



Electrospun Nanofibrous Membranes for Air Filtration: A Critical Review

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Abstract: Air filtration is an urgent global need because, in many countries and regions, the high concentration of inhalable suspended particles in the air is causing irreversible damage to human health. The use of nanofibrous membranes can help to reduce airborne particulate matter because of their large surface area, extremely porous structure, and adjustable pore size. However, despite their unique properties, the main drawbacks of nanofibre membranes are their poor mechanical properties. This review focuses on nanofibrous membranes prepared by electrospinning, a versatile technique in which the process parameters allow control of the morphology and dimensional characteristics of the nanofibres. Recent literature on air filtration is reviewed, focusing on the performance of materials such as pure or mixed polymers, organic–inorganic composites, and 'green' materials in the form of nanofibrous membranes. Finally, the recently proposed layered structures for nanofibre-based air filters are reviewed, offering the latest and most innovative solutions.

Keywords: air filtration; electrospinning; nanofibres; filtration efficiency; nanofibrous membranes

1. Introduction

Air pollution is the presence of physical, chemical, and biological pollutants in the Earth's atmosphere that alter the natural characteristics of the atmosphere. These pollutants are usually not present in the normal composition of air or are present but at a low concentration. In recent decades, air pollution has become an urgent problem because of its harmful effects on the environment and human health. For this reason, it is necessary to adopt effective means of removing pollutants from the air.

Various air purification systems have been developed with different removal efficiencies. For example, cyclones, scrubbers, sedimentation tanks and systems based on electrostatic precipitation are highly efficient in removing particles larger than 10 µm but may be ineffective for smaller particles [1]. A suitable alternative is a filtration using porous filters or membranes. For example, high-efficiency particulate air (HEPA) filters can remove 99.97% of airborne carbon particles as small as 0.3 μ m [2]. Although membrane filtration is considered to be an effective method of protecting against airborne pollutants, filters made of fibres and micron-sized pores have some drawbacks in terms of efficiency. To date, researchers and manufacturers have put a lot of effort into researching advanced materials and new technologies to build highly efficient, low-cost, and environmentally sustainable filters. One effective solution is the use of nanoscale fibres with diameters of less than 0.5 µm, also known as nanofibres. A suitable technique for producing nanofibres is electrospinning, which allows nanofibre diameters to be engineered in a wide range from 40 to 2000 nm by a suitable combination of polymers and solvents. Filters made from electrospun nanofibres can have outstanding properties, such as a high surface-to-volume ratio, controllable morphology and connectivity, and a low-pressure drop [3].

The interest of the scientific community in the electrospinning technique has increased significantly in recent years because of its many advantages, such as the versatility of electrospinnable materials, the cost-effectiveness of the equipment and the possibility of



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Copyright: © 2023 by the author. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). scaling up. Considering only publications with "nanofibres + electrospinning + filtration" as keywords, the number of articles increased from a few dozen to over two thousand within a few years (Figure 1).



Figure 1. Increment of scientific publications related to electrospun nanofibres for filtration (results of research on Scopus).

After considering the nature and characteristics of the physical pollutants, this review focuses on (i) the filtration properties to be assessed, (ii) the technology used to produce nanofibres and their operating principle, and (iii) the elements that influence the effectiveness of electrospun membranes for air filtration, namely, the process parameters, materials used, and the macroscopic structure of the membranes.

The membrane examples discussed in this manuscript, mainly related to materials and macroscopic structures, show how the typical problems of nanofibrous membranes caused by their poor mechanical properties can be solved with significant results. To this end, many of the articles published in recent years have been read critically. Those that were considered the most interesting for the reflections they inspired have been cited in this review. It must be mentioned that it is not possible to draw clear conclusions since the vast literature on the subject reports the use of a wide variety of materials and sometimes contradictory results. However, trends and some general suggestions for future research can be identified.

2. Nature and Sources of Physical Pollutants

Air is mainly composed of gases, but it also contains a very large number of particles of various sizes and shapes, commonly known as airborne particles. These particles are produced both by natural processes, such as mineral soil dust, sea spray, dust from volcanic eruptions and forest fires, and by anthropogenic sources associated with human activities [4]. The quantity of anthropogenic emissions is very large, and the main sources are combustion processes in the industry, transport, and biomass burning in households. Many of the substances released into the atmosphere are highly reactive and can form secondary pollutants. Primary and secondary particles released into the atmosphere are then transported up to thousands of kilometres from their sources [5]. During atmospheric transport, the particles may be subjected to various chemical and physical processes that change their shape, size, and composition, and are ultimately deposited on the ground. Some substances in the atmosphere, commonly known as pollutants, cause damage not only to human health but also to the environment and cultural heritage sites (buildings, monuments, and materials) (Figure 2). Among the substances considered pollutants, airborne particulate matter (PM) is particularly detrimental to the quality of life. PM is a collection of solid/liquid particles suspended in the atmosphere, ranging in size from 0.001 to 20 µm, and consisting of various chemical constituents, including some heavy metals

such as arsenic, cadmium, mercury, and nickel [6]. WHO [7] has provided a definition and classification of PM according to the aerodynamic diameter of the particles (Figure 3).



Emissions of particulate matters and pollutant agents





Figure 3. Classification and main composition of PM.

PM is known to affect the environment and human health, and as an indirect consequence, the historical development of the global economy [8]. In addition, PM has direct and indirect effects on climate. PM contributes to the acidification and eutrophication of terrestrial and aquatic environments. It can also carry pathogenic bacteria, which can remain suspended in the atmosphere for long periods of time [9]. In particular, PM2.5, due to its smaller particle size and larger surface area, could enter the respiratory and circulatory systems with human breathing and cause respiratory and cardiovascular diseases. [10]. Unfortunately, air pollution does not only occur outdoors but also indoors in homes, offices, and schools [11]. WHO estimates that in 2012, there were 3.7 million deaths worldwide from urban and rural sources, and 4.3 million deaths related to indoor air pollution. Measures to reduce short-lived climate pollutants (SLCPs), particularly black carbon, could save around 2.4 million lives a year by 2030 and reduce global warming by around half a degree Celsius by 2050 [12]. All of these considerations should enable us to understand the importance of limiting the presence of PM in the atmosphere or reducing the risk of exposure by protecting against PM.

A proportion of PM consists of microplastics (MPs), defined as fine plastic particles that are less than 5 mm in size. As PM, MPs can induce various toxicities in organisms and

have therefore attracted increasing attention since their first occurrence was detected in the aquatic environment [13]. Based on their structural characteristics, microplastics can be categorised as fibres, foams, pellets, films, and fragments [14].

Compared to water and soil environments, relatively little is known about MPs in the atmosphere [15]. However, in urban atmospheres, a high proportion (>90%) of airborne MPs have been found to consist of fibrous microplastics (FMPs) [16–18]. FMPs can be categorised as natural fibres, semi-synthetic fibres, and synthetic fibres, depending on the original materials used and the production process. Essentially, they are filamentous plastic pieces with a length of 0.1 mm to \leq 5 mm and a width of at least 1.5 orders of magnitude smaller [19]. The sources of FMPs are fabrics, clothing, carpets, upholstery, and ropes that shed short strands through use, washing, tearing, and rubbing. A recent study showed that the concentration of FMPs is higher indoors than outdoors, simply because of their source [20]. In addition, it has been found that air conditioning systems, which are widely used to regulate room temperature and recirculate mostly indoor air, can act as both a sink and a source of FMPs [21].

Particular attention has been paid to the risk of inhalation of FMP from surgical masks, following their intensive use as a means of protection against the SARS-CoV-2 virus [22–24]. Disposable plastic face masks consist of two outer layers of microfibres and an inner layer of nanofibres. During use, micro- and nano-sized particles may be released, mainly in plastic fibres and silicate grains [24]. Ma et al. [25] detected irregularly shaped plastic debris in leachate and found that one piece of the mask can release up to $1.6-3.8 \times 10^9$ nanoplastics (size < 1 µm) and $1.3-4.4 \times 10^3$ microplastics (size 1–600 µm). In addition, disposable face masks may be a potential source of human exposure to chemicals, given the large number of additives and some treatments used in their manufacture [24].

3. Air Filtration: Definitions

Filtration involves separating some solid substances from a fluid as it passes through a porous medium or filter. It is important that the material removed from the fluid does not form a cake on the surface of the porous medium, as the high-pressure drop across the filter would end the life of the filter in a short time. In general, a fibrous filter consists of loosely packed fibres, where the distance between the fibres is large compared to the size of the particles. The space between the fibres allows the air or liquid to flow while blocking the particles it contains. To evaluate the performance of filters, filtration efficiency, particle penetration, and quality factor are the most commonly used parameters. Filtration efficiency, η , defines the ability of a filter to remove airborne particles and is measured as

$$\eta = 1 - \frac{C_a}{C_b}$$

where C_a is the average concentration of particles downstream of the filter and C_b is the concentration upstream of the filter. Filtration efficiency is dependent on the filter operating conditions, filter life, and filter material. In particular, the factors that affect particle filtration efficiency are fluid velocity through the filter media, particle size, fibre diameter, dust loading on the filter, and thickness of the filter media. Typically, there is a particle size at which filters exhibit minimum efficiency, usually around 300 nm, called the most penetrating particle size (MPPS). In fact, particles larger than 300 nm are intercepted by impact or diffusion, whereas those smaller than 300 nm are more likely to be blocked by diffusion (Figure 4). For this reason, standard tests of the collection efficiency of high-efficiency filters use 300 nm particles, as this size is very close to the minimum efficiency point.



Figure 4. Filtering efficiency for individual mechanisms and total efficiency. Reproduced from [26] with permission of Elsevier.

Particle penetration, *P*, is in a positive correlation with the fibre diameter and in a negative correlation with the filter thickness and is defined as the fraction of particles that penetrate the filter. It is expressed as

$$P = 1 - \eta$$

Another important parameter characterising filters is the quality factor (*QF*). The pressure drop (ΔP), defined as the difference between the upstream and downstream pressures, is often combined into the *QF* parameter by the following relationship:

$$QF = \frac{\ln(1/P)}{n\Delta P}$$

where *n* is the number of layers of the same filter [27]. A filter with a high value of filtration efficiency and a low value of pressure drop has a quality factor higher than 0.01 Pa^{-1} [28].

4. Use of Nanofibres for Air Filtration

Air purification systems commonly used to remove pollutants have different efficiencies depending on particle size. Cyclones, scrubbers, and sedimentation tanks are highly efficient in removing PM10 but can be ineffective for particles smaller than 10 μ m. By using nanofibre filters, the filtration efficiency is increased due to the following two factors: (i) the slip-flow effect, which produces low airflow resistance, especially when the nanofibres are less than 500 nm in diameter [29]; (ii) the larger specific surface area of nanofibres compared to that of microfibres [30].

Nanofibrous air filters can be produced using three methods: spunbonding, meltblown, and electrospinning. The first two are widely used as industrial processes and have been heavily used in recent years due to the intensive production and use of face masks and surgical masks against the SARS-CoV-2 virus. The electrospinning technique allows the design of nanofibre diameters in a wide range from 40 to 2000 nm using a suitable combination of polymers and solvents. Electrospun nanofibre filters can have excellent properties, such as high surface-to-volume ratio, controllable morphology and connectivity, and low-pressure drop. These characteristics make them attractive for achieving excellent PM2.5 filtration performance.

The electrospinning process allows the formation of nanofibres by starting with a charged jet of polymer solution (e.g., see [31,32]). A classical electrospinning apparatus consists of three main elements: (i) a power supply that provides high voltage, (ii) an electrode that acts as a collector, and (iii) a syringe that contains the polymer solution and acts as a spinneret (Figure 5). The whole system can be arranged vertically or horizontally.

Essentially, a high voltage, typically in the range of 5 to 40 kV, is applied between the collector and the syringe tip, imparting a charge to the polymer solution as it is pumped out of the syringe. During the formation of the droplet at the tip of the syringe, charges accumulate on the surface of the solution, resulting in two forces: electrostatic repulsion and surface tension. At an appropriate voltage, the repulsive force overcomes the surface tension, forming a Taylor cone extending from the syringe needle tip [33–35]. In this way, the solution moves towards the collector in the form of a long and thin filament. As it moves towards the collector, most of the solvent evaporates, producing solid fibres with diameters ranging from a few nanometres to a few micrometres.





The morphology and dimension of electrospun fibres depend on several parameters, the most important of which are processing conditions, solution properties, and environmental conditions. Processing conditions include (in order of influence) applied voltage, the distance between the needle tip and collector, flow rate, needle diameter, and the material and shape of the collector electrode. Among the solution properties, concentration, viscosity, surface tension, conductivity, and the molecular weight of the polymer and the solvent have to be chosen appropriately and carefully, as they particularly influence the quality of the electrospun nanofibres. Finally, humidity and temperature are the environmental parameters that have a significant impact on the electrospinning process. Table 1 shows the main process parameters and their effects on the morphology of the electrospun nanofibres.

Table 1. Main f	factors affecting	the morphol	logy of electi	cospun nanofibres.
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Process Parameters	Effects on the Nanofibrous Membrane	Ref.
Applied electric field	An increase in the applied electric field results in thinner nanofibres due to the increase in electrostatic charge repulsion within the polymer jet.	[30]
Tip-collector distance	When the distance between the tip and the collector is increased, the flight time of the jet is increased as well as the time for the evaporation of the solvent. This reduces bead formation.	[31]
Viscosity of polymeric solutions, molecular weight, and polymer chain	Solution viscosity is related to polymer concentration and polymer chain entanglement. Fibre morphology is affected by these parameters. Uniform, bead-free fibres with larger diameters can be obtained at high polymer concentrations in the electrospinnable solution.	[32]
Flux rate	High flow rates produce beaded fibres due to the greater amount of solution that has to lose solvent during flight time.	[32]
Ambient temperature and humidity	Thinner fibres are produced as the temperature rises because the solvent evaporation rate increases. Humidity has a marked effect on the solidification process and therefore on fibre diameter.	[36]

It is worth noting that although this method appears complex because it depends on many parameters, there are significant advantages to its use: it is a one-step process that produces a network of 3D nanofibres; it allows the production of thin fibres with small diameters down to tens of nanometres; it allows the processing of a wide variety of synthetic and natural polymers, even in combination, or containing nanometre-sized inorganic particles; it is cost-effective compared to fibre extruders operating at high temperatures, and has the potential for scale-up production. In addition, electrospun nanofibres can be collected to be aligned or non-aligned. Li et al. [37] report a recent and comprehensive review of the latest advances in the development of electrospinning, including challenges and perspectives in the use of this advanced technique to produce novel nanofibres with performance in several demanding areas.

Although the electrospinning technique undoubtedly has many advantages, the main negative aspect should not be overlooked, which is the large variability of the results obtained, corresponding to a small variation in the process parameters. This obviously has an impact on the quality of the final product, and in the case of filtration, on the performance of the electrospun membranes.

There are many factors that influence the performance of an electrospun nanofibre membrane. In addition to the operating parameters used in the electrospinning process, the materials, the structure of the nanofibres, and the configuration of the filtration system are important (Figure 6). In the next part, some interesting examples of filter systems in terms of materials and structure will be considered.



Figure 6. Factors affecting the filtration efficiency in electrospun nanofibrous filters.

5. Effects of the Electrospinning Process Parameters on the Filtration Effectiveness

An efficient fibrous filter must have the thinnest possible fibres and the smallest possible pore size. Thin, small-diameter fibres allow for rapid removal of PM and the increased spatial density allows for increased filtration efficiency of the filter media. In addition, lower porosity, defined as the ratio of pore volume to total filter volume, can increase the flow resistance [29]. In this respect, electrospun nanofibre filters have great potential, provided that the characteristics of the nanofibres are such that an optimum balance between collection efficiency and pressure drop is achieved.

The influence of electrospinning parameters on the morphological properties of electrospun nanofibres has been extensively reported in the literature (e.g., see [32,38]). For filtration, it is important to correlate the fibre properties with the performance of the fibre filter. A study on the relationship between the structure of electrospun membranes and their air filtration performance in terms of filtration efficiency, pressure drop, and quality factor was carried out in [39,40]. More fibre collection methods are used, including rotary and static collectors. In this way, different fibre size distributions are obtained by varying the fibre orientation and interconnectivity. Therefore, filters with different pore sizes and morphologies are realised and then tested for the filtration of standard airborne KCl particles. The overall performance is correlated with the fibre morphology and the highest filtration efficiency (99.78%) for 300–500 nm particles is obtained when the nanofibres are larger and more uniformly distributed [39]. In addition, the performance of membranes with different degrees of fibre alignment obtained by varying the collection conditions was

investigated. Increasing the fibre orientation improved the overall membrane filtration performance [40].

Similarly, the thickness of the nanofibre filter is a critical parameter for filtration efficiency. To investigate the relationship between filter thickness and filtration efficiency, Naragund et al. [41] prepared polyacrylonitrile (PAN) electrospun membranes with different thicknesses and porosities by varying the electrospinning times. The apparent porosity of the membrane increases with the increasing thickness/process time of the membranes. This is attributed to charge accumulation on the fibres, which causes an inter-fibre repulsion. The resulting higher porosity creates a cushioning effect, especially in thicker membranes. For variable-thickness membranes, the air permeability is higher at lower thicknesses due to a less tortuous path for the air to follow. Clearly, the filtration efficiency and air permeability increase with decreasing thickness when the inter-fibre spacing matches the most penetrable particle sizes. When used as a filter mask, lower pressure drops are desirable to facilitate breathing with minimal effort. Therefore, electrospun PAN nanofibre membranes with low thickness could be suitable for air filtration applications such as face masks.

6. Effects of the Electrospun Material on the Filtration Effectiveness

Another very important factor in the efficiency of nanofibre filters is the type of electrospun material, which can be a single polymer, a blend of polymers, or a nanocomposite consisting of nanofillers embedded in the molten polymer.

Selectivity towards a specific pollutant or biological agent can be imparted to the nanofibre mat after the electrospinning process by means of functionalisation. The following is a review of the recent literature on the performance of various electrospun materials. The classes of materials considered are (i) high-performance and newly designed organic (or polymeric) materials, (ii) interesting organic–inorganic combinations, and (iii) electrospinnable green materials.

6.1. Polymer Nanofibres

To date, several recent studies have described the filtration performance of membranes based on electrospun nanofibres of numerous polymers, such as PAN [41,42], polyvinyl alcohol (PVA) [43], polyethylene terephthalate (PET) [44], polyamide [45,46], polyurethane (PU) [47], polysulphone (PSU) [48], polyvinylpyrrolidone (PVP) [49], polystyrene (PS) [50], polyvinylidene fluoride (PVDF) [51], poly (methyl methacrylate) (PMMA) [52], and polylactic acid (PLA) [53]. Some examples of the performance of nanofibrous electrospun membranes are provided in Table 2.

Polymer	η (%)	ΔP (Pa)	QF (Pa ⁻¹)	Ref.
PAN	99.99	110	0.100	[54]
PAN	99.99	92	0.100	[55]
PAN	99.71	40	-	[41]
PVC	94.33	154	0.19	[56]
PA-56	99.995	111	0.891	[57]
PVDF	99.99	93	0110	[58]
PLA	98.56	29	0.140	[59]
TiO ₂ /PVA	97.8	198	-	[60]
Fe ₃ O ₄ /PVDF	99.95	58	0.130	[61]
GO */PI/PAN	99.5	92	0.058	[62]
PSU/PAN/PA-6	99.992	118	0.8	[63]

 Table 2. Air filtration performance of nanofibrous electrospun membranes.

* GO = graphene oxide.

Unfortunately, single polymer-based air filtration membranes often do not have high filtration efficiency or lack good mechanical properties, ultra-lightness, and flexibility, which are key aspects of membranes. However, there are some successful examples.

Recently, Farhangian et al. [56] demonstrated that electrospun nanofibres manufactured from PVC have superior filtration performance compared to commercial PVC filters, in terms of higher filtration efficiency and lower pressure drop. They tested electrospun PVC membranes by sampling airborne crystalline silica, which is classified as a class 1 carcinogen by the International Agency for Research on Cancer (IARC) (e.g., see [64]). It is worth noting that silica dust is of great concern to industrial hygienists because the risk of exposure to silica dust is present in many workplaces, for example in the construction industry, where concrete or brick handling activities are carried out. Farhangian found that the electrospun PVC nanofibre filter had, on average, a higher efficiency for $0.3-10 \mu m$ silica particles and a lower pressure drop compared to the commercial PVC filter for silica particle concentrations ranging from 0.5 to 10 times the threshold limit values (TLV) for crystalline silica exposure (as defined by the Centers for Disease Control and Prevention [65]).

In high-temperature industrial processes, the removal of exhaust gases is more complex and difficult than the removal of dust particles at low temperatures. In these cases, the filter materials must be able to withstand both high temperatures and chemical corrosion. Conventional high-performance nonwoven filters made from micron-sized fibres of polyphenylene sulphide (PPS), polytetrafluoroethylene (PTFE), or glass are inefficient at capturing and adsorbing fine particles due to their inherently large inter-fibre spaces. If such filters were made of nanofibres, they would be more efficient. Unfortunately, it is difficult to obtain homogeneous solutions suitable for electrospinning using PSS and PTFE. An effective alternative may be the use of poly(arylene sulphide sulfone) (PASS), a new type of high-performance engineering polymer structurally modified from PPS [66]. Compared to PPS, PASS has better mechanical strength and thermal stability, a higher glass transition temperature, and a higher thermal deformation temperature. It is soluble in tetrahydrofuran (THF), N-N-dimethylformamide (DMF), and other solvents; unfortunately, it is quite aggressive but can be made into nanofibres by electrospinning [67]. PASS nanofibre filters are made by optimising the size and pore size of the nanofibres by varying the concentration of the solution. These filters have high efficiency in removing particulate matter (99.98%), low-pressure drop (68 Pa), and high-quality factor QF (0.125 Pa^{-1}). In addition, after oxidation treatment by immersion in acetic acid, the PASS nanofibrous membranes are transformed into O-PASS nanofibrous membranes, which have better mechanical properties and thermal stability than PASS nanofibrous membranes.

For filtration efficiency, not only are the fibre diameter and porosity of the nanofibrous membrane significant but also the chemical affinity of the membrane material with the substance to be removed. In [68], it is demonstrated that nanofibrous membranes based on polyamide-6 (PA6) and PVA have different filtration efficiencies compared to cigarette smoke. By comparing the FT-IR spectra acquired before and after 1 min of exposure, PA 6 membranes are shown to be effective at filtering carboxylic acids and basic alcohols, whereas PVA membranes are more efficient at filtering alkanes.

6.2. Organic-Inorganic Nanofibres

Recently, the synthesis of organic–inorganic hybrid materials, also known as nanocomposites, has attracted great interest in material science. Composite nanofibres, obtained by mixing nanometre-sized inorganic and organic materials, are of interest for filtration because they have improved properties compared to pure polymers, such as mechanical, thermal, electrical, antimicrobial, antifouling, and catalytic properties. The filtration efficiency of numerous multifunctional nanofibrous membranes is reported, for example, in [69].

An inorganic compound widely used in composites is titanium oxide (TiO₂). It is well known that TiO₂ in the form of nanoparticles (NPs) has many advantages: it is an n-type semiconductor that is abundant in nature, sustainable, and non-toxic. In addition, it has good stability, hydrophilic properties, UV-blocking ability, and excellent photocatalytic and antimicrobacterial ability [70,71]. The photocatalytic degradation ability of TiO₂ NPs has been extensively studied. TiO₂ effectively converts UV-visible light energy into chemical

energy, which in turn can be used to decompose harmful organic materials in air and water [72]. For these reasons, TiO₂ NPs are used to produce multifunctional membranes for air filtration.

Electrospun fibrous mats were prepared by using a mixed solution of NPs of TiO₂ and PVA [60]. The performance of the nanofibrous mats was investigated in terms of pressure drop, particle filtration, and volatile organic compounds (VOC) removal. The use of TiO₂ as an additive influenced fibre morphology and pressure drop. It was hypothesised that the beaded morphology (which is usually considered undesirable) of the PVA/TiO₂ nanofibres was responsible for the lower pressure drop because the beads resulted in larger pores, as confirmed by the measured average pore size. This result is in contrast to other studies on PAN/TiO₂-based electrospun nanofibre filters, where a higher pressure drop was measured than that in pure PAN filters [73]. Although the efficiency of these filters against particles and VOCs is promising, the pressure drop needs to be further optimised, especially if the designed filter is to be used to improve indoor air quality in residential and office buildings.

TiO₂ NPs exist in both amorphous and crystalline forms, and they have been shown to have good antibacterial activity in both forms. In a recent study, a multifunctional nanocomposite membrane for air filters based on PVP and amorphous titanium oxide (mTiO₂) was prepared by electrospinning [69]. The starting solution was an acetic acid/ethanol solution containing PVP-Ti(OBu)₄. The combination of PVP and amorphous titanium oxide was chosen because the titanium precursor has high antibacterial activity and shows improved PM2.5 filtration properties, whereas PVP has a high dipole moment and is therefore easily electrospinnable. Unfortunately, PVP is hydrophilic. The hydrophilic nature of some polymers makes them unsuitable for prolonged use in air filtration in high-humidity environments, where, in the form of membranes, they absorb water molecules and can be easily damaged. The challenge is to prepare a nanofibrous polar polymer membrane with a hydrophobic nature. The addition of $mTiO_2$ solved this problem by making the PVP fibres highly hydrophobic. In fact, the average water contact angle of the PVP-mTiO2 membranes is many times greater than that of the PVP nanofibers. This is attributed to the presence of numerous -C=O groups in the linear PVP polymer, which gives it a hydrophilic nature. In fact, pure PVP tends to absorb water via the formation of hydrogen bonds. In contrast, the PVP-mTiO₂ nanofibres (-Ti-O-Ti-) network structures created by the hydrolysis-condensation process of titanium alkoxide and linked to PVP-C=O repel water from the PVP-mTiO₂ nanofibres (Figure 7). The resulting nanofibre filters have a PM2.5 filtration efficiency of 99.9% and a low-pressure drop of 39 Pa. A long-term filtration test for 160 h shows that the PVP-mTiO₂ nanofibres maintain a filtration efficiency of 99% for PM2.5. Regarding the antibacterial activity of this nanofibrous membrane, titanium oxide is demonstrated to act as a bacterial barrier for Escherichia coli and Staphylococcus aureus for ten days.

Another type of oxide used in the form of nanoparticles for the preparation of composite nanofibres is manganese oxide (MnO₂). The interest in this material is due to its catalytic activity, especially for the oxidation of formaldehyde (HCHO) [74]. Birnessite-type MnO₂ has special 2D layered structures with the most active oxygen species, resulting in the highest oxidation activity of HCHO at room temperature. This aspect makes birnessite-type MnO₂ of practical value in indoor air filtration. Hu et al. [75] used MnO₂ in two steps of the preparation process: first, they added MnO₂ to the PS electrospinning solution to obtain composite nanofibers and then coated the composite mat by dipping it in a KMnO₄ solution to increase the surface area of the nanofibrous membrane. This results in a membrane with filled nanopores and an increased surface area is obtained. Effective air purification is achieved for both PM2.5 and HCHO with remarkably low resistance. HCHO gas easily permeates through the mesopores of the PS nanofibres and comes into contact with MnO₂. As a result, a PM capture efficiency of 99.77% and an excellent HCHO removal efficiency of 88.2% are achieved.



Figure 7. Schematic diagram of the interaction of water with PVP nanofibre and with PVP-mTiO₂. Adapted from [69] with permission of Elsevier.

The efficiency of capturing metal oxide dust can be improved by using magnetic nanoparticles embedded in the nanofibres. Kim et al. [76] prepared a filter from PVP nanofibres containing Fe₃O₄ magnetic nanoparticles at different concentrations. At a concentration of 20 wt% and in the presence of an external magnetic field, the collection efficiency was ~29% higher than that of the filter without magnetic particles. The pressure drop was ~17 Pa, which was lower than that of the control filter (~27 Pa) and a conventional HEPA filter (~269 Pa), achieving a metal oxide dust removal quality 4.7 times higher than that of the control filter. This improvement in performance is attributed to the high concentration of magnetic nanoparticles in the filter: the greater the number of magnetic domains aligned along the magnetic field, the greater the magnetisation, and the greater the ability to trap the metal oxide dust particles. However, it should be noted that when using a magnetised filter, the benefits of the improved filter efficiency must be weighed against the costs of energy consumption and maintenance.

6.3. Green Nanofibres

Environmental and economic sustainability is a driving force for research into "green" aspects of filtration systems, including materials and processes. Electrospinning can be considered a sustainable process due to its simplicity, low time, and low energy consumption.

More challenging is the research on "green" materials with which to produce filters with high air filtration efficiency. In this respect, it would be preferable to adopt new formulations by incorporating bio-based materials, which are abundant and often inexpensive. In addition, less toxic and more environmentally friendly solvents should be used, not only to improve the economics of the process but also to protect the health of operators and users (Figure 8). All these considerations have led to the term "green electrospinning" [77].

An interesting effort towards sustainability is that of Keleş [78], who studied the preparation of a nanofibrous filtration membrane from industrial wastes, such as shrink film packaging of low-density polyethylene (LDPEWs) and acrylic fibres (AFWs). In addition, Cho [79] used renewable agricultural resources to synthesise bio-based polyesters and to prepare electrospun fibre filters with good filtration performance. Unfortunately, the solvents suitable for the dissolution of waste polymers and the synthesis of bio-based polyesters, typically toluene or DMF, are harmful.

Jiang et al. [80] avoided the use of DMF in the electrospinning of polyimide (PI) nanofibres. Polyimide is a polymer with excellent thermal and chemical resistance and satisfactory mechanical properties. Therefore, it can be used for applications at temperatures up to 400 °C, such as for hot gas filter membranes. Typically, PI solutions suitable

for the electrospinning process have a polymer concentration of less than 30 wt% in DMF or dimethylacetamide (DMAc). This requires the use of large amounts of organic solvents that are toxic, flammable, and environmentally unfriendly. In his work, Jiang obtained PI nanofibres by electrospinning ammonium salts of polyamic acid (PAA) from an aqueous solution, followed by an imidisation step to remove ammonia. The resulting PI/water nanofibres appear almost "fluffy," a property that increases their filtration efficiency against particles smaller than 0.8 μ m.



Figure 8. Peculiar factors in the definition of "green electrospinning".

In general, the use of synthetic polymers should be avoided because their degradability after disposal is a serious environmental concern [81]. Synthetic polymers continue to release harmful VOCs even after disposal [82] and recycling [83], with lethal effects on aquatic and terrestrial ecosystems [84]. Therefore, biodegradable polymers may be the best choice for air filters. It would be preferable for them to perform more than one function to be more effective against chemical and biological pollution. Among the biodegradable materials, those of natural, animal, or plant origin, such as chitosan and cellulose, have good potential for use in fibrous membranes produced by electrospinning. A separate discussion should be made for PVA. It is a water-soluble, low-cost polymer with good biocompatibility, degradability, and non-toxicity to the human body and has been widely used in the field of electrospinning. However, the mechanical properties of pure PVA nanofibrous membranes are poor, and their filtration performance is easily reduced due to nanofibre breakage. Therefore, the incorporation of inorganic components is a strategy used to overcome the drawbacks of PVA [60,85], as described in the previous section.

Chitosan is the second most abundant natural biopolymer in the world and is characterised by biocompatibility and biodegradability. In solution, it has a high viscosity and low stability, so other polymers, such as PU, PVA, and PEO, are added to obtain electrospinnable solutions of chitosan. A chitosan/PEO membrane was successfully prepared by electrospinning [86]. This membrane was then used as a substrate to grow MOF-5 nanocrystals along the electrospun chitosan/PEO nanofibres, resulting in a composite membrane. MOF-5 is a metallorganic-5 framework of Zn4O(BDC)₃ composition (BDC = 1,4-benzenedicarboxylate) with an extended three-dimensional cubic porous structure. It is one of the most studied MOFs and is characterised by a large surface area, chemical and thermal stability, high porosity, and tunable pore size. Due to its structure and chemical nature, MOF-5 has potential applications in gas storage and separation, dye removal, catalysis, and molecular adsorption [87]. The chitosan/PEO@MOF-5 nanofibrous membrane shows a PM2.5 filtration efficiency of 99.95% and a quality factor of 0.0737. In [86] the excellent filtration performance was attributed to the synergistic effect of the chitosan/PEO nanofibres and porous MOF-5, which causes the following behaviour. First, the multilayer nanofibrous structure of the electrospun membrane intercepts most of the particles via classical filtration mechanisms. MOF-5 provides abundant voids and gas adsorption sites to remove PM2.5 using electrostatic interactions and polarisation. In addition, the roughness of the membrane caused by surface protrusions due to the MOF-5 coating increases the probability of intercepting the collision between the pollutant particles and the fibres. Finally, the chitosan/PEO@MOF-5 composite membranes are free-standing and flexible, making them suitable as air filters with excellent filtration performance.

Cellulose and its derivatives are widely known as "green" materials. Cellulose is the most abundant polysaccharide on Earth and can be derived from a variety of sources, such as the cell walls of wood and plants, or from some species of bacteria and algae [88]. Due to its remarkable mechanical and thermal properties and its large surface area, nanocellulose is used in environmentally friendly composites. In addition, the reactive properties of cellulose can be a good advantage, especially in nanofibrous membranes characterised by a large surface area. Recently, nanoscale cellulose acetate (CA), the major organic ester of cellulose, has played an important role in the development of new membranes with good gas permeability and filtration performance. Nanofibrous composite membranes containing CA/PEO have a quality factor of 0.01338 Pa^{-1} and a filtration efficiency of 98.12% at an air flow velocity of 85 L/min, which are better than those of a two-layer nonwoven fabric of 0.00496 Pa^{-1} and 4.14%, respectively [89].

It is not trivial to have multifunctional filters capable of retaining both liquid aerosol droplets and VOC-containing gases. In fact, the mechanisms of PM and VOC filtration are completely different because PM and VOC differ in size and chemical composition. PM filtration is based on mechanical filtration mechanisms that depend mainly on fibre diameter and pore size. VOC filtration relies on physical and chemical adsorption, which requires a functional polymer surface [90]. Synthetic polymer filters have excellent environmental resistance and stability, but as multifunctional filters, they have some limitations due to the poor surface functionality required for VOC adsorption. In addition, as mentioned above, the degradability of synthetic polymers is a serious environmental concern, as they continue to release harmful VOCs even after disposal. A fully degradable biopolymer, such as gelatin, can be used to form composite nanofibrous mats suitable for air filtration. Gelatin is an inexpensive and readily available protein biopolymer obtained by partial hydrolysis of collagen found in various biomaterials [91]. Recently, gelatin nanofibres have also shown antibacterial activity and the ability to absorb gaseous pollutants due to their surface functionality [92]. Kadam et al. [93] prepared composite gelatin/β-cyclodextrin $(\beta$ -CD, a low-cost cyclic oligosaccharide) nanofibres by electrospinning for air filtration. They found that the addition of β -CD to the gelatin allowed the production of nanofibrous filters with excellent adsorption of xylene, benzene, and formaldehyde compared to a commercial face mask. In addition, the combination of gelatin/ β -CD nanofibres with their green nature, excellent dual-function filtration, low air resistance, and high potential to trap small viruses makes them suitable for respiratory filtration.

7. Effects of the Fibre and Filter Structure on the Filtration Effectiveness

One of the main issues in the manufacture of nanofibrous membranes is their handling and maintenance of their structural integrity, as these membranes are thin and fragile at low thickness. Filters are expected to maintain their structural integrity under continuous or changing air pressure. Electrospun nanofibrous membranes with reduced thickness are either too thin or too fragile to meet this requirement, and therefore need to be coupled with other support layers (i.e., spunbonded nonwovens, metal mesh, etc.) [94] to resist cyclic air flows, high pressure, or heavy cake formation due to particle dust deposition. It is not easy to unambiguously determine the mechanical properties, including the strength, of electrospun nanofibres because they can be measured on the individual fibres or the mat, and because they depend on the measurement techniques. A comparison of literature data on the mechanical properties of individual fibres and mats of the same material shows that the experimental details of the measurements are often unknown, or the test methods differ. A comprehensive and critical review of the data reported in the literature on the mechanical properties of different types of nanofibres and mats is reported in [95], where various factors affecting the mechanical properties, including materials and process parameters, are analysed.

In addition to optimising the electrospinning parameters, at least three different approaches can be used to improve the mechanical properties of electrospun nanofibrous membranes, which can be used individually or in combination. A substrate, such as a non-woven fabric, can be used as a carrier for the nanofibres. Alternatively, nanofibres with a composite structure and size can be used or fillers can be incorporated into the nanofibres during the electrospinning process. In this way, the fillers are aligned along the axial direction of the nanofibres, giving them stiffness. Another trick could be to create a sandwich structure in which the nanofibrous membrane is sandwiched between two layers of microscale fibres. The latter method is used for surgical masks.

Special Filtering Structures

Nanofibres of different polymers, such as PU, PC, PA, and PVDF, were electrospun onto a spunbonded PP substrate or onto a commercial nonwoven fabric [96,97]. Electrospinning processes that allow spatial nanostructures with microspheres, as well as structures with a broad fibre distribution, have been reviewed by Kimmer et al. [97]. They utilised the presence of microspheres deliberately created during electrospinning, to increase the distances between nanofibres and increase the active surface area for particle capture. Indeed, the incorporation of bead spacers into the nanofibrous structures results in an increase in the thickness and mass per square area of the material. This also has a positive effect on the mechanical properties of the fibrous structure and the filtration performance.

The mixed use of polymers can be useful in improving the strength of nanofibrous membranes, provided that their composition is optimised. As an example, PVDF and PS are electrospun separately on cotton using two syringe pumps to form a mixed structure with nanofibres of both polymers [98]. The presence of the two polymers in different proportions affects the tensile strength of the PVDF/PS composite fibrous mat. In fact, the tensile strength of the membrane decreases from 3.62 to 2.27 mPa when the amount of PVDF is higher than that of PS. This is attributed to the high packing density of the PVDF fibrous membrane (0.43 g/cm³) and the significant slip of the PVDF/PS composite fibres along the stress direction.

Compared to conventional electrospun materials, which have plain and homogeneous geometries, a honeycomb structure of the nanofibrous nonwoven material can provide a filter with improved mechanical properties. This structure was developed by Chen et al. [99]. By electrospinning PSU and PU nanofibres onto a hollow metal manifold with hexagonal geometries, honeycomb nonwovens with controlled hexagonal dimensions are produced (Figure 9). Two syringe pumps filled with PSU and PU are placed on opposite sides of a stainless steel mandrel manifold with hexagonal porous patterns. Different hexagonal pore sizes have been used. The PSU and PU solutions are spun simultaneously on a spindle collector using the same electrospinning parameters. In the presence of a strong electric field, polymer jets are induced to settle on the conductive grids of the hexagons. This results in an inhomogeneous distribution of nanofibres, with a much higher fibre density in the hexagonal grids than in the empty areas. The fibre orientation is also mainly determined by the hexagonal geometries. Indeed, no fibre alignment is observed in the hollow areas, whereas the fibres on the grids are well aligned along the hexagonal grid. Finally, a bimodal distribution of fibre diameter is found in the honeycomb samples: the average fibre diameter in the hollow areas (~520 nm) is significantly smaller than that of the fibres on the grids (~780 nm). This phenomenon is attributed to the accumulation and dissipation of electrical charges, which are enhanced at the metal trunks and attenuated at the hollow areas during electrospinning on the hexagonal collector. As a result, the fibres deposited on the metal grids are thicker than those in the hollow areas, where the

residual charge causes fibre elongation. The mechanical properties of PSU/PU nonwovens are also affected by their hexagonal size and tensile direction. Reducing the size of the hexagons results in less flexibility but better strength. In addition, the removal efficiency and pressure drop of the nonwovens increase as the hexagonal size decreases. Compared to plain membranes formed from homogeneous diameter fibres, honeycomb membranes with relatively small hexagonal sizes show similar removal efficiencies and significantly lower pressure drops. The explanation for the lower pressure drop of the honeycomb membranes is attributed to the higher honeycomb porosity observed in all patterns, where more than 90% of the areas are empty. The fibre layers in these regions are thin (average thickness ~17.2 μ m) and have large pores, allowing air to penetrate easily.



Figure 9. (a) Schematic diagram showing the fabrication process of honeycomb-like nanofibrous membranes. (b) Optical microscopy images of plain nonwoven and honeycomb-like membranes with various hexagonal dimensions. (c) SEM images and 2D FFT alignment plots of nanofibres located on the grid and empty areas of a hexagon (D = 3.5 mm). Two intensive FFT peaks at ~ 150° and 330° (bottom right) of fibres on the grid suggest the direction of alignment. Adapted from [99] with permission of Elsevier.

Another example of a micro/nanofibrous filter system is reported in [100], where an electrospun PA mat is combined with a meltblow PE nonwoven. The entire nonwoven contains a thin layer of electrospun PA nanofibres and a layer of meltblown PE microfibres bonded at the interface.. The assembled PE/PA nonwoven has two orders of pore size: a larger pore size (~143 μ m) formed by the PE microfibres and a smaller one (~6 μ m) formed by the PA nanofibres. This structure results in an improved filtration efficiency (>99%) compared to the PE nonwoven alone, which is measured at 62%. The addition of

nanofibres to PE tissue significantly reduces the pore size of PE/PA nonwovens, adsorbing and blocking small particles of less than 0.5 μ m in diameter. Increasing the thickness of the PA membrane results in a higher filtration efficiency and also a higher pressure drop. With regard to their use as a face mask, these combined PA/PE nonwovens show good mechanical strength, which is considered a key performance for wearability.

8. Conclusions

Air pollution is a pressing problem that is expected to worsen in the coming years unless effective solutions are not found. Nanotechnology could help with the use of filtration devices characterised by a high area-to-volume ratio, enabling high filtration efficiency and low-pressure drop. Nanofibrous membranes produced by electrospinning have a porous structure in which the pore size, fibre diameter, and thickness can be optimised by adjusting the process parameters. A wide variety of polymers, both synthetic and bio-based, can be used. In addition, functionalisation and the addition of nanofillers can improve the reactivity of nanofibres with air pollutants. A large number of articles and reviews have been published in the last two decades, reflecting the great interest of researchers in nanofibrous membranes obtained by electrospinning. In particular, for applications in the filtration of airborne particles, the literature contains an enormous amount of data on materials, polymer solutions, and process parameters, which are often inconsistent and contradictory. As a result, it is not possible to identify a clear indication of the best performing material or the most appropriate process parameters. Despite the advantages discussed above, electrospun nanofibrous membranes have some limitations. Electrospun polymers often require toxic or harmful solvents, and their resistance to temperature, chemicals, and mechanical stress is not very satisfactory. In addition, filters made from synthetic polymers can be difficult to recycle or dispose of, as they continue to release harmful VOCs even during reuse processes. For these reasons, sustainable and biodegradable bio-based or naturally occurring materials have attracted the interest of researchers over the last decade, who have devoted considerable effort to the preparation of reliable nanofibrous membranes with excellent filtration properties. The data reported in the literature are promising but not conclusive.

During operation, filters must maintain their structural integrity in the presence of continuous or variable air pressures. Low-thickness electrospun nanofibrous membranes are either too thin or too fragile to meet this requirement. Certainly, the filtration performance and mechanical resistance to the operating conditions of nanofibrous filters are greatly improved by using multiple mats of thin nanofibres. In addition, nanofibrous media sandwiched between organic or inorganic microfibrous media can improve strength and service life, but these types of structures often compromise filtration efficiency. These are the reasons for the enormous efforts in the study of nanofibrous membranes.

As for disposable nanofibrous masks, can they be a source of nanofiber debris that is dangerous to inhale during use? The answer is probably yes. Like other fabrics, nanofibre membranes used in face masks can release inhalable fragments through rubbing or prolonged use. However, it should be noted that the risk associated with face masks is also due to the release of certain chemicals used in the manufacturing process. It is difficult to determine which of the two factors is more dangerous to human health. In any case, there is no evidence or reason to believe that the risk from fibrous nano-plastics from membranes is greater than the risk from micro- and nano-plastics from other sources in the air.

In conclusion, the best results for obtaining nanofibrous membranes that are more efficient in filtering airborne particles seem to be obtained using nanofibres composed of polymer blends that are also enriched with inorganic nanophases. For each polymer formulation, the electrospinning parameters must be optimized, in particular, to ensure good mechanical properties and low-pressure drops. Filters structured by combining nanofibrous membranes with microfibre-based membranes can provide greater structural strength to the filter. Table 3 summarises the effects of the main characteristics of electrospun nanofibrous membranes on filtration effectiveness.

Issue	Parameters	Effect on the Filtration Effectiveness
Characteristics of the nanofibrous membrane (See Section 5)	Thickness Porosity Surface roughness of fibres	Fluid flowing through the membrane is controlled by porosity, pore size distribution, and membrane pore tortuosity. Filtration efficiency increases when fibre diameter and pore size are smaller and pore tortuosity is greater. Exceeding these parameters can lead to excessive pressure drop and membrane fouling. The presence of protrusions on the surface of the fibres, causing surface roughness, increases the probability of interception of flow particles.
Material (See Section 6)	Pure polymer Polymer blend Organic–inorganic nanocomposite "Green" materials	Various pure polymers have been used to form nano-fibrous mats suitable for filtration. Polymer blends may have better filtration performance. Functionalised nanofibres can have a higher interception capacity, especially if there is a chemical affinity between the functional groups and the intercepting particles. Due to their large effective surface area, nanofibres can carry a large number of functional agents. The combination of inorganic compounds and polymers can have a beneficial effect on the filtration efficiency and mechanical properties of nano-fibrous membranes. Bio-based polymers, natural materials used as additives, and environmentally friendly solvents are preferable for a "green electrospinning" process. Although the filtration efficiency of "green membranes" can be higher, their mechanical properties are sometimes poor.
Membrane structure (See Section 7)	Monolayer of nanofibres Multilayer of nanofibres Multilayer of micro and nanofibres	To improve the mechanical properties of the monolayer electrospun membrane, it is preferable to use high-strength polymers, such as polypropylene. Alternatively, multilayer membranes can be used, also in combination with microfibre layers (e.g., surgical masks). Carbon fibres or other non-woven fabrics can be used as substrates for nanofibres.

Table 3. Effect of the electrospun nanofibrous membranes properties on the filtration effectiveness.

The antimicrobial efficacy of nanofibrous membranes has not been considered in this review, as the importance and relevance of this issue should receive wide and dedicated attention, as the recent pandemic has shown. However, it is worth noting that the same solutions proposed to overcome the disadvantages of air filtration systems can also be used to limit the disadvantages of nanofibrous membranes in antibacterial and antiviral applications.

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