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Facile fabrication of low-cost activated carbon bonded polyethersulfone membrane for efficient bacteria and turbidity removal

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ABSTRACT

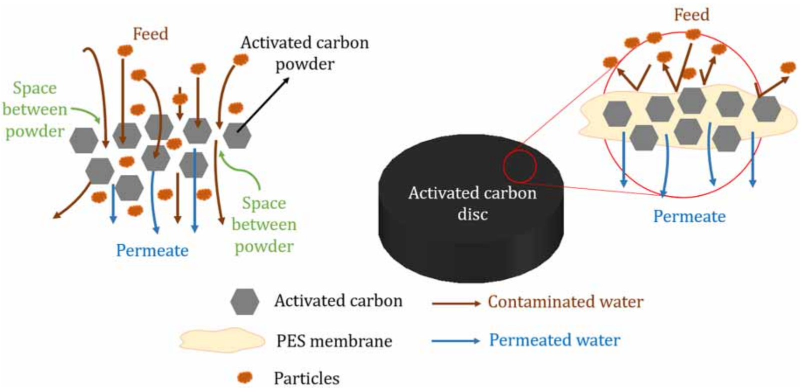
The current research aimed to fabricate a cost-effective activated carbon disc for bacteria and turbidity removal in contaminated water using polyethersulfone membrane solution as a bonding agent. The mixing compatibility and bonding stability of the blend activated carbon disc were studied with a bonding strength test. The morphology of activated carbon discs was studied by a microscope. The activated carbon discs had a thick dense layer between the powder. Activated carbon discs significantly removed the total coliforms populations (99%) when evaluated against river water whilst removal by the powder was only up to 90%. The turbidity removal efficiency for the activated carbon increased from 29%-79% with the utilization of the membrane as the bonding agent in forming the disc. However, the pH of water treated by the activated carbon powder and disc did not change significantly, yet it lay within the pH range of safe drinking water (6.5–7.7). It revealed the important role of PES membranes for the activated carbon discs to improve coliform and turbidity removal in the water, ensuring the quality of water resources.

Key words: activated carbon, good health, polyethersulfone membrane, total coliform, turbidity, water filtration

HIGHLIGHTS

- Activated carbon disc was fabricated by using polyethersulfone (PES) dope solution as a bonding agent.
- Fabricated activated carbon disc reduced water turbidity up to 79%.
- Activated carbon disc removed total coliform in the water up to 99%.

GRAPHICAL ABSTRACT



1. INTRODUCTION

Freshwater is a fundamental resource for human well-being and living things; it is the most essential natural resource in the world (Liu *et al.* 2015; Yimyam 2017). Limited access to clean water due to water pollution, improper water treatment, poor distribution of water resources, and climate changes have become a major challenge in some countries (Pintilie *et al.* 2016; Khan *et al.* 2017). Many countries deal with the increasing demand on fresh water supplies and struggle to tackle inadequate clean water resources. Water sources are now produced in many ways to fulfill the increasing demand. For example, people collect water from ponds and rivers, but now they can obtain water from unconventional sources, such as simple water treatment and water purification (Ang *et al.* 2011; Lee *et al.* 2017). Currently, wastewater reclamation has received much attention as an alternative source of freshwater for irrigation and industrial purposes (Diaz-Elsayed *et al.* 2020; Echevarría *et al.* 2020).

Different methods, such as adsorption (Aghababaei *et al.* 2021; Aragaw 2021) and membrane filtration (Davari *et al.* 2021; Mahmoud & Kochameshki 2021) have been used for the water treatment process. In terms of adsorption, adsorbents for wastewater treatments are made of many materials, such as activated carbon (Belhamdi *et al.* 2020), hydroxyapatite (Hariyanto *et al.* 2020), polymers (Lou *et al.* 2021), zeolites (Konale *et al.* 2020), and clay (Ntwampe 2020). Due to the large surface area, low fabrication cost, and materials in use, activated carbons commonly become adsorbents for removing pollutants in contaminated water (Vargas & Lopes 2020; Ashiqa *et al.* 2021; Mueanpun *et al.* 2021; Sidiqua & Priya 2021). Besides, the main resources to produce activated carbon are from agricultural and industrial waste, which is widely available (Udaiyappan *et al.* 2017; Lakshmi *et al.* 2018). An activated carbon has also been combined with ultrafiltration in removing antibiotics, beta-blockers, psychiatric drugs, and steroid micropollutants (Sbardella *et al.* 2018; Tagliavini & Schäfer 2018).

For ultrafiltration, polyethersulfone (PES) membrane is considered as the core polymer material infiltration because of its good mechanical strength, chemical resistance, and thermal stability (To *et al.* 2015; Madaeni *et al.* 2015; Abdel-Aty *et al.* 2020; Yonita *et al.* 2020).

The powder and granular activated carbon, known as a low-cost adsorbent, has become a water filter (Balaji *et al.* 2020; Mukherjee & Bandyopadhyaya 2021). However, the contaminants in the water still can pass through in between activated carbon powder or granules, resulting in low filtration and adsorption efficiency. Commercial disc and hemispherical activated carbon have better water filtration performance. Researchers used a pitch foaming process to prepare high surface-area activated carbon discs (Gao *et al.* 2017a, 2017b). However, foaming processes carried out at a high pressure promote an additional complexity, for example, cost and risk for laboratory or even industrial scale.

This current study proposed an approach of using a low-cost dope solution as a bonding agent to produce an activated carbon disc. This method could reduce the use of advanced equipment and energy required to fabricate activated carbon discs at a high pressure and temperature. Polymer solution as the bonding agent may serve as a matrix in powder composites to improve the mechanical strength of the discs and provide additional filtration alongside the surface area of the activated carbon particles.

Activated carbon powder was selected as filler particles because they were relatively inexpensive compared to carbon nanotubes or graphite. As a matrix producing activated carbon discs, a polyethersulfone membrane solution was selected. The different composition ratios of PES solution (30 wt.% and 50 wt.%) to produce an activated carbon disc were investigated along with the ratios of activated carbon powder. A filtration test was conducted to find the activated carbon disc efficacy; that is, its capability for removing bacteria and turbidity from the water sample and its bonding strength.

2. MATERIALS AND METHODS

2.1. Fabrication of activated carbon discs

Commercial activated carbon powder was used as a filler, and polyethersulfone (Sumitomo Chemical Co., Ltd, Japan) was used as a polymer in dope solution. Polyvinylpyrrolidone (PVP) and 1-methyl-2-pyrrolidone (NMP) were purchased from Merck KGaA, Darmstadt, Germany. The preparation of the PES dope solution was performed similarly to what the researchers have studied (Prihandana *et al.* 2014). PES (20 wt.%) and PVP (20 wt.%) were dissolved in NMP at 80 °C. The dope solution was then added to the activated carbon powder at different weight ratios in percentages (30 wt.% and 50 wt.%) and properly mixed until a homogeneous blend was formed. The discs (40-mm diameter) were formed in a hydraulic mold, pressed, and dried in an oven at 100 °C for one hour. After the drying process, the discs were then weighed, and their thickness was measured (see Figure 1). The surface morphology of the fabricated discs was then observed using a digital microscope.

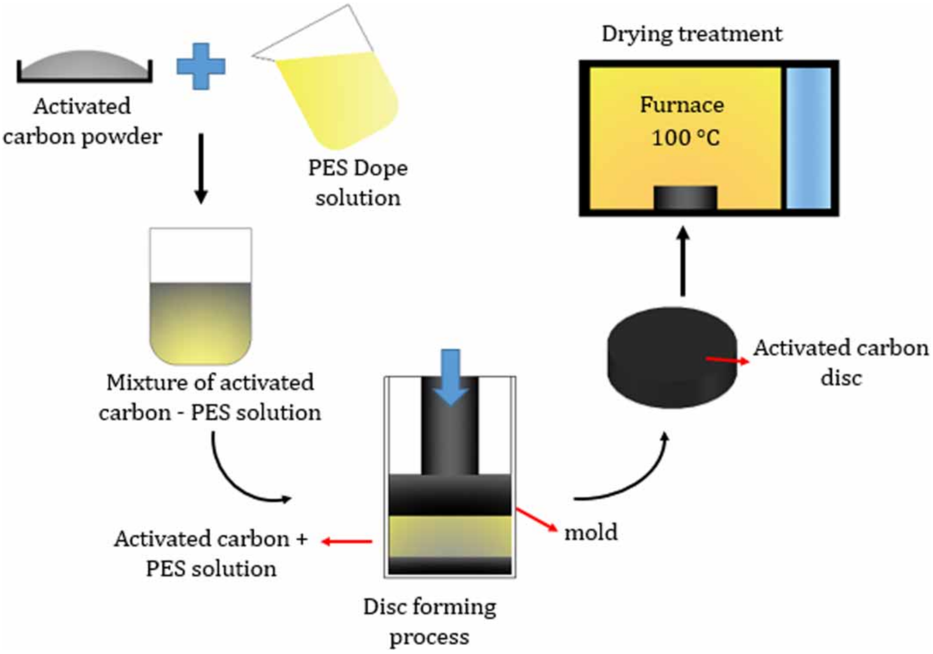


Figure 1 | Preparation of activated carbon discs.

2.2. Surface morphology of the activated carbon discs

Surface morphology analysis was conducted by using Dino-Lite Microscope DINO AM3103. This analysis provides visual information on the surface morphology of the discs, the activated carbon powder, and the dense layer formed by PES solution amongst the fine grains.

2.3. Bonding strength test

A bonding strength test was conducted to measure the strength of the dense layer in preventing the discs from crumbling by immersing the discs into the water for 48 hours. After immersion, the discs were dried in the oven at 100° C until the water was removed. In the real application, the discs should have had a direct contact with water during the filtration process. Thus, measuring the bonding strength is necessary when the disc is immersed in the water. The bonding strength of the dense layer was calculated according to the wear formula and particle loss during the immersion test (Zodrow *et al.* 2009; Chowdhury *et al.* 2021).

$$WLR\ (\%) = \frac{W_a - W_b}{W_a} \times 100\% \tag{1}$$

W_a is the weight of the activated carbon disc before the water immersion test, W_b is the weight of the activated carbon disc after the water immersion test and WLR is the weight loss ratio of the activated carbon disc.

2.4. Filtration test

The disc was placed at the base of the filtration cell to be tested for its permeation under gravity force. Figure 2 presents the experimental set up for water filtration. A non-woven fabric was placed at the bottom of the filtration cell. Should any carbon powder have detached from the AC disc, the non-woven fabric would prevent the powder from being washed away by the water flow during the filtration process. The filtration cell was filled with the sample water taken from a river in the Special Region of Yogyakarta, Indonesia. The permeation of the sample water through the disc was then analysed for bacterial removal, pH, and turbidity (U.S. Environmental Protection Agency 1973; APHA/AWWA/WEF 2012).

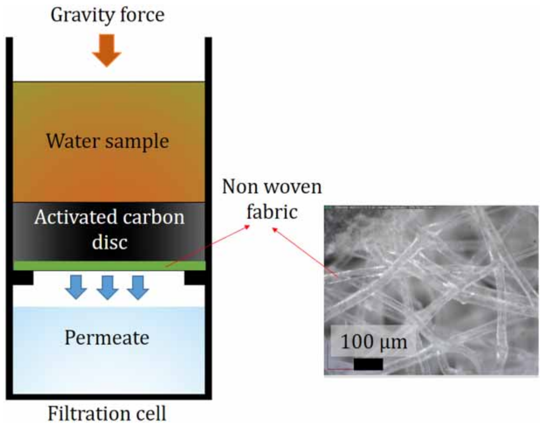


Figure 2 | Filtration test on activated carbon discs and morphology of non-woven fabric.

2.4.1. Total coliform test

A coliform test was conducted for microbiological examinations of water samples to determine the quality of the filtrated water. The coliform group of bacteria is the main indication of the suitability of water for domestic use and others. The presence of coliform bacteria causes water to be potentially unsafe for consumption or use. In this study, the multiple-tube fermentation (MTF) method was performed to examine the presence of coliform in the water sample (APHA/AWWA/WEF 2012).

2.4.2. pH test

Feed water was collected from a river in Yogyakarta, Indonesia. This river stream has a natural surface water inlet and outlet. The pH of the collected water was found to be 6.5. According to Zhang *et al.* (2021), this pH value is still safe for consumption.

2.4.3. Turbidity test

Water turbidity, as one of the basic parameters in the water quality analysis, was measured using a turbidity meter. Turbidity represents the existence of suspended and dissolved solids in the water (Khiari *et al.* 2020). However, turbidity does not always interpret as a direct risk to public health; the suspended solids responsible for turbidity can be a carrier to heavy metals and microbial pathogens in the water (Bilotta & Brazier 2008).

2.4.4. Fe filtration test

A Fe separation test was conducted using Ferrous Chloride Tetra Hydrate ($FeCl_2.4H_2O$) to make Fe ions solutions. The feed solution with the individual metal of Fe was provided at an initial concentration of 10 mg Fe/l (Kasim *et al.* 2017). The concentration of Fe in the water was measured using a pack test ion-selective kit from Kyoritsu Chemical-Check Lab. Corp., Japan.

3. RESULTS AND DISCUSSION

3.1. Surface morphology of fabricated activated carbon discs

Figure 3 shows the appearances of the fabricated activated carbon disc surface with different ratios of PES solution. Activated carbon blend polymers were fabricated using a dry phase inversion method known as precipitation by solvent evaporation. In this method, the solvent in the dope solution is allowed to evaporate in the atmosphere to free the water vapour, allowing a dense, homogeneous membrane to be formed (Mulder 1996), since the bigger the demixing gap is, the easier it is to form a denser membrane structure (Han *et al.* 2010). In Figure 3, the activated carbon disc, which was made of 50% PES solution, has more coverage of PES dense layer attached in the disc surface and structure compared to the AC-Disc 30. This result was due to the higher volume of PES solution generating more binder utilized in fabricating AC-Disc 50.

Table 1 presents the weighted activated carbon discs before and after the bonding strength test. Activated carbon discs delivered a low weight loss ratio after the bonding strength test and remained intact. The bond created by the PES dope solution successfully secured the powder from collapsing.

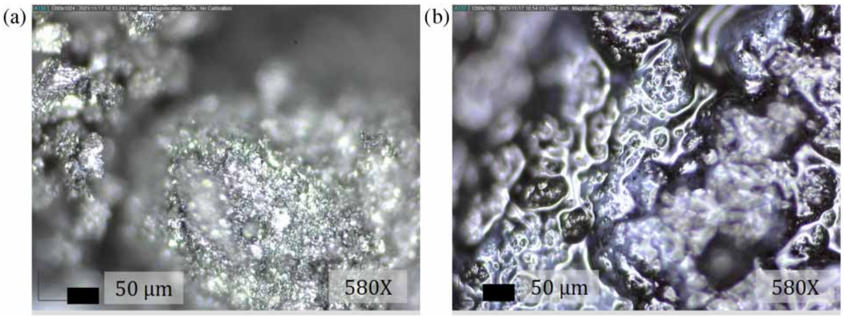


Figure 3 | Activated carbon discs; (a) 30% of PES solution, (b) 50% of PES solution.

Table 1 | Weight loss ratios of the activated carbon discs after the immersion test

		Disc weight (grams)		
No	**AC types**	**Before**	**After**	**Weight loss ratio (%)**
1	AC-30%PES (AC Disc 30)	25.7	22.4	12.8
2	AC-50%PES (AC Disc 50)	29.3	24.4	16.5

In addition to the bonding test, a filtration test was performed for 10 minutes under gravity force. A non-woven mesh was used to prevent the detached carbon grains plunged into the permeate chamber during the filtration test, as shown in Figure 2. Experiment results showed that the flow rate of the AC-powder, AC-disc 30, and AC disc 50 within 10 minutes were less than 1 ml, 5 ml, and 20 ml, respectively. The no-binder AC powder gave the lowest flow rate because some carbon powder blocked the pores of the non-woven fabric. The AC-30 had a lower flow rate because the PES solution could not bind the carbon grains as tightly as that in AC-50. This resulted in some powders detaching from the disc, and filled the pores of the non-woven fabric that acted as separator in the water filtration experimental set-up. Due to this random blockage, the flow rate given is lower. This is in accordance with the flow rate of no-binder AC powder, which gave the lowest flow rate since the fabric pores were mostly blocked by the AC powder. On the other hand, the AC-Disc 50 provided the highest flow rate compared to the AC-Disc 30. This was due to the membrane being able to bind the powder properly, thus preventing it from blocking the non-woven pore.

3.2. Filtered-river water using an activated carbon disc

Biological and physical parameters were used in analysing river water filtration. The amount of total coliform was used as a biological parameter, whilst turbidity and pH were analysed as physical parameters.

3.3. Rejection of total coliform

Table 2 presents the amount of the total coliform before and after the filtration test. The number of total coliforms in the river water was 24×10^6 MPN/ml. Detected in the filtrated water, it significantly decreased by the application of the filter.

Table 2 | The detected number of total coliforms before and after filtration test

	Amount of bacteria (MPN/ml)			
	River	**AC-powder**	**AC Disc 30**	**AC Disc 50**
Total coliforms	24×10^6	24×10^4	1,400	5,400

Figure 4 summarizes the result of the coliform removal by different types of filters presented in percentage. The filtration efficiency of filters was measured for coliform population. The activated carbon powder removed 90% of coliforms from the river water due to its pore size, where it is too small for bacteria (1–2 μm × 0.5 μm) to enter (Walker & Weatherley 1998; Lu *et al.* 2020). According to Wang *et al.* (1995) the pore-size range for activated

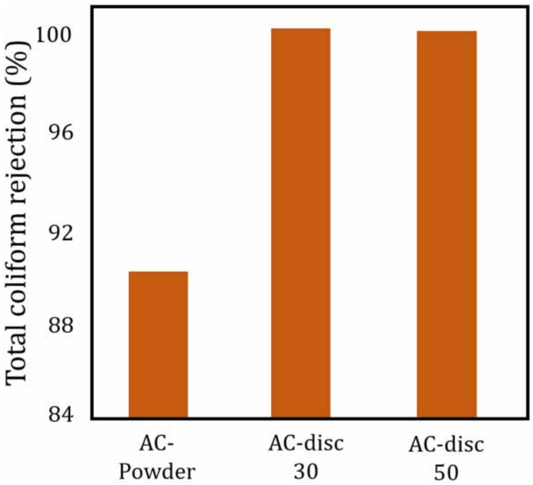


Figure 4 | Removal of total coliforms in the river water.

carbon particles is thought to be 2–5 μm, where it guarantees sufficient high adsorption capacity while allowing for bacterial adhesion on the surfaces. However, the activated carbon disc had better performance than the activated carbon powder in removing the total coliforms up to 99%. Larger spaces between adjacent powder might have obstructed the sorption of the contaminants from the water. Similar to these findings, Wegelin (1996) and Ni'matuzahroh *et al.* (2020) asserted that a roughing filtration process by powder filter is not optimal in reducing the number of total coliform in the contaminated water. Furthermore, the PES membrane solution connected activated carbon fine grains and combined them into one compound for significant reduction. The formed PES membrane, which is attached to the surface of the activated carbon powder, created a smaller filter pore (see Figure 5). Principally, the coliforms attached to the water impurities; when the impurities are not allowed to go through the filter, the coliforms population that passes through the filter was reduced (Tripathi *et al.* 2019).

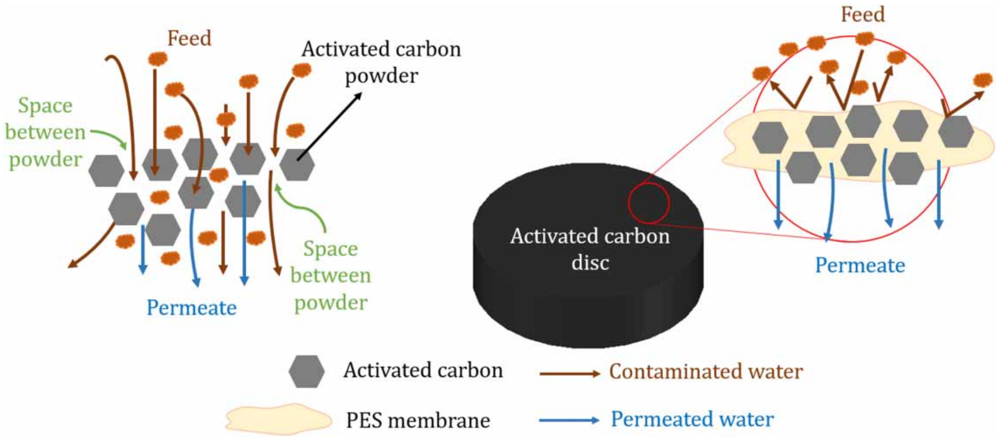


Figure 5 | Mechanism of blended PES membrane-activated carbon in removing coliforms.

According to Kanagaraj *et al.* (2015) and Sarbolouki (1982), the solute rejection (in this case bacteria rejection) above 80% can used to determine the average pore size. Thus, the average pore size of the activated carbon disc can be determined by using the following equation:

$$\bar{R} = 100\left(\frac{\propto}{\%SR}\right) \tag{2}$$

where $\bar{R}$ is the average pore size (radius), $\propto$ is the radius of the bacteria (0.5 μm) in the water sample and SR is the bacteria rejection.

Based on the reference and experimental result, the radius of the bacteria is 0.25 μm (Walker & Weatherley 1998) and the rejection of bacteria is 99%, therefore the average pore size (radius) of the AC-Disc is 0.25 μm.

3.4. pH test

Table 3 presents the results of the filtrated water in terms of pH value. The pH value of the river water was 6.5. After the filtration process using the activated carbon powder, the pH value changed to 7.3. The changes in pH values in the filtrated water by activated carbon and AC-30 was caused by an ion exchange phenomenon, where the surface of carbon sorbs the anions and corresponding hydronium ions from the water, resulting in pH increase. (Farmer *et al.* 1996). In the case of pH filtrated water by AC-50, it seems that the higher amount of PES membrane on the disc reduced the effect of increasing pH caused by activated carbon powder. The pH values for the filtrated water using the activated carbon discs were in the safe limit range of 6.5–7.7, in which most drinking water has the pH range of 6.5–8.5 (APHA/AWWA/WEF 2012; Zhang *et al.* 2021).

Table 3 | pH of the tested water

	Results			
Parameters	**River water**	**AC-powder**	**AC Disc 30**	**AC Disc 50**
	6.5	7.3	7.7	6.5

3.5. Turbidity test

Table 4 presents the results of the turbidity test on the filtrated water. The turbidity of the river water was at 14 NTU, and it was reduced to 10 NTU after the filtration using the activated carbon powder. The reduction likely occurred by the influence of sorption capacity and a high surface area of the activated carbon powder. Being filtrated with the activated carbon disc (AC Disc 50), the turbidity of river water was measured at 3 NTU. The result indicated that the introduction of the PES membrane into activated carbon contributed a significant effect on NTU reduction of up to 79%, compared to the individual effect of the activated carbon (Dialynas & Diamadopoulos 2008; Vargas & Lopes 2020). Moreover, these variations in turbidity filtration are related to different pore dimensions of discs and powder (Someya *et al.* 2021), as well as the free space between powder, allowing particulate matter to pass through the filters (Suzuki *et al.* 2020).

Table 4 | Turbidity of the tested water

	Results (NTU)			
Parameter	**River**	**AC-powder**	**AC Disc 30**	**AC Disc 50**
Turbidity	14	10	5	3

3.6. Fe separation test

The results of the Fe concentration before and after the filtration process are explained in Table 5. The AC-powder removed the Fe ions in the water sample by up to 50% due to its suitable surface functional groups and appropriate pore diameter (Mariana *et al.* 2021). However, some Fe ions were still detected during the permeation since there were spaces between the powder. On the other hand, the AC Disc 50 gave the lowest Fe concentration because of the PES membrane. Such PES membrane can perform the adsorption mechanism which is associated with functional groups to remove the metal ions (Khulbe & Matsuura 2018).

Table 5 | Fe concentration of the water sample

	Results (mg/L)			
Parameter	**Feed (water sample)**	**AC-powder**	**AC Disc 30**	**AC Disc 50**
Fe concentration	10	5	1	0.5

4. CONCLUSIONS

A low-cost innovative method using the PES membrane-embedded activated carbon powder successfully increases the selectivity of the carbon powder. The visual analysis verifies that the PES solution forms a dense layer to fill in spaces of the fine powder, thereby making the mixture one solid compound. The embedded PES membranes in the fine powder removed the total coliforms in the selected river water up to 99%. The membrane in the activated carbon disc reduced the turbidity of the river water by up to 79%. In conclusion, the activated carbon powder embedded in the PES membrane is a low-cost yet promising filter for water filtration treatment.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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