the growth of common bacterial strains, including *Escherichia coli*, *Salmonella*, and *Enterobacter*.

Table 1. Starting materials for preparing solutions in each case

Solution	Pan (g)	$Ag(NO_3)$ (g)	DMF (ml)
PAN	4.00	-	40.00
PAN@Ag-10	4.00	0.40	40.00
PAN@Ag-20	4.00	0.80	40.00
PAN@Ag-40	4.00	1.60	40.00

3. Results and Discussion

The physical, structural, and functional properties of the synthesized carbon nanofibers (CNF) and carbon-silver nanocomposite fibers (CNF@Ag) were characterized using a variety of techniques to ensure their suitability for membrane filtration and antimicrobial applications.

3.1 Characterization of CNF and CNFs@Ag

3.1.1 Morphological analysis (SEM and TEM)

The surface morphology and fiber diameter of the CNF and CNF@Ag samples were analyzed using SEM as shown in Figure 1. The SEM images revealed a uniform distribution of fibers with diameters ranging from 527 to 750 nm. Silver nanoparticles, with sizes between 6 to 35 nm, were uniformly embedded within the fibers, as confirmed by high-magnification SEM images. TEM was used to further investigate the internal structure of the nanofibers and the dispersion of silver nanoparticles within the carbon matrix. In Figure 2, the TEM images show the precise embedding of silver nanoparticles throughout the fibers, confirming the uniformity and stability of the composite structure.

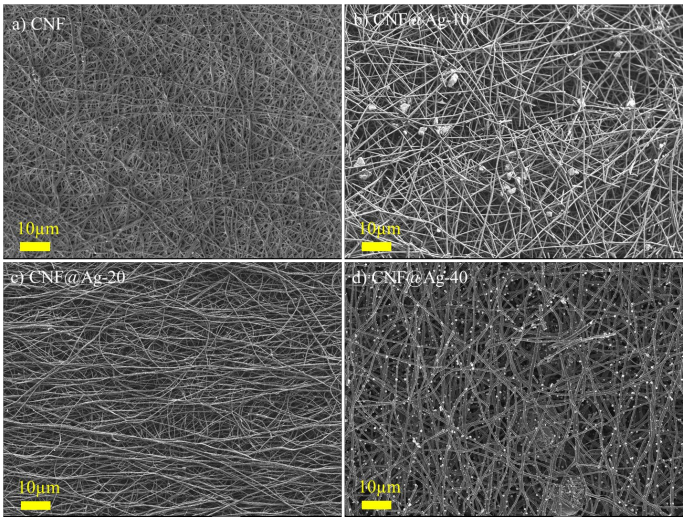

Figure 1. Scanning electron microscope images of metal oxide carbon nanocomposites fabricated by electrospinning technique using different concentrations (a) CNF, b) CNF@Ag-10, c) CNF@Ag-20, and d) CNF@Ag-40

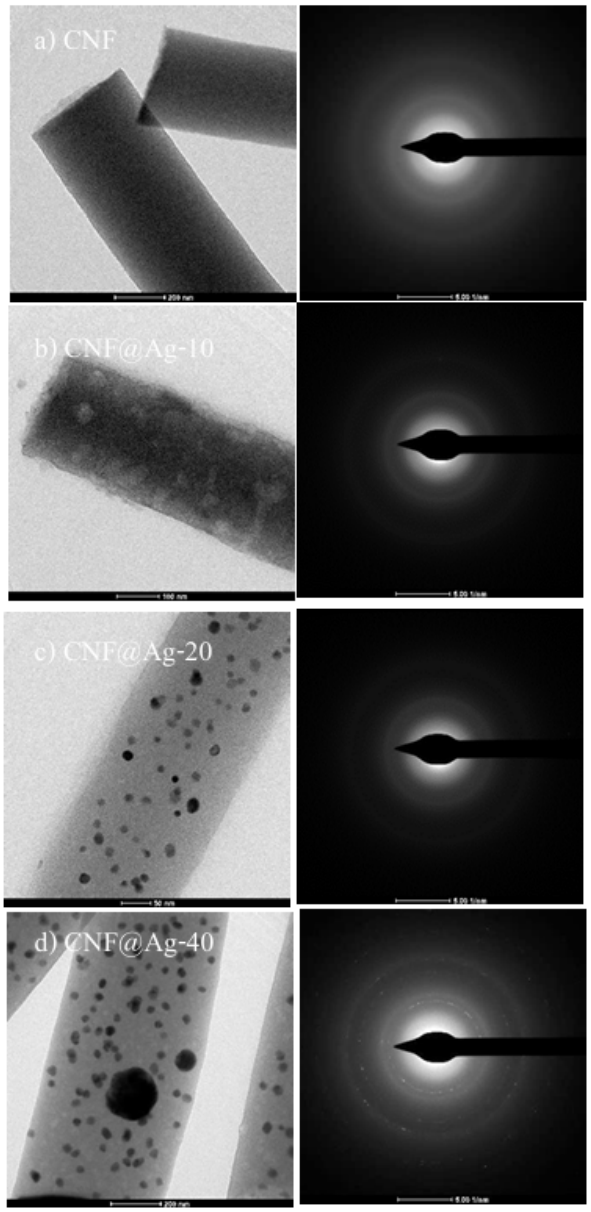

Figure 2. Transmission electron microscope images of metal oxide carbon nanocomposites containing different conditions of a) CNF, b) CNF@Ag-10, c) CNF@Ag-20, and d) CNF@Ag-40

3.1.2 Crystalline structure (XRD)

X-ray diffraction analysis was performed to determine the crystalline phases present in the CNF and CNF@Ag samples. The XRD patterns confirmed the successful incorporation of silver nanoparticles, with the characteristic peaks of silver (Ag) and graphitic carbon

observed. The presence of graphitic carbon indicated successful carbonization of the polyacrylonitrile precursor as shown in Figure 3 (Liu et al., 2019). XRD analysis was employed to investigate the crystalline structure of the carbon and silver-embedded carbon nanocomposites (Niu et al., 2010). This technique helped identify the phase composition and crystalline characteristics of the samples. XRD measurements were conducted in the 2θ range of 10° to 90° using a Cu Kα X-ray diffractometer (λ = 1.5406 Å) to record the intensity and diffraction angles. The XRD patterns of both the CNF and CNF@Ag samples displayed peaks indicating the presence of graphitic structures, with a significant peak observed at 26.5°, corresponding to the (002) plane of graphitic carbon. For the CNF@Ag samples, additional peaks at 38.2°, 44.4°, and 64.5° were detected, confirming the presence of silver nanoparticles in a crystalline form (Jemal et al., 2017; Destyorini et al., 2021). The XRD analysis confirmed that the CNF@Ag samples maintained a high-quality graphitic structure while also demonstrating the successful incorporation of silver nanoparticles. This combination of structural integrity and nanoparticle distribution is crucial for enhancing the filtration and antimicrobial properties of the developed membranes.

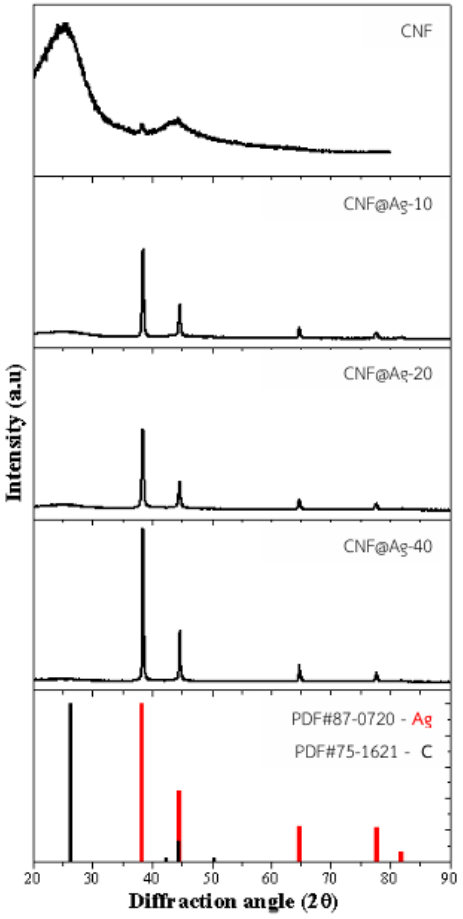

Figure 3. X-ray diffraction patterns of samples at different compositions and concentrations

3.1.3 Surface chemistry by Raman spectroscopy

Raman spectroscopy was used to assess the degree of graphitization in the carbon fibers (Zou et al., 2003) and the results are shown in Figure 4. The intensity ratio of the D-band (1340 cm^{-1}, defects) to the G-band (1590 cm^{-1}, graphitic structure) was measured (Ali, 2015). Both the CNF and the CNF@Ag samples exhibited typical Raman spectra, with the D/G intensity ratio indicating a partially graphitized structure in the carbon matrix. The Raman analysis confirmed that both the CNF and CNF@Ag fibers maintained a primarily graphitic structure, with the addition of silver nanoparticles introducing a controlled amount of disorder. This balance between graphitization and defects is crucial for enhancing the membrane's filtration and antimicrobial properties, as the defective sites can improve adsorption capabilities (Chen et al., 2023).

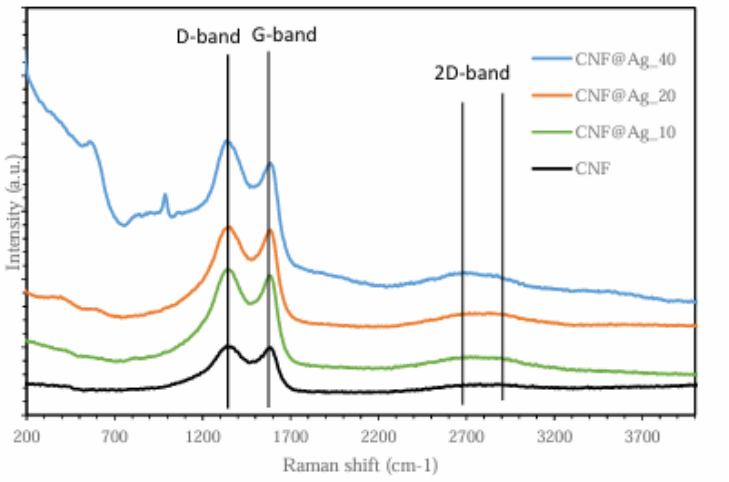

Figure 4. Raman spectra of metal oxide carbon nanocomposite fiber materials CNF, CNF@Ag-10, CNF@Ag-20 and CNF@Ag-40

3.1.4 Surface area and porosity

Figure 5 shows the specific surface area and porosity of the CNF and CNF@Ag membranes as analyzed using Brunauer–Emmett–Teller (BET) surface area analysis (Taha, 2015). The BET surface area of the composite fibers ranged from 206 to 511 m^2/g, depending on the silver nanoparticle content. The CNF had small pores of approximately 1.75 nm, while all the CNF@Ag samples had mostly mesoporous pores with an average pore size between 2.03-2.07 nm. The porosity observed in the samples was associated with a high silver particle content, which is essential for effective water filtration and microbial inhibition (Vimala et al., 2010). In addition, the size of silver nanoparticles in CNF@Ag composites calculated by BET technique was in the range of 12-20 nm. The surface area and porosity of metal oxide carbon nanocomposite fiber materials under different conditions of metal oxide nanoparticle size and precursor concentration are summarized in Table 2. However, the decrease in surface area with increase in silver content was due to the increased mass of dense silver nanoparticles relative to the mass of carbon fiber.

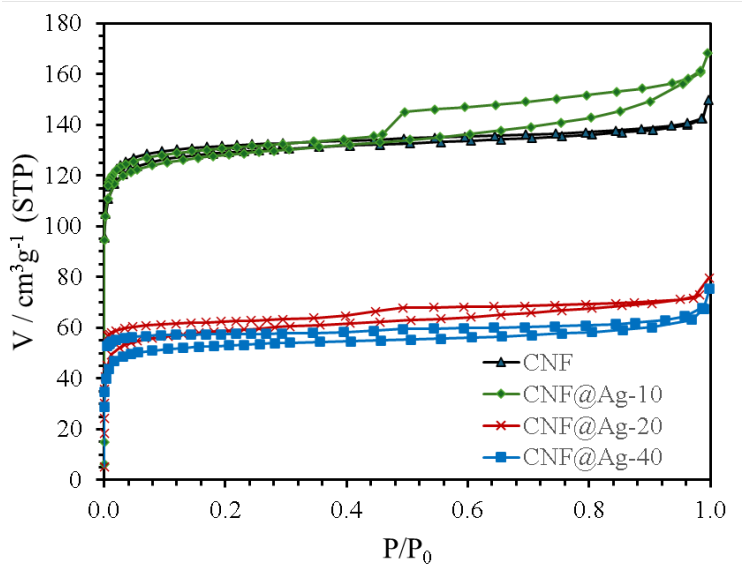

Figure 5. Isotherm linear plot of all samples

Table 2. Surface area and porosity of metal oxide carbon nanocomposite fiber materials under conditions of metal oxide nanoparticle size and different precursor concentrations of 0%, 10%, 20% and 40%

Sample	S_{BET} (m^2/g)	D_{mean} (nm)	V_{pore} (cm^3/g)	Nanoparticle Size (nm)
CNF	511	1.75	117.47	-
CNF@Ag-10	500	2.03	114.89	10 ± 6
CNF@Ag-20	229	2.03	52.77	12 ± 5
CNF@Ag-40	206	2.03	47.48	20 ± 4

3.2 Membrane preparation and filtration testing

3.2.1 Membrane preparation

Nanofibrous membranes were prepared using a process involving the mixing of CNF and CNF@Ag with a binding agent. The CNF and CNF@Ag fibers were ground into fine powders using a mortar and pestle. The ground samples were then mixed with a wet starch paste at a 1:1 weight ratio (w/w). The CNF-starch mixture was pressed into stainless steel molds using a hydraulic pressing machine at a pressure of 2 MPa for 2 min to form membranes with a thickness of 5 mm and a diameter of 25 mm. The molded membranes were then heat-treated at 200°C for 4 h to ensure structural stability. A schematic of the membrane synthesis is shown in Figure 6. Following this, the membranes were sterilized in an autoclave to eliminate any potential contaminants. After sterilization, the membranes were ready for microbial filtration tests. The heat-treated and sterilized membranes were used in subsequent experiments to evaluate their filtration efficiency and antimicrobial properties.

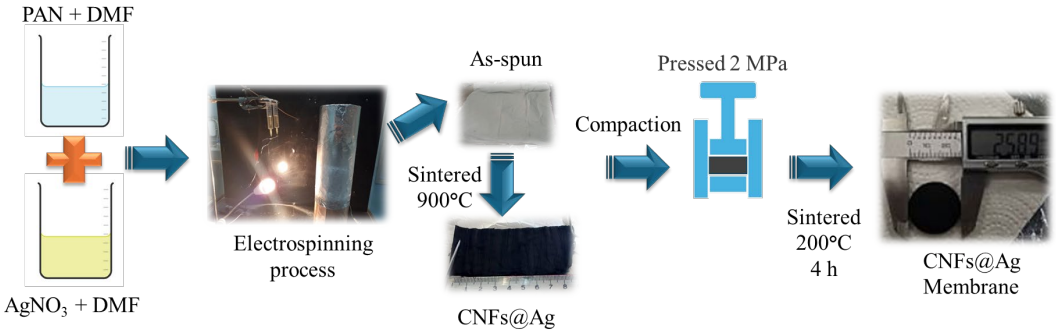

Figure 6. Schematic of silver doped CNF membrane synthesis process

3.2.2 Design of water filter set with carbon nanofiber composite reinforced with silver particles

A schematic of filtration system is shown in Figure 7. The filtration apparatus consisted of a pressure-reducing bottle that resembled a standard pink bottle but featured an extended small arm from the neck. This design allowed for improved air evacuation and enhanced filtration speed. The pressure-reducing bottle was clamped securely with a Buchner funnel (Sharp, 2012). The funnel was positioned on top of the bottle, and the nanocarbon fiber composite filter reinforced with silver nanoparticles was placed between the Buchner funnel and the small arm of the bottle.

The small arm at the neck of the pressure-reducing bottle was connected to a vacuum pump system to create negative pressure within the bottle. This setup effectively removed air from the pressure-reducing bottle, promoting faster water filtration compared to traditional methods (Zhang et al., 2020). The carbon nanofiber composite filter served as the primary filtration medium. It was designed to capture contaminants while leveraging the antimicrobial properties of the silver nanoparticles embedded within the fibers.

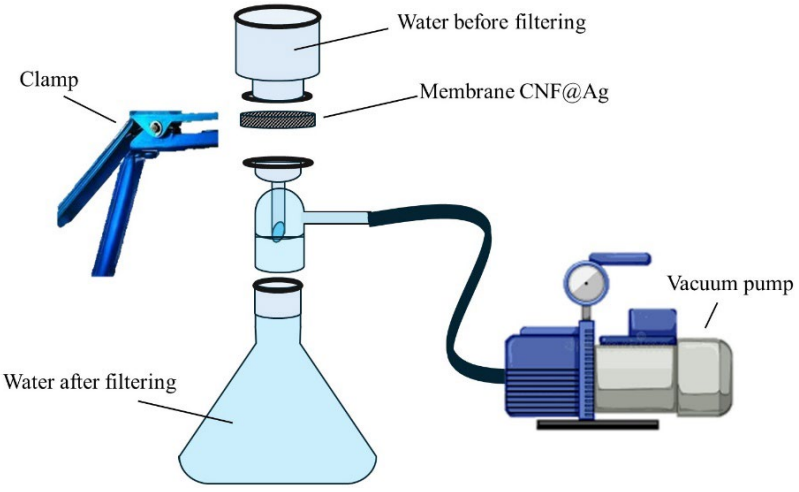

Figure 7. Schematic of filtration system

3.2.3 Study of the effectiveness of inhibiting bacteria in water

The equipment for the water filter set and the composite carbon nanofiber filters with and without silver particles were sterilized in an autoclave before microbiological experiments were conducted. The filter set was installed as illustrated in Figure 7. After installation, the proper operation of the filter set was verified by conducting the necessary operational checks. Water (250 mL) was prepared with a known initial concentration of each type of bacteria. The vacuum pump was then turned on, and the water sample was slowly poured into a filter funnel with the carbon nanofiber composite filter with silver particles. The filtered water was then taken to count the number of remaining microorganisms using the pour plate method (Terrones-Fernandez et al., 2023). The efficiency of microbial inhibition of each carbon nanofiber composite with silver particles was studied. Each type of experiment was repeated three times and the results of the microbiological analysis of the water mixed with each type of bacteria were used.

The bacterial counts in water after filtration were compared for carbon nanocomposite fibers containing silver particles under each condition using the formula:

$$\text{Numbers of colonies (CFU/mL)} = \frac{\text{Average number of colonies counted per plate}}{\text{Dilution level}}$$

From the given equation, when counting the number of bacteria after filtration and comparing it with the number of bacteria before filtration for each condition, the percentage was calculated, and the results are shown in Table 3.

Table 3. Results of percentage of microbial elimination reduced by filtration compared to the pre-filtration substrate of each sample

Types of Microorganisms	Percentage of Microbial Elimination			
	CNF	CNF@Ag-10	CNF@Ag-20	CNF@Ag-40
Escherichia coli	90.23	93.47	95.61	92.33
Salmonella	87.95	59.46	97.79	90.18
Enterobacter	89.74	79.10	87.44	98.23

3.2.4 Filtration rate test

The filtration rates of the composite nanocarbon fiber membranes were conducted using a vacuum filtration system. In this experimental procedure, the process began by preparing water and pouring it into the vacuum filter funnel. The machine was then activated to initiate filtration, with the flow rate set at 8 bars. The water gradually flowed through the membrane, and the process was timed for a duration of 5 min using a stopwatch. Once the set time was reached, the volume of water that passed through the membrane was collected in a flask beneath the vacuum filter and measured using a measuring cylinder. This volume was recorded for further calculations. To ensure accuracy and reliability, the experiment was repeated three times, with the filtration rate calculated for each replication. The results were then compared to identify the best-performing sample. It was found that the sample containing 40% silver exhibited the highest filtration rate compared to the other samples. This may be due to the membrane made from CNF@Ag-40 fibers that have silver

nanoparticles composite on both inside and outside of the fibers as observed from the SEM and TEM image in Figures 1 and 2, respectively. Therefore, there were more gaps between the fibers, allowing water to flow through quickly, resulting in the highest flow rate of 48.7 mL/min. Detailed results are presented in Table 4.

Table 4. Results of the filtration rate study of CNF and CNF@Ag

Sample	Flow Rate (ml/min)	Filtration Rate (cm^3/min)
CNF	10.6	13.5
CNF@Ag-10	6.2	7.9
CNF@Ag-20	11.2	14.3
CNF@Ag-40	48.7	52.4

3.2.5 Antibacterial properties of the membranes

The antimicrobial performance of the carbon nanofiber membranes, both with and without silver nanoparticles (AgNPs), was evaluated using water contaminated with three types of microorganisms: *Escherichia coli*, *Salmonella*, and *Enterobacter*. The filtration tests demonstrated that the membranes exhibited high efficiency both in filtration and in inhibition of microbial growth. The results indicated that the membranes reduced the microbial count in filtered water by 59.46% to 98.23%. CNF@Ag-20 showed the highest elimination of *Escherichia coli* and *Salmonella* at 95.61 and 97.79% whereas CNF@Ag-40 gave the highest elimination of *Enterobacter* at 98.23% (Table 3). Despite these high inhibition rates, the incorporation of AgNPs into the carbon nanofiber membranes did not show a significant increase in antimicrobial activity compared to the membranes without AgNPs. The performance difference between the two types of membranes was minimal. Nevertheless, both types of membranes exhibited excellent antimicrobial properties.

The summarized performance of both membrane types (with and without silver nanoparticles) in filtering and inhibiting *Escherichia coli*, *Salmonella*, and *Enterobacter* indicates that even without silver, the membranes maintained high antimicrobial activity. This finding is noteworthy as it contrasts with previous studies where the incorporation of silver nanoparticles generally enhanced antimicrobial properties (El-Shanshoury et al., 2011; Pryskoka et al., 2015; Panáček et al., 2016; Saeed et al., 2020). Silver nanoparticles have been well-documented for their strong antimicrobial activity, and their inclusion typically improves antimicrobial performance in membrane systems.

However, in this study, the carbon nanofiber membranes alone demonstrated strong antimicrobial efficacy. This suggests that the main contributing factor to bacterial inhibition might be the membrane's pore size, rather than the bactericidal effect of the silver nanoparticles embedded in the membrane. In cases where silver nanoparticles are expected to exhibit bactericidal activity, prolonged contact between bacteria and nanoparticles is necessary, typically over several hours (Yin et al., 2020; Raza et al., 2023) In contrast, the bacteria in this experiment were in contact with the silver nanoparticles for a relatively short duration (of the order of minutes) due to the rapid water flow through the membranes.

These findings present a distinctive perspective in the field of membrane filtration, particularly with the potential role of the nanofiber structure itself in antimicrobial action. Further studies are required to explore the long-term effects of silver nanoparticle interactions with bacteria under varying conditions of contact time and concentration.

4. Conclusions

We successfully fabricated carbon nanocomposite fibers containing silver particles at concentrations of 0-40% using electrospinning. The average diameters of the fibers ranged from 527 to 750 nm, and silver nanoparticles with diameters between 6 and 35 nm were uniformly distributed throughout the fibers. These nanoparticles were embedded within the amorphous carbon structure, with some forming on the exterior of the fibers. The fibers exhibited a specific surface area of 229-511 m^2/g and a pore size ranging from 1.75 to 2.07 nm. Filtration tests revealed a filtration rate of 7.9-14.28 mL/min at a pressure of 8 Bar. In experiments using three types of microorganisms *Escherichia coli, Salmonella, and Enterobacter* and in terms of the filtration properties and the antibacterial properties, it can be predicted that in the case study, the main factor was probably the pore size rather than the toxicity of the silver nanoparticles.

5. Acknowledgements

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6. Authors' Contributions

Tanayt Sinprachim: Conceptualization, Methodology, Writing- Original draft preparation, Natta Kachenpukdee: Data Curation, Methodology, Kattinat Sagulsawasdipan: Data Curation, Methodology, Chakhriya Chalad: Data Curation, Methodology, Maytungkorn Sermsuk: Data Curation, Somchai Sonsupap: Writing- Reviewing and Editing, Visualization, Investigation

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7. Conflicts of Interest

The authors declare that there is no conflict of interest in this study.

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