



Electrospun nanofibrous membranes with antimicrobial activity for air filtration

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ABSTRACT

Air pollution, including airborne pathogens and particulate matter (PM), has become a prominent issue affecting human health and safety. Conventional air filtration materials do not meet the requirements for efficient PM capture or do not instantly kill pathogens, leading to increased risk of direct/indirect contact transmission and infection due to the accumulation of pathogens during filtration. Electrospun nanofibrous membranes have emerged as a promising platform due to their rich porous structure, finer fiber diameters, good internal connectivity, and the ability to easily incorporate active chemicals for antimicrobial function. In this review, antimicrobial mechanisms of nanofibrous membranes for air filtration and PM capture mechanisms of nanofibers were firstly investigated, and various types of electrospun nanofibrous membranes with different antimicrobial agents for efficient air filtration were described in detail, including organic antimicrobial agents, inorganic antimicrobial agents and metal–organic frameworks. We hope this work could provide a better practical insight for designing novel electrospun nanofibrous membranes with antimicrobial efficacy for efficient air filtration.

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1. Introduction

Air pollution, especially suspended disease-causing microorganisms and particulate matter (PM), has become a high-profile environmental and health issue. Airborne transmission of pathogens or viruses is the main route of transmission for most respiratory infectious diseases, such as *Yersinia pestis* [1], *Mycobacterium tuberculosis* [2] and the coronavirus disease 2019 (COVID-19) [3]. Additionally, even ordinary airborne PM have been also verified to be a cause of many health problems, such as preeclampsia [4], cardiovascular disease [5] and atherosclerosis [6]. In particular, the smaller size of PM_{2.5} can easily penetrate into the bronchi and lungs and cause very serious damage [7]. More seriously, the latest research shows that plastic particle pollution is found in human blood [8], which further highlights the importance and necessity of air filtration.

Air filtration is an excellent means of protecting people from the above pollutants. Present practical applications are dominated by fiber-based air filtration materials prepared by extruded, drawn or meltblown techniques, such as glass fibers and various types of meltblown fibers. However, such fibrous materials often exhibit poor or instability filtration performance for fine airborne nanopar-

ticles due to the large pore size formed by the microfibers. In addition, the accumulation of viruses, bacteria or PM particles on the fibers during using not only reduces filtration efficiency, but increases risk of transmission and infection [9]. The majority of bacteria (90%) have been reported to survive on masks even after 8 h, and what's more, some bacterial pathogens can survive on textile materials for 1–90 days, which may pose a health hazard when using filters [10]. A series of protective regulations for the antimicrobial properties of filters have been published, such as the group standard T/CIAA 003–2020 "Antimicrobial Masks". It is clear that the development of air filtration materials with high filtration performance and antimicrobial activity is a need for the future.

Compared to microfibers, electrospun nanofibrous membranes show decreased pressure drop due to the slip effect and can improve on the deficiencies of aforementioned conventional filtration materials in high performance filtration, due to their rich porous structure, finer fiber diameters, good internal connectivity, and the ability to easily incorporate active chemicals for multifunctionality [11,12]. High efficiency nanofibrous membranes can be achieved via enhanced space charge strategies by adding electret particles or enhanced functional group interaction strategies for capturing PM via ion-dipole interactions [13]. To further meet filtration needs and achieve greater safety, new filtration nanofibrous membranes with antibacterial and antiviral properties are being investigated for various applications, such as personal protective equipment, air

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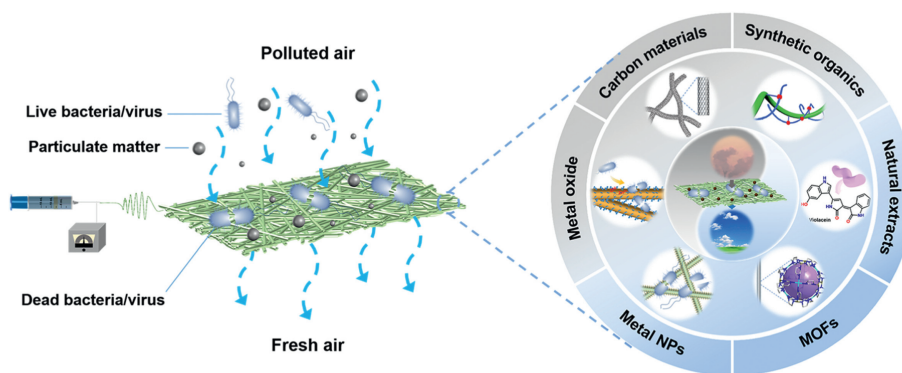


Fig. 1. Recent developments in electrospun nanofibrous membranes with different antimicrobial agents for efficient air filtration.

filters for ventilation systems, and filters for medical devices. For instance, ultrafine nanofibers with enhanced antimicrobial effects were fabricated from nylon 6, chitosan and curcumin for development for personal protection [14]. At this juncture in development, we feel it necessary to conduct a recent review to summarize the progress and to highlight the need for further research and development.

In this review, we summarized recent progress on electrospun nanofibrous membranes in air filtration, especially those with antimicrobial property. In the first part, the antimicrobial mechanisms of nanofibrous membranes for air filtration and PM capture mechanisms of nanofibers were explained. The second part summarized various types of nanofibrous membranes with antimicrobial efficacy for efficient air filtration, including organic antimicrobial agents-based nanofibrous membranes, inorganic antimicrobial agents-based nanofibrous membranes and metal–organic frameworks-based nanofibrous membranes (Fig. 1). Finally, the future perspectives for their development and the challenges encountered on the way to commercialization were discussed.

2. The mechanism of antimicrobia and PM capture

2.1. The selection criteria for antimicrobial agents

Antimicrobial agent, as the active ingredient of antimicrobial activity, occupies an extremely important role in antimicrobial nanofibrous membranes, and the requirements for antimicrobial agents include: (1) Broad-spectrum antimicrobial resistance. It is resistant to a wide range of germs, even fungi and viruses. (2) Persistence. It possesses the properties of abrasion resistance, heat resistance, sunlight resistance, long life, and not easy to decompose and fail. (3) Compatibility. It is easy to be added to fiber materials, and is not easy to change color or reduce the use value of the product. (4) Safety. It is harmless to human health and does not cause environmental pollution. (5) Low cost. It should be characterized by a wide range of sources, ease of preparation and relatively mature production technology. (6) Bacteria are not easy to produce drug resistance.

2.2. Antimicrobial mechanism of nanofibrous membranes for air filtration

Currently, antimicrobial agents used in nanofibrous membranes for air filtration can be divided into three major categories, organic antimicrobial agents, inorganic antimicrobial agents and composite antimicrobial agents, based on their chemical composition. Typical antimicrobial ingredients in organic antimicrobial agents include quaternary ammonium salts, biguanides, *N*-halamine compounds, phenolic compounds, and so on. The underlying mechanisms by

which these take their roles in antimicrobial are also different. For instance, quaternary ammonium salts, the electrostatic interaction between the positively-charged group and the negatively-charged phosphoryl group in the phospholipid component of the bacterial cell membrane changes the permeability of the bacterial cell membrane, resulting in the bacteria rupture and die caused by the outflow of internal substances such as DNA [15]. *N*-Halamine compounds, the halogens can be delivered into bacteria by direct contact or released into the environment before being transferred into bacteria, destroying internal substances such as enzymes, proteins and DNA [16,17]. Phenolic compounds, taking 5-bromosalicylic acid (BSA) as an example in Fig. S1 (Supporting information) [18]: (1) High levels of phenolic compounds alter the conformation of the phospholipid bilayer by crowding the internal space of the bacterial cell membrane, leading to impaired membrane function and material leakage. (2) The dissociation of phenolic compounds into the bacterial cytoplasm releases large amounts of protons into the cytoplasm, which further leads to the excretion of large amounts of intracellular cations such as potassium ions. As potassium is the core ion for the synthesis of adenosine triphosphate (ATP), potassium deficiency deprives bacteria of the energy required for normal activity and leads to bacterial death.

For inorganic antimicrobial agents, such as metal nanoparticles, metal oxygenates and carbon materials, the main antimicrobial mechanisms include contact reaction mechanism, active oxygen mechanism and thermal sterilization mechanism in Fig. S1. Metal ions (such as copper) can combine with the phospholipid membrane or peptidoglycan membrane or proteins in cell membrane, thus destroying the integrity and permeability of the cell membrane [19,20]. Nanoscale metal has well-known antimicrobial properties, which can arise from physical interactions [21]. Rupture of the cell membrane can result in an osmotic imbalance and leakage of the internal cell components, which results in cell death [22]. Moreover, some materials can convert electrical or light energy into heat when powered or irradiated. Then, the high-temperature layer can kill bacteria and even virus [23]. As the main bactericidal effect, oxidative stress induction has been extensively studied. After the materials absorb the energy of the environment, the oxygen in the air or water adsorbed on the surface of the materials is activated to generate hydroxyl radicals and reactive oxygens, which have strong redox ability and accelerate the oxidation process within the bacteria and ultimately destroy internal substances such as enzymes, proteins and DNA, which thus achieve disinfection [24].

2.3. The mechanism of PM capture

PM, composing of droplets and small particles, is a complex mixture of organic and inorganic substances. For the different sizes

of particles, PM_x refers to particles smaller than $x \mu\text{m}$ in diameter. PM capture mechanism of fibrous membrane is mainly based on the following mechanisms (Fig. S2 in Supporting information) [25]: (1) Inertial impact: This effect occurs when the particles are larger than $1 \mu\text{m}$ in diameter with a larger mass. Due to their greater inertia they cannot change their direction of movement in time along the curve paths of the air and thus collide with the fibers and adhere to them [26]. (2) Gravitation: The effect of gravitational settling also occurs when the particles are heavy. If their mass is light or their diameter is less than $0.5 \mu\text{m}$, then this effect is even negligible [27]. (3) Interception: This effect occurs for particle diameters greater than $0.1 \mu\text{m}$ and is particularly dominant in the range of $0.1\text{--}1 \mu\text{m}$. The fine particulate matter comes into contact with the fibers via the van der Waals forces and is adsorbed by the fibers [28]. (4) Diffusion: This effect occurs in the case of very light particle masses, especially small particles below $0.1 \mu\text{m}$ in diameter, which spread irregularly with the direction of airflow movement by Brownian motion and are immediately captured and adsorbed if they collide with fibers [29]. (5) Electrostatic attraction: Traditional air filters are passively trapped via small enough pores to intercept ultrafine particles, so breathability is bound to be compromised. If the fibers carry an electrical charge, they will capture PMs via an electrostatically attractive manner, making the filtration efficiency much higher [30,31]. For example, commercially available masks must pass through an electrostatic electret step through a high-voltage electric field, which causes the fibers to carry an electrical charge to improve filtration performance. However, the electrostatic charge of the fibers is easily dissipated in a humid environment, leading to a decline in filtration performance [32]. So, new forces such as dipole-dipole interaction or ion-dipole interaction have shown great potential for stable and long-lasting air filtration, and functionalized fibrous membrane materials are in urgent need of intensive research to provide new options for air filtration [33,34].

2.4. Characterization of PM filtration performance

The characteristics of high filtration performance, low airflow resistance, stable air pressure drop, and high filter quality factor (QF) constitute the focus of filtration properties. The filtration efficiency η is calculated by Eq. 1:

$$\eta = (1 - N/N_0) \times 100\% \quad (1)$$

where N_0 and N are the particle number before and after filtration, respectively [35]. To evaluate the overall performances of membranes in terms of both filtration efficiency and pressure drop, the quality factor (QF) is calculated by Eq. 2:

$$QF = -\ln(1 - \eta)/\Delta P \quad (2)$$

where η is PM filtration efficiency and ΔP is pressure drop [35].

3. Antimicrobial nanofibrous membranes for air filtration

Over the past few years, a variety of electrospun nanofibrous membranes with different antimicrobial agents, such as organic antimicrobial agents, inorganic antimicrobial agents and composite antimicrobial agents, have been proposed that can be used to solve the trade-off between filtration efficiency and pressure drop in air filtration. At the same time, they can kill the attached microbia to prevent secondary infection. These emerging materials are discussed in the following sections.

3.1. Organic antimicrobial agents-based nanofibrous membranes

3.1.1. Synthetic organics

In recent years, some research work on electrospun nanofibrous membranes to improve air quality was reported, including

polyacrylonitrile (PAN), polyvinylpyrrolidone (PVP), polyvinyl alcohol (PVA) and polystyrene (PS) nanofibrous membranes [36–39]. The difference between the several nanofibrous membranes was that the polarity of the polymer decreases in turn, resulting a decrease in filtration performance in turn. These results demonstrated that the filtration efficiency of nanofibrous membranes increased with increasing polymer polarity, as strong dipole-dipole forces favored the attraction between PM and nanofibers, which also laid the foundation for stable and long-lasting filtration performance of future nanofibrous membranes. Moreover, some groundbreaking studies on two-dimensional ultrafine nano-nets were developed [40–42]. The extremely small pores of these nanofibrous membranes prevented even $PM_{0.3}$ from passing through, which was another way of achieving the goal of efficient and stable filtration. However, in order to be safer, it is reasonable to endow the nanofibers antibacterial and antiviral properties in an environment where disease-causing microorganisms are prevalent.

Airborne also contains a variety of bacteria, viruses, fungi and a number of other microorganisms that can cause harm to human health, forcing fibers to possess antimicrobial properties. Towards this aim, Pan *et al.* [43] fabricated an environmentally friendly polylactic acid (PLA)/chitosan (CS) nanofibrous membrane that could achieve 98.99% filtration of PM and 99.4% and 99.5% bactericidal effect against *E. coli* and *S. aureus*, respectively. However, chitosan alone cannot achieve higher antimicrobial efficiency, so substances with stronger bactericidal properties need to be introduced. For instance, a novel *N*-halamine biopolymer [44], P(ADMH-NVF), was synthesized as the mainstay of antibacterial activity. It was then assembled with PVA/CS to form multilayer membranes (Figs. 2a and b) that exhibited superior mechanical flexibility when subjected to bending, rolling, folding and unfolding (Fig. 2c). The nanofibrous membranes exhibited a filtration efficiency of 99.4% for oil-based aerosols, as well as excellent antimicrobial capability. And a novel rechargeable chlorination-capable nanofibrous membrane was fabricated by functionalizing the nanofibers with dimethylphenol-5,5-dimethylhydantoin (DMDMH) (Figs. 2d-g) [45]. Its inactivation of bacteria exceeded 99.9999% in 3 min and its filtration efficiency of PM exceeded >99.5%. Specifically, the N-R group located between two carbonyl groups of DMDMH could be hydrolyzed into N-H groups, which can easily form an N-Cl bond when exposed to chlorinated solutions, providing strong bactericidal properties, as well as giving the membrane the ability to regenerate Cl by repeated impregnation with chlorinated solutions. In addition, considering the easily dissipated static electricity, Lee *et al.* [46] synthesized a poly(styrene-2-(dimethylamino)ethyl methacrylate-co-acrylonitrile) (ABC)-type terpolymer nanofibrous membrane with bactericidal sulfobetaine zwitterionic groups and cyano groups for PM capture. Its filtration efficiency and pressure drop were hardly affected even after 24 h of treatment with isopropanol vapors. This was due to the dipole-dipole interaction of the polar cyano group with the PM with polar groups, thus allowing a stable PM capture, which was one of the ways to achieve long-lasting high filtration efficiency, apart from the electrostatic electret process. This method of polymerizing functional monomers prior to the polymer masterbatch is more promising than the fiber reprocessing methods described above. From the point of view of process devices and processing costs, the addition of new reactors or soaking tanks and other devices after the fiber production devices is required for the fiber reprocessing method, which increases the cost of devices and the overall process steps, and makes it more difficult to achieve industrialisation. Although the accompanying regeneration process may seem to improve the use of membranes and save costs, this step may not only affect the performance of nanofibrous membranes, but also be limited by the treatment method [47]. Therefore, one-step spinning method

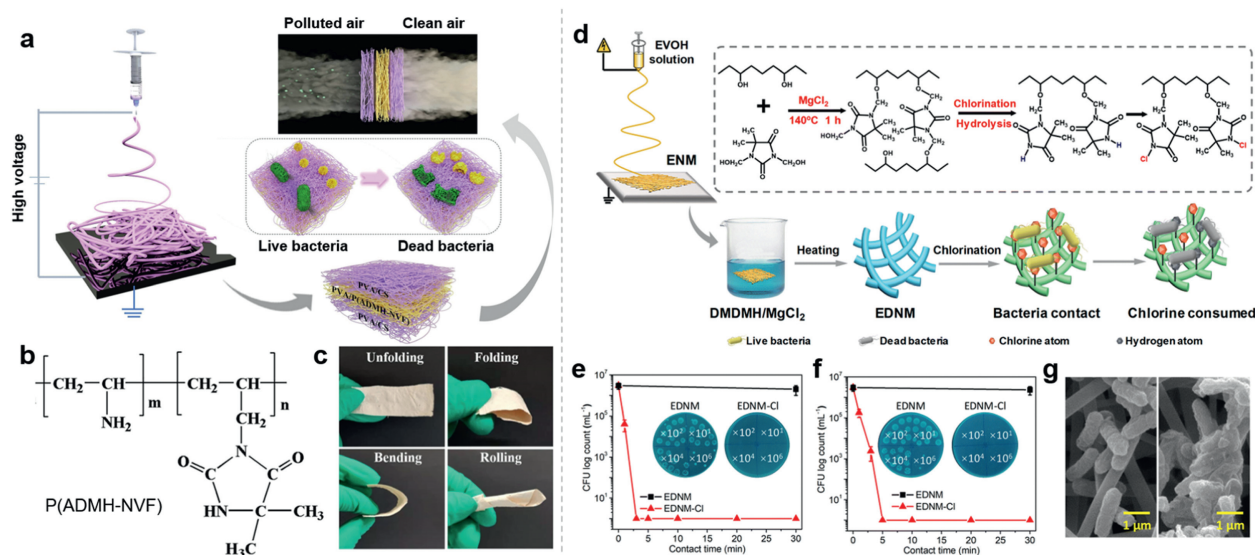


Fig. 2. (a) Schematic diagram of the preparation and application on the air filtration of the antibacterial multilayer membranes. (b) Molecular model and formula of P(ADMH-NVF). (c) Digital photographs of the nanofibrous membrane under different deformations. Reproduced with permission [44]. Copyright 2020, Elsevier. (d) Schematic diagram of the preparation process of Cl-functionalized nanofibrous membranes. (e-g) Bactericidal activity of Cl-functionalized nanofibrous membranes. Reproduced with permission [45]. Copyright 2019, Springer Nature.

or functional monomer polymerization to prepare masterbatch is more conducive to industrial production.

At present, the outbreak of the new crown epidemic has made antiviral fibers the focus of global attention. Some compounds with antiviral properties such as tetrahydropyrimidine (PTHP) were introduced into the nanofibers by an electrospinning process [48]. The prototype mask made from these nanofibrous membranes possessed a 96.9% filtration efficiency and excellent antiviral effect. The authors explained that PTHP disrupted the surface envelope protein or transmembrane envelope protein of the lentivirus, which affected the uncoating of the virus and inhibited the release of viral nucleic acids into the cytoplasm, thus achieving an antiviral effect. Besides, more research should focus on anti-viral fibrous membranes and expand the introduction of some compounds into fibers, such as terpyridine methylammonium chloride [49], marine microbes-derived natural products [50], to reduce the harm caused by airborne viruses. Compared to other antibacterial and antiviral compounds that are simply spun in blends with polymers, organic synthetic compounds possess longer-term applications because they are diverse in design and easier to graft onto polymers. Research should also be devoted to the synthesis of cheap, non-toxic and effective anti-bacterial and anti-viral compounds to enrich anti-viral air filtration nanofibrous membranes.

3.1.2. Natural extracts

Natural herbs have been used in medical fields such as antibacterial, antiviral and anti-inflammatory since ancient times [51,52]. Based on this antibacterial and antiviral properties, natural extracts can be used to produce air filtration fibrous membranes to reduce the risk of pathogenic microorganisms such as COVID-19 [53]. For example, violacein, a naturally antibacterial ingredient cultivated from *Clostridium violatum*, possesses improved biocompatibility. Combining it with PAN nanofiber (VIO-NF) via electrospinning technology as shown in Fig. S3a (Supporting information), it achieved 99.7% and 100% filtration efficiency for PM_{1.0} and PM₁₀ respectively and maintained long-term stability as prototype masks (Fig. S3b in Supporting information), this excellent filtration performance was contributed by the polar groups of PAN [54]. It also rapidly inactivated bacteria and viruses, such as influenza and human coronavirus. The specific antibacterial mechanism was that

the pyrrolic N-H moieties interfere with the bacterial cell membrane, leading to the efflux of endolysate and further disrupting the intracellular osmotic balance, causing bacterial death. *Apocynum venetum* extracts (AVE), extracted from *Apocynum venetum* L., contain gallic acid, hyperoside, catechin, and rutin, which can disrupt the structure of bacterial cell membranes and possess long-lasting antibacterial properties [55]. The prepared PVB/AVE nanofibrous membranes showed antibacterial rates of 99.38% and 98.96% against *S. aureus* and *E. coli*, respectively, and 98.3% of filtration efficiency when the pressure drop was 142 Pa. Blending berberine hydrochloride (BH) extracted from *Coptis chinensis*, *Phellodendron amurense* and berberis root with PVB not only had an air filtration performance of 96.4%, but also showed good antibacterial property for *S. aureus* [56]. Most of these natural extracts are biodegradable, and when combined with equally biodegradable polymers such as polyvinyl alcohol (PVA) [57], polylactic acid (PLA) [58], and polycaprolactone (PCL) [59], the resulting fibrous membranes will exhibit excellent environmental friendliness in addition to the above properties.

In fact, antibacterial herbal extracts have been extensively studied as antibacterial agents for air filtration fibrous membranes due to their low toxicity, high antibacterial activity, mild environmental impact, and low cost [60,61]. However, the antibacterial activity of herbal extracts may be affected or even degraded by natural oxidative processes in practical applications, so their durability remains an issue to be addressed [62,63]. Moreover, the industrialization of such antimicrobials is a challenge for the extraction process of the herbs.

3.2. Inorganic antimicrobial agents-based nanofibrous membranes

3.2.1. Metal ions/nanoparticles

Metal ions/nanoparticles possess a wide range of chemical and physical properties that determine their antibacterial mechanisms. Mⁿ⁺ may be associated with different targets in bacterial cells, including enzymes, membranes and DNA molecules [64]. For example, metal ions or nanoparticles interact with bacterial cell walls and cell membranes through electrostatic attraction, causing damage to them and resulting in imbalance in the transport of substances or even the release of endosol. Or they may enter the

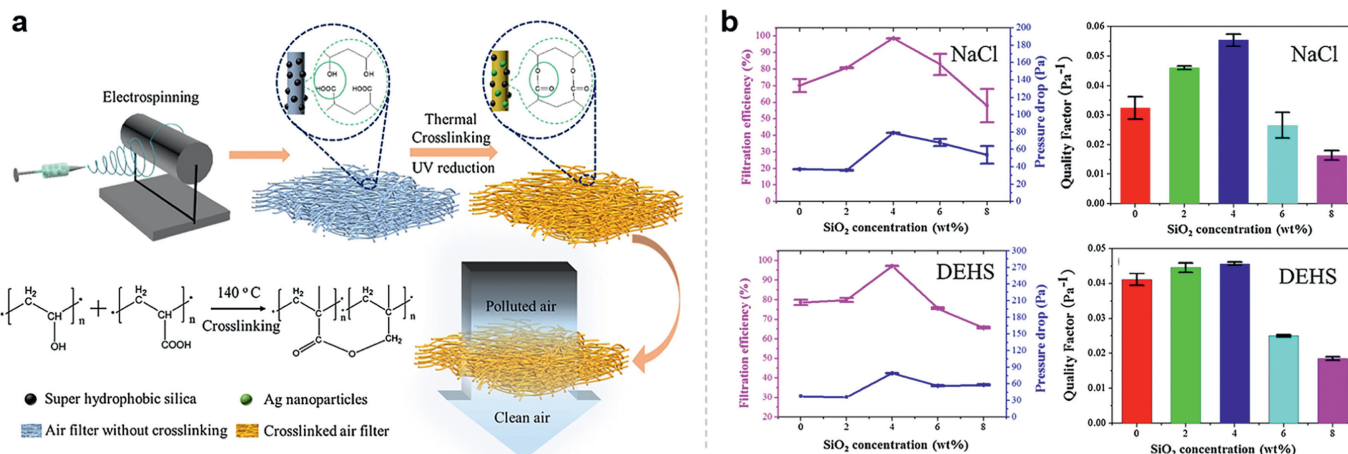


Fig. 3. (a) Schematic diagram of the preparation of silver-doped nanoparticle nanofibrous membranes. Reproduced with permission [80]. Copyright 2018, Elsevier. (b) Filtration efficiency, pressure drop and quality factor of CS-PVA nanofibrous membranes. Reproduced with permission [81]. Copyright 2019, Elsevier.

bacteria directly and interfere with the normal activity of proteins and DNA. Or they can catalyze the production of reactive oxygen species, which can oxidize and damage the internal material of bacteria [65].

More research is on electrospun nanofibers doped with inorganic antimicrobial agents, especially metal ions/nanoparticles, such as Ag NPs, which can be added into a polymer matrix to form fiber membranes *via* electrospinning, which are stable and harmless to human cells [66]. For instance, AgNPs were doped into PAN [67,68], polyamide-6 (PA6) [69], PVA [70,71], polyvinylidene difluoride (PVDF) [72,73], polyarylene sulfide sulfone (PASS) [74], PCL [75], polyimide (PI) [76] or other fibers [77] to obtain composite membranes with antimicrobial property for air filtration. Typically, several nanofibrous membranes consisting of PVDF blended Ag were prepared [78]. The antibacterial rate of all the membranes exceeded 99.5%. In order to improve the hydrophobicity of the membranes and reduce air resistance, an additional Al₂O₃ component was added to the nanofibrous membranes. The resulting composite membranes (PVDF-Ag-Al₂O₃) have increased its filtration efficiency for particulate matter to over 99%, compared to 94% for pure PVDF nanofibrous membrane. And the increase in Al₂O₃ content did not affect the antibacterial properties of the composite membranes.

In addition to Al₂O₃, SiO₂ NPs also possess hydrophobic properties [79]. Huang *et al.* [80] first prepared polyvinyl alcohol/polyacrylic acid (PVA-PAA) nanofibrous membranes by electrospinning technique using water as the solvent, then super hydrophobic silica nanoparticles were embedded in the nanofibers to reduce air resistance, and finally Ag nanoparticles were introduced by UV reduction to confer excellent antibacterial properties to the nanofibrous membranes (Fig. 3a). Spherical Ag NPs of about 5 nm in diameter could be observed on the surface of the nanofibers in transmission electron microscopy (TEM) images, demonstrating the successful reduction of Ag ions. As expected, the filtration efficiency of the nanofibrous membrane exceeded 98% for PM_{2.5} and possessed strong antibacterial properties. Another unique feature of this work was the replacement of dangerous organic solvents with water, thus avoiding any potential toxicity problems caused by organic solvent residues. The next year, they replaced PAA with chitosan to prepare nanofibrous membranes with the same excellent filtration properties and highly effective antibacterial activity [81]. As shown in Fig. 3b, the composite membranes containing 4 wt% SiO₂ NPs possessed the best filtration efficiencies of 98.73% (for NaCl, QF=0.055) and 97.30% (for DEHS, QF=0.047), respectively. Furthermore, the pressure drop of the membranes was not

significantly affected by the content of SiO₂ NPs. To add another reusable property to the nanofibrous membranes, Ag nanoparticles were embedded into porous SiO₂-TiO₂ nanofibers [82]. The composite membranes with adsorbed particles could be regenerated by a simple calcination process. Their filtration performance remained stable over 5 filtration-regeneration cycles and 12 h of prolonged filtration, showing excellent reusability.

The antimicrobial properties of air filtration fibrous membranes are ultimately intended to be highly effective in killing the viruses that cause infection and transmission of respiratory infections. Dong *et al.* [83] reported polyamide-6 electrospun nanofibers anchored with silver nanoparticles through hydrogen-bond. This nanofibrous membrane exhibited high PM_{2.5} filtration efficiency of 99.99% and low pressure drop of 31 Pa, superior antibacterial performance, antiviral property against *Porcine Deltacoronavirus* in particularly and not significant cytotoxicity. Besides, there are some other antiviral studies for air filtration, such as PCL/AgNPs membrane against *E. coli* phage as a double-stranded DNA virus [84] and PAN/PVDF/AgNPs membrane against H1N1 influenza virus [85]. Adding metal nanoparticles is a proven method to impart antiviral properties to nanofibers because they can interact with biologically important entities such as DNA, antigens, antibodies and proteins to disrupt their structure [86]. Based on the studies that have proved that metal nanoparticles are effective against viruses in recent years [87,88], we have reason to believe that the fibers containing silver nanoparticles in the above reports all have antiviral application potential. However, a serious question, extensive use of NPs leads to leakage and accumulation of NPs in the environment (*e.g.*, soil and water), which is one of the most serious threats to the ecology and public health [89,90]. As there is no chemical bond anchoring the metal nanoparticles to the nanofibers, there is a risk of detachment during use, which not only affects the performance of the nanofibers, but also causes concern to the user. Therefore, we believe that although inorganic nanoparticles are still predominant in current practical applications, the future will tend to be organic synthetic compounds as mentioned above.

The antibacterial mode of action of metal nanoparticles can also take advantage of other modalities, such as electrical stimulation and thermal effects. Wang *et al.* [91] prepared a bilayer fibrous membrane consisting of a cotton fibers sterilisation layer modified by magnetron sputtering Ag/Zn coating and a PVDF/PS nanofibrous membrane filtration layer, respectively (Fig. 4a). And in this study, PM interception and air resistance of fibrous membranes were constructed by the FibreGeo module, the FlowDict module and the FilterDict module in the GeoDict software, which was one of the

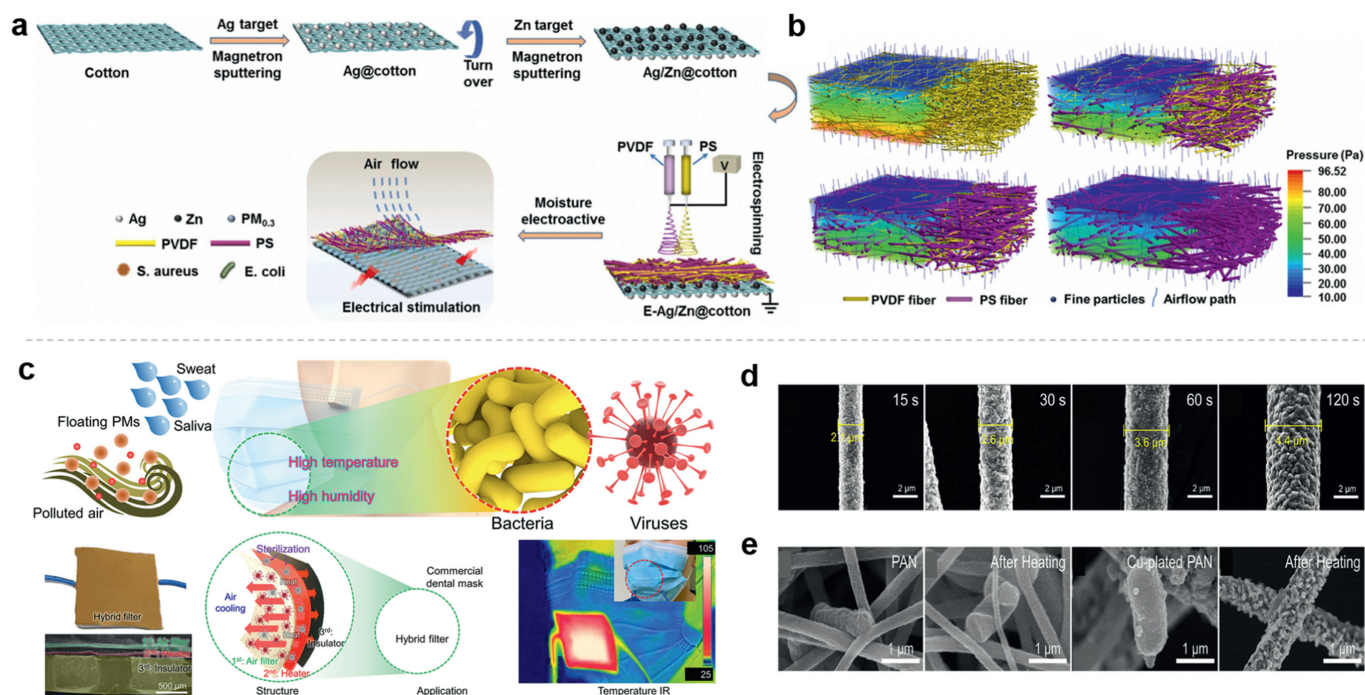


Fig. 4. (a) Schematic diagram of the preparation of a bilayer fibrous filtration membrane. (b) 3D simulation of the airflow distribution and PM capture process of PVDF/PS nanofibrous membranes. Reproduced with permission [91]. Copyright 2022, Elsevier. (c) Schematic of the existing facemask. (d) SEM images of Cu microfibers with different Cu electroplating time of 15, 30, 60 and 120 s. (e) Specimens SEM images of PAN and Cu-plated PAN after incubation for 1 h before and after heating. Reproduced with permission [92]. Copyright 2021, American Chemical Society.

few reports applied to theoretical simulation studies. Among them, the FibreGeo module was responsible for simulating 3D membrane structures based on real structural data, the FlowDict module was responsible for reproducing experimental data on 3D membranes, and the FilterDict module was responsible for the analysis of particle capture processes. In detail, Fig. 4b showed that as the PS nanofiber content increases, the module color tended towards blue, *i.e.* the air resistance became less, which was consistent with the experimental data. NaCl aerosol particles were intercepted by the three-dimensional nanofibers and entered the interior of the membrane. Moreover, the most interesting part of this work was that an electrical stimulation (Zn as the negative electrode and Ag_xO as positive electrode) was generated inside the composite fabric with the benefit of external moisture and thus endowed the fibrous membrane antimicrobial function. This work may provide a new inspiration on designing high-performance fibrous membranes for air filtration.

Since several bacteria and viruses are incapacitated under high temperatures, fibrous membranes with thermal effects, such as electric heating, holds great promise for scenarios where antibacterial and antiviral resistance is required. Yoon *et al.* [92] prepared a multilayer air filtration fibrous membrane consisting of a filtration layer, a heating layer and a thermal insulation layer using the electroplating method (Fig. 4c). As shown in Fig. 4d, the average diameter of Cu microfibers increased from 2.1 μm to 4.4 μm as the electroplating time increased from 10 s to 120 s. The fibrous membrane possessed a filtration efficiency of 95% for PM_{1.0} and could be efficiently sterilized by generating temperatures above 100 °C (Fig. 4e). However, the pressure drop of the fibrous membrane reached 2000 Pa, probably due to being too dense, which was detrimental to practical applications. Therefore, the next focus of this work should be to try to increase the permeability. Killing germs and viruses by thermal effect is a particularly effective solution. However, the power equipment that it needs to be connected to, such as batteries or triboelectric nanogenerators (TENG), is a problem

that must be considered in practical applications, especially for masks. This approach is not possible in most applications, so research in this area should focus on demanding specialized environments rather than bulk commodities.

3.2.2. Metal oxide

Metal oxides are mainly used to generate reactive oxygen species (ROS) for antibacterial, such as titanium dioxide with photocatalysis properties [93,94]. TiO₂ can be doped into polymers to achieve PM filtration and antibacterial functions [95,96]. For instance, Ting *et al.* [97] reported a PAN nanofibrous membrane embedded with commercial TiO₂ that possessed a filtration efficiency of 96.75%. And it exhibited excellent antibacterial activity under 30 min of UV irradiation. Although these studies have also introduced TiO₂ into the antibacterial air filtration electrospun nanofibers, there is still a problem that has to be faced. The prepared nanofibrous membranes must be irradiated with ultraviolet light to generate ROS and possess the ability to sterilize. In the process of practical application, such as wearing a mask, this purpose cannot be achieved.

To solve this problem, nitrogen was doped into commercial TiO₂ using urea as the nitrogen source [24], and the band gap energy of the obtained N-TiO₂ was reduced, allowing it to absorb visible light significantly (Fig. 5a). Nanofibrous membranes containing this visible photocatalytic material achieved 100% bacterial disinfection in only 10 min under natural sunlight. Also, as shown in Figs. 5b-d, masks made from this membrane possessed a stable filtration efficiency of more than 90% even after more than 120 min of continuous wear. This work enabled the mask to continuously generate ROS to fight antibacterial as long as there was sunlight in the process of wearing the mask. Therefore, this mask may be a good alternative for the current mask to address its urgent need under the ongoing COVID-19 infection.

In addition, another metal oxide that has attracted researchers is zinc oxide because it can readily generate reactive oxygen

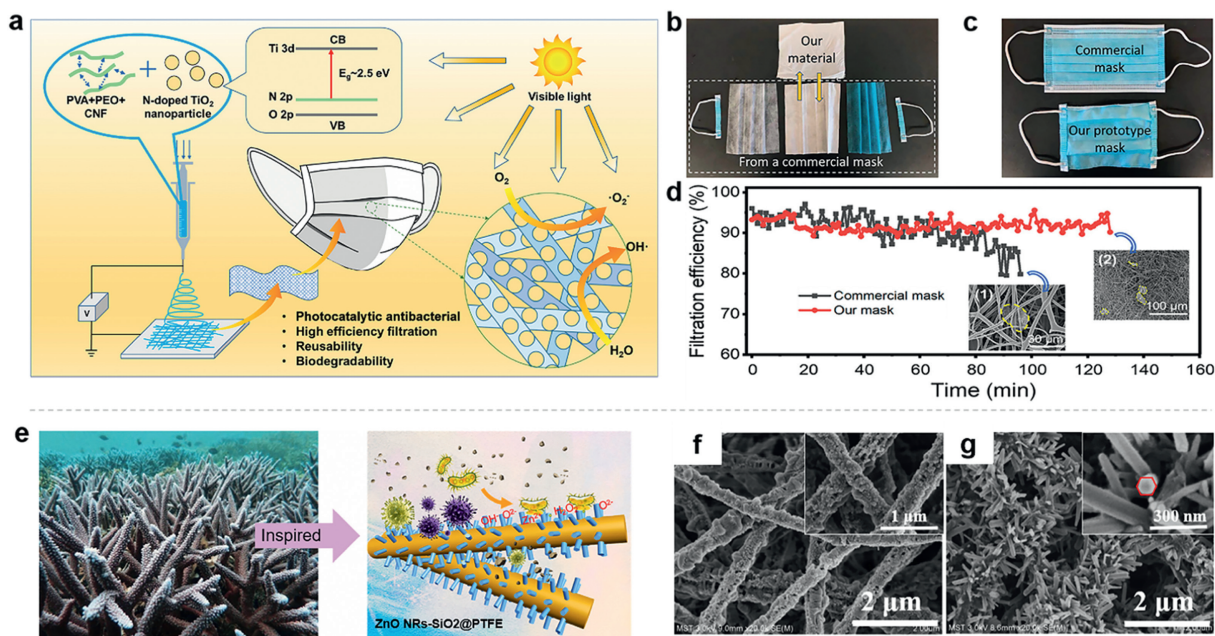


Fig. 5. (a) Schematic diagram of the preparation of a visible photocatalytic disinfection mask. (b, c) Fabrication and photo images of a prototype mask. (d) The filtration efficiency of two masks over time. Reproduced with permission [24]. Copyright 2021, American Chemical Society. (e) Schematic diagram of the preparation of ZnO NRs-SiO₂@PTFE nanofibrous membrane. (f, g) SEM images of membranes after dipping in solutions. Reproduced with permission [101]. Copyright 2022, Elsevier.

species in sunlight [98], and release Zn²⁺ with the inherent antimicrobial nature [99]. For instance, PVA and konjac glucomannan (KGM)-based nanofibrous membranes loaded with ZnO nanoparticles were prepared through green electrospinning and ecofriendly thermal cross-linking [100]. Obtained nanofibrous membranes not only showed 99.99% of filtration efficiency but also showed superior antibacterial activity. And SiO₂@PTFE nanofibers decorated with a tentacle-like ZnO nanorods (NRs) were prepared in a coral-inspired manner (Fig. 5e) [101]. It could be seen from Figs. 5f and g that ZnO nanoparticles were uniformly attached on the nanofiber surface and a continuous and uniform nanorod structure grew on the surface of the nanofibers after treating. The obtained ZnO NRs-SiO₂@PTFE nanofibrous membrane maintained a superior filtration efficiency of 99.99% for PM_{0.3} in 10 cycles and a good antibacterial rate of 99.67% and 99.93% for *E. coli* and *B. subtilis*, respectively. However, this method is limited by the need to reprocess the fibers and the still more demanding *in situ* growth method. The reason for this is that the reprocessing of the fibers not only affects the pressure drop of the membrane, but more importantly it is difficult to achieve in industrial production.

3.2.3. Carbon-based materials

Many researchers have prepared air filtration nanofibrous membranes using nanocarbons with high specific surface area and excellent adsorption capacity [102]. Carbon nanotubes (CNTs) can provide several additional advantages for air filtration [103,104]: (1) The CNTs have little effect on pressure drop due to the smaller diameter, (2) ultrathin CNTs create ultra-small pores to intercept PM, (3) CNTs possess excellent potential for photothermal and electrothermal conversion. These attractive properties make it possible to construct high-performance and self-sterilizing air filtration membranes. Yu *et al.* [103] constructed a multiscale nanostructured nanofiber/carbon nanotube (NF/CNT) network with high filtration efficiency and low resistance through a unique needle-free electrospinning/spraying mesh technique, as shown in Fig. 6a. NF/CNT demonstrated ultra-fast photothermal-driven self-sterilization (>99.986% in 5 min) under 1 sun, and electrothermal-driven self-sterilization in sunless scenes due to their instantaneously

electrothermal properties. More specifically, an extremely high antibacterial efficiency over 99.9999% when applied a small current of 61 mA for 2 min in Figs. 6b and c.

Graphitic carbon nitride (g-C₃N₄), as a sp² hybrid material, is a semiconductor photocatalyst with visible light response [105]. Its band gap energy is 2.7 eV, and the maximum adsorption wavelength is about 460 nm [106]. g-C₃N₄ also has the advantages of strong mechanical properties, large specific surface area, non-toxicity, and easy synthesis and availability, which makes it a revolutionary material in membrane construction [107,108]. The integration of g-C₃N₄ into nanofibers enabled the composite membrane to achieve 99% disinfection efficiency under visible light irradiation for 120 min while maintaining mechanical strength [109]. In addition, it is shown that the ultra-thin surface of graphene oxide (GO) can also weaken or even kill bacteria by disrupting their outer membrane and exerting oxidative pressure on them [110]. Importantly, the unique structure and ionic charge of GO can neutralize the virus by inhibiting the receptor and disrupting the viral structure before it enters the host cell [111]. Therefore, graphene-based materials have demonstrated many excellent properties and potential for many applications [112,113]. However, if some of the problems of practical applications are taken into consideration, then the carbon-based materials mentioned above may be more suited to research in fields such as electrochemistry rather than air filtration.

3.2.4. Other materials

Recently, MXenes, an emerging 2D material, a large family of 2D carbides and nitrides, exhibit unprecedented gas separation performance [114–117]. As one of them, titanium carbide Ti₃C₂T_x, due to its ultra-thin thickness, the modification of nanosheets cannot change the physical properties of the modified nanofibers [118,119]. The groups (O, OH, and F) bound on the surface of MXene nanosheets during chemical layering induced strong interactions with PM and bacteria, resulting in enhanced filtration performance. Furthermore, the antibacterial properties of Ti₃C₂T_x nanosheets and membranes are attributed to the physical effect of the ultra-thin edges that cut through the bacteria and the gener-

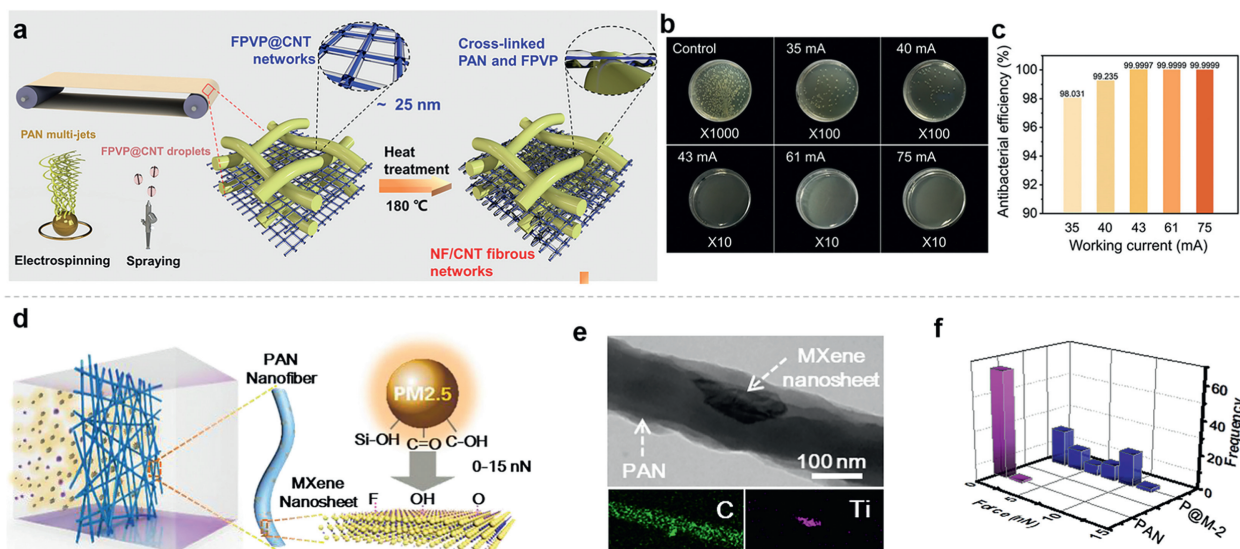


Fig. 6. (a) Schematic diagram of the preparation of NF/CNT fibrous networks. (b, c) Performance of NF/CNT fibrous networks against *E. coli* at different currents for 2 min. Reproduced with permission [103]. Copyright 2021, Wiley-VCH. (d) Mechanism of polar interactions between PM_{2.5} and MXene nanosheets. (e) TEM images of P@M nanofibers and the elemental distribution of C and Ti. (f) Forces between nanofibers and PM_{2.5}. Reproduced with permission [118]. Copyright 2019, Elsevier.

ation of reactive oxygen species (ROS) that chemically oxidize the bacteria [120,121]. In a recent paper by Wang *et al.* [118], Ti₃C₂T_x nanosheets were introduced into PAN nanofibers to fabricate air filtration membranes (Figs. 6d and e). Interestingly, for the first time, they examined the interaction force between the nanofiber surface and PM_{2.5} by force-distance based atomic force microscopy (AFM). As shown in Fig. 6f, the force range between PAN nanofibers and PM_{2.5} was 0–5 nN, while the force range between fabricated nanofibers and PM_{2.5} becomes 0–15 nN, which led to better adsorption and higher filtration performance. This test clearly and intuitively showed the reason for the excellent performance.

3.3. Metal organic frameworks-based nanofibrous membranes

Metal–organic frameworks (MOFs), constructed from metal cations or clusters and organic ligands, are a new class of crystalline hybrid materials with reticular topology and possess the fascinating characteristics of high surface areas, predesigned pore size, homogeneously dispersed active sites [122]. They can be used to create MOFilters with high crystallinity, ultra-high surface area, rich chemical functionality and structural tailorability, suitable for a broad range of applications [123]. On the one hand, it possesses excellent antibacterial activity as follows [124]: (1) MOFs kill bacteria by slow release of central metal ions and organic ligands. (2) The metal nodes in the structure of MOFs act as active sites and kill bacteria through the direct interaction between the MOFs and bacteria. (3) MOFs serve as carriers and kill the bacteria through the release of metal ions and loaded small molecule drugs. (4) Photosensitive MOFs kill bacteria through photothermal or photodynamic forces. (5) MOFs kill bacteria through chemodynamic to kill bacteria. (6) MOFs kill bacteria based on gas therapy. (7) MOFs kill bacteria by mimicking enzymes. (8) MOFs kill bacteria through the synergistic effect of multiple active compounds. On the other hand, the unbalanced metal ions and defects in MOFs provide a positive charge that enhances the polar interactions between MOFs and PM, which makes MOFs potentially better and more stable filtration properties [125,126]. For the first time, Wang *et al.* [127] have explored the interactions between MOFs and PM through four unique MOFilters (Figs. 7a and b): (1) Binding to the open metal sites on MOFs, (2) interacting with the functional groups on MOFs and/or polymers, (3) electrostatic interactions with MOF nanocrystals.

Based on the above antibacterial and filtration properties, Wang *et al.* [128] investigated the performance of some MOFilters using the photodynamic bactericidal properties of MOF. Specifically, the MOFilter made by growing ZIF-8 nanoparticles on non-woven fibers possessed a killing efficiency of over 99.99% for airborne bacteria within 30 min of simulated solar irradiation and the filtration efficiency of 97% for PM. In addition to photocatalysis, the antimicrobial mechanism of MOFilters is dominated by action of the organic ligands or central ions. For instance, the active composite UiO-PQDMAEMA were constructed by grafting a layer of antibacterial polymeric quaternary ammonium compound (QAC), that was, poly[2-(dimethyldecylammonium)ethyl methacrylate] (PQDMAEMA), onto the surface of UiO-66-NH₂ [129]. The nanofibrous membranes prepared by electrospinning of it mixed with PAN were effective in killing airborne *S. epidermidis* and *E. coli*. This effect was mainly due to the strong electrostatic interaction between the positively charged nitrogen of the quaternary ammonium salts and the cell walls and cell membranes of the bacteria, resulting in severe damage to the cell membranes. The filtration efficiency was comparable to that of commercial N95 respirators at around 95% for particles of 80 nm. In order to play the role of the central metal ion, growing ZIF-67 on nanofibers to provide a source of the bactericidal ion Co²⁺, while forming enokitake-like and bead-on-string-like structures (Fig. 7c) [130]. This unique structure effectively reduced the pore size of the fibrous membranes and increased the exposed surface of the MOFs, resulting in a filtration efficiency of 97.5% for PM_{2.5}. The SEM image of the fibrous membrane after 1 h of filtration was shown in Fig. 7d, showing a large amount of PM was captured and adsorbed by the fibers. Analogously, there are many types of MOFs that can also be prepared as MOFilters to be applied in air filtration applications, such as Cu-MOF [131,132], Ag-MOF [133,134], Zn-MOF [135–137] and Al-MOF [138].

Although MOFilters with promising filtration properties show great potential for air purification, there are some challenges for MOFilter preparation [139]: (1) Good adhesion and compatibility of MOF with the substrate, *i.e.*, the problem of easy shedding of MOF and service life of MOFilters, (2) scalability of current methods of producing MOF and MOF filters, *i.e.*, the problem of their industrialization prospects, (3) complex preparation steps, expensive organic ligands and low recyclability, *i.e.*, the issue of cost, (4)

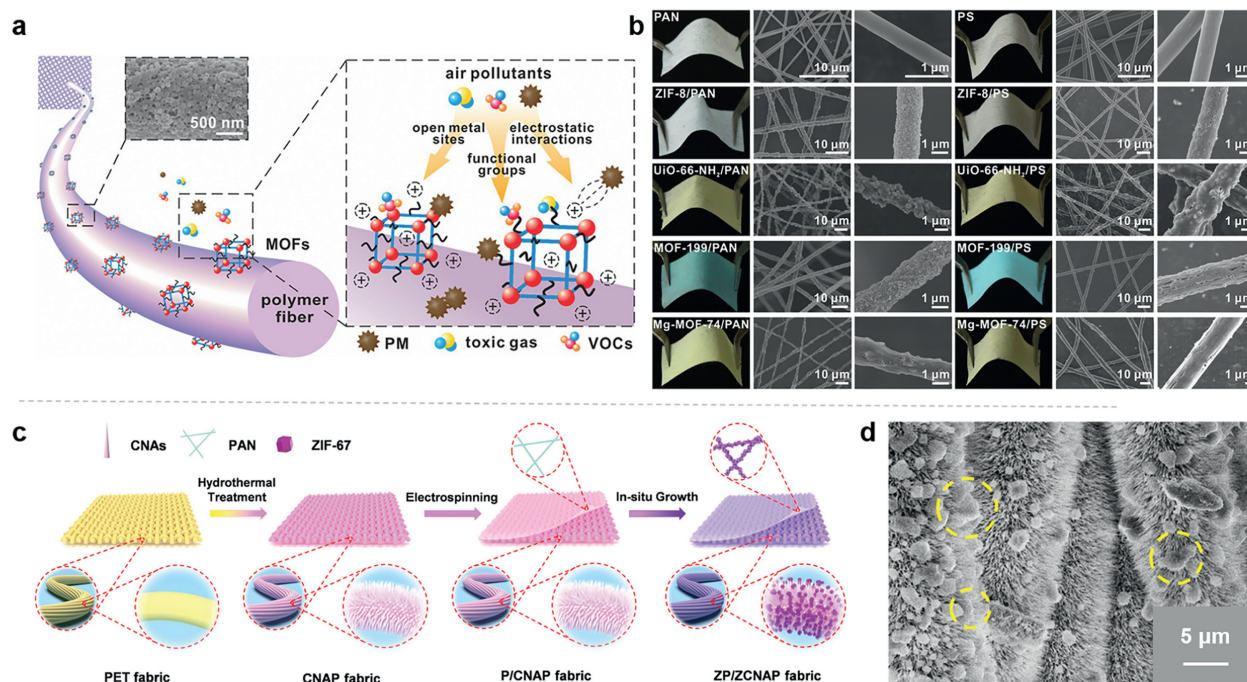


Fig. 7. (a) Air pollutants capture mechanism of the MOFilter. (b) Photographs and SEM images of the MOFilters supported on nonwoven fabrics. Reproduced with permission [127]. Copyright 2016, American Chemical Society. (c) Schematic diagram of the preparation of ZP/ZCNAP. (d) The surface morphology of ZP/ZCNAP3 after filtering. Reproduced with permission [130]. Copyright 2019, Elsevier.

the lack of a comprehensive understanding of the pore system, *i.e.*, the issue of theoretical guidance. Therefore, MOF-based filtration materials may also be limited to theoretical research in the future, and it is difficult to move towards practice.

4. Summary and outlook

Recently, electrospun nanofibrous membranes have been considered to be a new generation of air filtration materials due to their rich porous structure, finer fiber diameters, good internal connectivity, and the ability to easily incorporate active chemicals for antimicrobial function. They possess promising applications in a wide range of air filtration uses including, but not limited to, personal protective equipment such as masks, air filters for ventilation systems especially in demanding locations such as medical and sterile clean rooms, and filters for medical equipment such as ventilators. To provide guidance for studies of high-performance antimicrobial electrospun nanofibrous membranes, this review mainly discussed antimicrobial mechanisms of nanofibrous membranes for air filtration and PM capture mechanisms of nanofibers, and introduced various types of electrospun nanofibrous membranes with different antimicrobial agents for efficient air filtration, including organic antimicrobial agents, inorganic antimicrobial agents and metal organic frameworks. Moreover, performances of different electrospun nanofibrous membranes with antimicrobial efficacy and air filtration function are summarized in Table S1 (Supporting information).

Future work should be directed toward the manufacture of environment-friendly and low-cost electrospun nanofibrous membranes with antimicrobial efficacy and efficient air filtration function. To achieve a balance between the antimicrobial activity, filtration efficiency and the pressure drop through precisely controlling the functional group, arrangement and porosity of nanofibers and the composition of antimicrobial ingredients. Specifically, many of

the challenges that exist in practical applications should be addressed:

- (1) Achieving lower cost and biofriendliness of antimicrobial agents under the premise of excellent antimicrobial activity is an urgent problem to be solved. It is also important that the antimicrobial agents are effectively and safely incorporated into the polymer, preferably prior to the preparation of the polymer masterbatch, rather than post-treating the fibers.
- (2) With the pursuit of efficiency improvement, the pressure drop is often difficult to meet the standard requirements. Therefore, the construction of high-efficiency nanofibrous membranes with low pressure drop to solve application problems is what is needed for future development.
- (3) Theoretical studies on the constitutive relationship of filtration materials are still relatively scarce. A model should be developed to systematically express the filtration efficiency and pressure drop of nanofibers with different structures, thus providing guidance for the structural design of nanofibrous membranes.
- (4) Many electrospinning solutions for nanofibers are dissolved by using toxic solvents, or the polymers themselves contain toxic monomers, which can have numerous adverse effects on the environment and human health. Safer materials and methods of preparation and production still need to be developed.
- (5) The preparation of many electrospun nanofibrous membranes and the associated post-treatment processes are still at the laboratory level, and further research is needed to achieve industrial production.

Overall, the ultimate goal is to push electrospun nanofibrous membranes from theory to practice, from laboratory preparation to industrial production and from simple models to bulk commodities.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.ccl.2024.109503.

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