Nanotechnology transition roadmap toward multifunctional stimuli-responsive face masks

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TABLE OF CONTENTS

ABSTRACT: In recent times, the use of personal protective equipment, such as face masks or respirators, is becoming more and more critically important because of common pollution; furthermore, face masks have become a necessary element in the global fight against the COVID-19 pandemic. For this reason, the main mission of scientists has become the development of face masks with exceptional properties that will enhance their performance. The versatility of electrospun polymer nanofibers has determined their suitability as a material for constructing "smart" filter media. This paper provides an overview of the research carried out on nanofibrous filters obtained by electrospinning. The progressive development of the next generation of face masks whose unique properties can be activated in response to a specific external stimulus is highlighted. Thanks to additional components incorporated into the fiber structure, filters can, e.g., acquire antibacterial or antiviral properties, self-sterilize the structure and store the energy generated by users. Despite the discovery of several fascinating possibilities, some of them remain unexplored. Stimuli-responsive filters have the potential to become products of large-scale availability and great importance to society as a whole.

KEYWORDS: nanostructured face masks, stimuli-responsive nanomaterials, electrospun nanofibers, active filtration, smart filters, COVID-19, antipathogen.

INTRODUCTION

The ongoing coronavirus (COVID-19) disease outbreak initially appeared in China at the end of 2019 and triggered a rapid rise in the incidence of severe acute respiratory syndrome coronavirus 2 (SARS-CoV-2) cases.[1] The ensuing viral respiratory disease-based pandemic is considered the most significant global public health issue of the 21st century so far.[2] The World Health Organization (WHO) suggested wearing a properly fitted mask to prevent and slow down the transmission of COVID-19.[3] Significant advancements in the development of dedicated vaccines reduced the impact of SARS-CoV-2 on the health of patients affected by this disease.[4] Nevertheless, even though additional infection reduction measures, such as social distancing and bans on large gatherings, have been implemented, after more than two years of the spread of the pandemic, wearing appropriate face masks is still the most effective strategy for protecting people from the inhalation of viral matter and the consequent infection.[5,6] Therefore, wearing face masks became part and parcel of everyday life for the overwhelming majority of humankind. Additionally, it is worth remembering that using face masks has always been at the core of standard safety practices for workers exposed to flying ashes/powders and healthcare workers. It is also the typical protection method in the presence of high concentrations of airborne pollution.[7]

In this perspective, the production of face masks rose considerably in the last couple of years, bringing to light some significant problems connected with the extensive use of these protective devices.[8] The membranes filtering the air to decontaminate it from solid matters such as ashes, powders, and biological materials (e.g. viruses, bacteria, and fungi) should meet the technical requirements on filtration properties relevant to the final targeted applications.[9]

The scientific achievements obtained in the previous decades have made it possible to produce face masks with outstanding filtration performances. Nevertheless, the extensive and continuous use of this personal protective equipment (PPE) revealed a few unexpected issues.[10]

The large-scale consumption of face masks caused a massive amount of waste materials.[8,11] This problem can be tackled by designing membranes produced with environmentally-safe polymers. In any case, one of the basic principles of chemistry and physics, the law of conservation of mass, has taught us that *"nothing is lost, nothing is created, everything is transformed."*[12] The development of recyclable polymer-based masks is advisable;[13] on the other hand, partly biodegradable polymers are environmentally hazardous, as confirmed by the extensive distribution of polymer microparticles throughout the ecosystem.[14] In recent years researchers have been exploring an entirely different strategy aiming to solve this issue, as well as other related problems. The approach presented in this paper is based on the idea that waste reduction is possible by introducing novel capabilities aimed at enhancing face mask performance for specific applications while extending their lifetime.[15] In this perspective, it is necessary to develop fibrous membranes with unique features that can be either constantly active or triggered on demand.[16] One of the most requested properties is the possibility of reducing the number of pathogens deposited onto face masks, thus enhancing the lifetime of face masks for frontline healthcare workers while reducing the possibility of cross-infection.[17] The ability to decontaminate the face mask structure without reducing the filter performance and stability opens the door to the development of long-term reusable and safe PPE.[18] In addition, nanotechnology-based strategies endow filter membranes with novel unique features, thus opening the field to brand-new devices such as energy-harvesting face masks with self-powered properties.[19]

Over the past decades, the entire scientific community of material scientists, working on both fundamental and applied research, has focused its efforts on developing innovative fibrous filtering materials, and this has led to innovative technologies.[20] The constantly growing effort and commitment spent by researchers in this field have focused on certain techniques through which it is possible to produce nanometric polymer fibers with a high surface-to-volume ratio,[21] high porosity,[22] exceptional mechanical properties, and stability, such as the electrospinning technique.[23] In recent decades, electrospun nanomaterials have been extensively investigated to produce high-performance face masks (Figure 1a). Most importantly, electrospinning is a versatile technique that ensures the possibility of incorporating additional materials, including inorganic,[24] metal,[25] and organic nanomaterials, in the nanofibrous structures produced.[26] Active compounds and stimuli-responsive nanomaterials can be combined with electrospun nanofibers, opening the field to the development of functional face masks.[27] The entire scientific community is concentrating the maximum attention on designing intelligent face masks capable of responding to external stimulation (Figure 1b).

Several significant reviews have been published on the application of electrospun nanofibers for face mask production.[28,29] The recent use of electrospinning in combination with active and stimuli-responsive nanomaterials to develop functional, smart face masks has not yet been analytically discussed. In this review, we aim to offer an in-depth picture focusing on the most important steps taken by material science researchers in developing a next-generation nanofiber-based functional face mask. First, a reasoned understanding of face mask principles, their historical development, and the electrospinning technique is provided. This is followed by a significant and inclusive review of the literature on the development of passive, active, and stimuli-responsive electrospun media to produce face masks, including the novel functional and on-demand activatable features. In addition, the vital importance of combining advanced techniques and novel materials with already-developed filtering protective devices is suggested, highlighting the future prospects and challenges in this dynamic field. Lastly, the review ends with a discussion of the valuable essential guidelines on the most effective pathways for developing a novel generation of face masks with brand-new, unique capabilities.

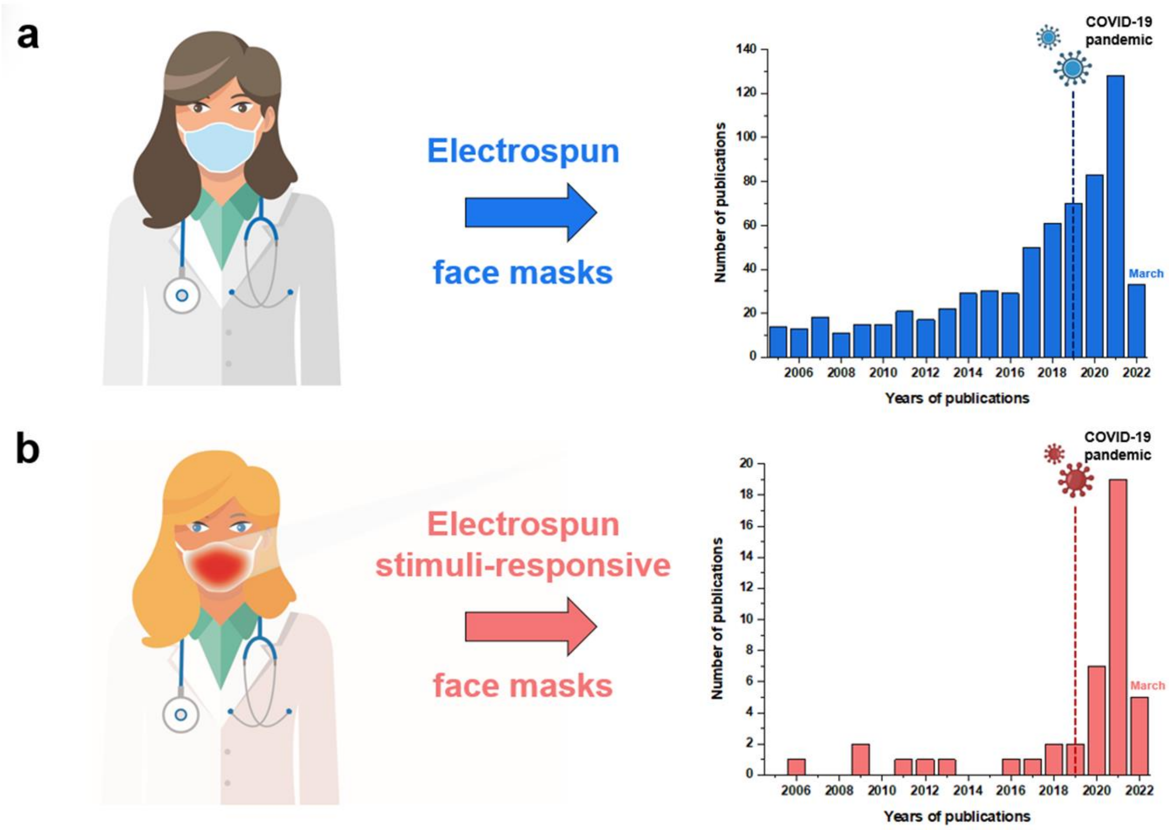

Figure 1. Impact of COVID-19 pandemic on the development of protective face masks. a) Sketch of an electrospun face mask for passive filtration and the number of published articles relating to passive filtering face masks in 2005-2022. Data were obtained from the Scopus database on 14/03/2022 and have been reproduced with permission.[15] Copyright 2020, Wiley-VCH. b) Representative illustration of an electrospun face mask for active filtration and the number of published articles relating to "smart" face masks in 2005-2022. Data were obtained from the Scopus database on 14/03/2022 and have been reproduced with permission.[15] Copyright 2020, Wiley-VCH.

FACE MASKS – A HISTORICAL OVERVIEW

Face mask use and technological development are interwoven with scientific and technological progress and epidemiological and historical backgrounds.[30] From a historical standpoint, the use of face masks has been driven by the need for protection from possible contaminations deriving from the breath of other people.[31] Indeed, in the 13th century AD, the

Venetian merchant Marco Polo, in his book "*Il Milione*",[32] reported on the use of an archetypal face mask. Noble people, in charge of preparing meals for the emperor Kublai Khan, kept their mouths and noses bandaged with drapes of gold and silk to prevent their breath contaminating the emperor's meals.[32]

According to a popular-science visual trope, the use of face masks for medical purposes dates back to the prototypical pandemic, namely the "Black Death" bubonic plague (1346-1353). During that period, doctors did not wear any personal protective devices; nevertheless, "plague doctors" were depicted, in retrospective artistic representations, wearing a costume complete with a beaked mask.[33] In reality, the beak mask was designed and fabricated by Charles de l'Orme in 1619, shown in section 1619 of Figure 2**,** to protect doctors from the plague outbreak in Paris. The mask's beak was filled with perfumes, spices, and garlic, which were expected to neutralize the "miasma" considered responsible for the plague outbreaks.[34]

However, in 1897, Polish surgeon Johann Mikulicz, shown in a portrait in section 1867 of Figure 2,[30,34] was the first researcher to suggest a scientific approach to the use of face masks. The scientific landscape of the second half of the 19th century was marked by remarkable signs of progress in the field of microbiology. Indeed, in 1867, Louis Pasteur's studies on the spread of diseases due to microorganisms inspired Joseph Lister to implement the concept of asepsis.[35,36] Asepsis consisted of using chemical substances to prevent the contamination of surgical wounds with germs transmitted by instruments, hands, sponges, and gauzes.[35] Thirty years later, driven by the results obtained through his cooperation with bacteriologist Carl Flügge, surgeon Johann Mikulicz wore a face mask in an operating room for the first time. Indeed, Carl Flügge demonstrated that very small respiratory droplets (Flügge droplets) contain culturable bacteria and, therefore, can be an additional cause of post-surgical infections.[34]

The extension of face mask use to patients and the population is historically associated with the outbreak of the Manchurian Plague (1910). Such a practice was promoted by Doctor Wu

Lien-the, who was appointed by the Chinese Imperial court to coordinate the efforts against the plague outbreak.[33] Doctor Wu advanced the breakthrough hypothesis of direct airborne transmission of the plague between humans, thus encouraging the widespread use of protective face masks. The "anti-plague mask" shown in section 1910 of Figure 2 was claimed to be Doctor Wu's personal invention. It was made of several protective layers and required a complex tying procedure, resembling the current surgical face mask.[33]

The use of protective face masks was mandatory for medical personnel and police forces (Figure 2, section 1918) during the influenza pandemic in 1918-1919, the so-called Spanish Flu. The widespread use of face masks was considered effective in decreasing the number of deaths.[34]

Since the 1960s, disposable face masks have been used by medical personnel and the population of some densely populated Asian countries such as China, Japan, and South Korea. Disposable masks (IIR type), represented in Figure 2, section 1960, are made of nonwoven synthetic fibers organized in three layers made of nonwoven polypropylene, resulting in an efficient network for filtrating the wearer's respiratory emissions.[37]

Respirators, on the other hand, can avoid the spread of respiratory emissions both from and to the wearer. The N95 (Figure 2, section 1995) is probably the most well-known and commonly used respirator. Its filtering efficiency is 94% for viruses and almost 100% for bacteria.[38] The technology behind the N95 filtering efficiency was conceived in 1992 and patented by Prof. Peter Tsai three years later. The trapping of hazardous substances, including dust, bacteria, and viruses, relies on the electrostatic attraction promoted by positive and negative charges inherent in the N95 material.[39]

The spread since December 2019 of the SARS-CoV-2 virus, sketched in section 2019 of Figure 2, has made the use of face masks one of the necessary global countermeasures against the transmission of COVID-19. Face masks have been strongly recommended by the World

Health Organization (WHO).[40] The impact of the SARS-CoV-2 outbreak was so severe that the general public was encouraged to use face cloths and surgical masks as an alternative to the N95 respirators.[41]

Scientists promptly contributed to tackling the COVID-19 pandemic and the issues related to the face mask shortage[42] by advancing challenging solutions that were not limited to improving the filtration efficiency of face masks and respirators, but could also endow face masks and respirators with new capabilities. Thanks to original nanotechnology-based approaches, face masks can now boast antimicrobial activity, sensing ability, greater comfort, and filtration efficiency.

Indeed, a filter medium for face masks, functionalized with Cu nanowires swaddled by a metal-organic framework, demonstrated antibacterial activity and antiviral properties against the SARS-CoV-2 virus.[43] Antiviral activity can also be achieved by coating face masks with a nanocomposite obtained from Cu nanoparticles embedded in shellac[44] or by directly depositing Ag nanoparticles onto textiles[45], as will be discussed in the section 4.2.3.

During the COVID-19 pandemic, face masks were also viewed as a system suitable for analyte sampling. Vaquer *et al.*, inferring that SARS-CoV-2 antigens accumulate in the inner hydrophobic polypropylene layer of medical face masks, fabricated a point-of-need biosensor that can be applied to the inward layer of face masks.[46] This biosensor is made of filter paper endowed with a reservoir containing Au nanoparticles functionalized with a mouse monoclonal antibody Anti-SARS-CoV-2 N-protein. The antibody recognizes SARS-CoV-2 antigens, thus triggering a color change that a smartphone app can quantify.[46]

Face masks can also be conceived as wearable sensing devices for wireless patient monitoring. A respiratory rate mask was proposed as a wearable sensor to monitor the respiratory rate based on an impedance alteration detected in a humidity sensor containing CsPbBr3 perovskite nanoparticles.[47]

Electrospinning-based nanotechnologies, however, offer outstanding opportunities for integrating multiple functions in face masks. An inspiring perspective for developing next-generation face masks has been proposed by De Sio *et al.*[15] The Authors proposed the design of a face mask based on multifunctional membranes. The membrane is made of electrospun nanofibers that offer more comfort to the wearer via two strategies. The first is the generation of a nanofiber architecture able to dissipate the humidity (moisture pump technology). The second is the design of a face mask which is adapted to the physical characteristics of users' face by an additive manufacturing technology. Moreover, the incorporation of Cu nanoclusters (outside the electrospun nanofibers) and Au bipyramids (inside the electrospun nanofibers) provides bactericidal and on-demand self-disinfecting properties, thus making the face mask reusable.

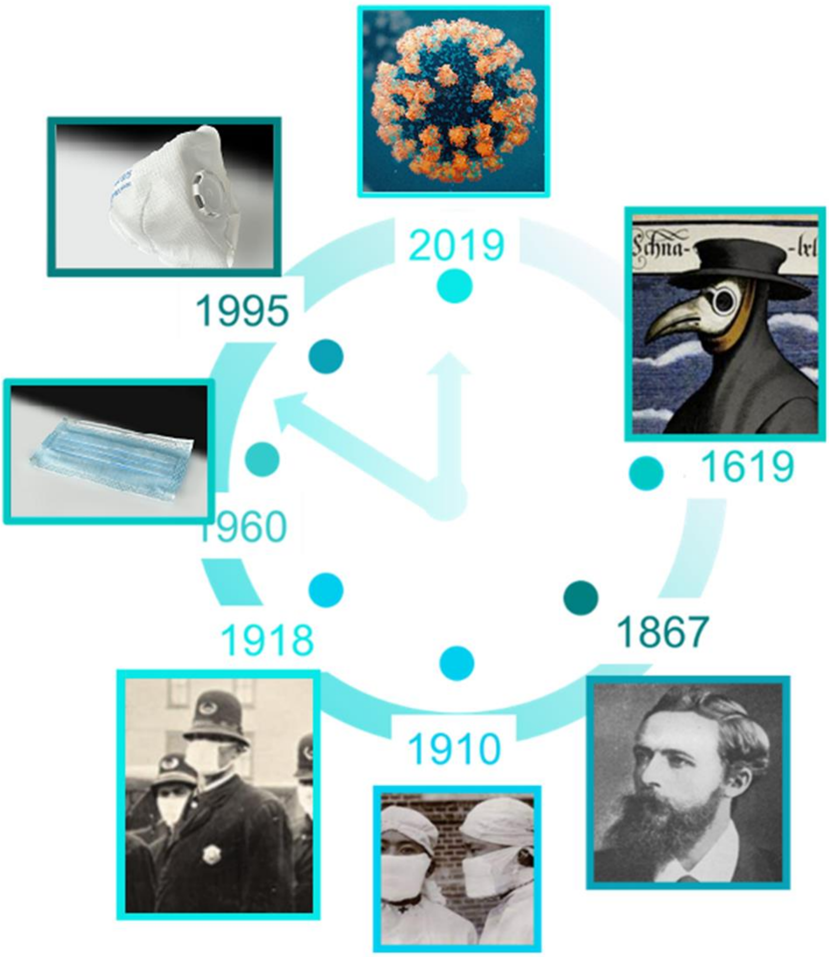

Figure 2. Evolution of face masks associated with the milestones of humankind history: from the "Black Death" bubonic plague of 1619 to the COVID-19 era beginning in 2019. 1619: a section of the picture "Doctor Schnabel von Rom" from Paulus Fürst illustrating the first face mask for medical use, designed by Charles de l'Orme in 1619. Reproduced with permission.[48] Copyright 2008, Wissen Media Verlag. 1867: portrait of the surgeon Jan Mikulicz-Radecki, who was the first to wear a face mask in an operating room. Reproduced with permission.[49] Copyright 1960, Lippincott-Raven Publishers, published by Wolters Kluwer Health, Inc.. 1910: multilayer face mask invented by Doctor Wu Lien-teh, designed for the health management of the pneumonic plague in Manchuria. Reproduced under terms of the CC-BY-NC-ND license.[33] Copyright 2018, the Author, published by Taylor & Francis Group, LLC. 1918: policemen wearing masks protecting them from influenza germs. The massive use of face masks among medical workers and the general public made a crucial contribution toward reducing "Spanish Flu" infections. Reproduced with permission.[50] Copyright 2008, Mitchell Lane. 1960: the disposable face mask. Since the 1960s, disposable face masks fabricated with artificial materials have replaced the use of cotton-fiber face masks. 1995: the N95 respirator patented by prof. Peter Tsai. 2019: a graphic representation of the morphology of SARS-CoV-2 virus, causing the COVID-19 disease. The COVID-19 outbreak opened up a new era for the widespread use of face masks, leading to the design of new face masks with multiple capabilities. Reproduced with permission.[51] Copyright 2020, Springer Nature Limited.

ELECTROSPINNING

Since the late 20th century, electrospinning has been gathering growing attention in the industrial and scientific communities.[52] It is considered a vital technique through which it is possible to produce 1D nanofibers with unique properties compared to other nanostructured materials.[27] The very first experiments related to electrospinning, however, can be found as far back as the 16th century, when attempts were made to understand electrostatic phenomena.[52,53]

At that time, William Gilbert (physicist to Queen Elizabeth I) observed that a charged piece of amber, at a sufficient distance away from a water droplet lying on a dry surface, attracted the droplets and pulled them into a cone shape.[53] With the growing use of the electrospinning technique in industry, in the 20th century, John Francis Cooley filed the first electrospinning patent for a method using different nozzles: conventional, coaxial, and airflow-assisted, and a spinneret featuring a rotating distributor.[54] Methods for the parallel spinning of multiple cellulose acetate fibers with simultaneous yarn formation attracted the interest of Anton Formhals, resulting in the patenting of numerous solutions in both the field of spinning nozzle construction and the process of fiber-winding devices.[55] Later in the 1990s, research interest gained momentum. With the development of a theoretical basis for the spinning process, the spread of this technique increased sharply in both the fundamental research and industry.[56]

The basic principle of electrospinning is based on a polymer solution being sprayed in an electric field to form continuous fibers.[56,57] During the electrospinning of a polymer solution, an electric current applied to a metal nozzle leads to the repulsion of unipolar charges in the solution droplet.[58] When the charge limit is exceeded, the surface tension forces are overcome. As a result, a stream of polymer is ejected and stretched. If the solution viscosity is too low, the polymer jet will break apart and form droplets (electrospray).[59] Uniform charges repel each other, causing the polymer jet to spin and stretch further. At the same time, viscoelastic forces prevent the jet from breaking apart. On its way to the collector, the solvent evaporates from the fiber, leading to its solidification and collection on a grounded collector in the form of a nonwoven fabric. The type of collector depends on the purpose of the material. Using appropriate collector shapes makes it possible to create tubes, 3-D scaffolds, yarns, or flat membranes with a large surface area with randomly and oriented fibers (Figure 3**)**.

The electrospinning technique has received a great deal of attention from science and engineering communities thanks to its easy operation and the possibility of an efficient production scale-up.[27,60] In terms of its application, it provides materials with a large specific surface area and ultra-fine porosity. For this reason, electrospun nanofibers have found wide use in filtration,[61] photocatalysis, drug delivery,[26,62] and tissue engineering.[21] It is also a very attractive technique, because it offers many options for altering material at the nanoscale to form nanocomposites. As for its use in air filtration, it is the only technique capable of producing materials with high porosity and therefore low filter resistance for air.[63] Another advantage is its high surface-to-volume ratio, providing space for functionalization to efficiently remove numerous pollutants such as bacteria, viruses, and harmful gases.

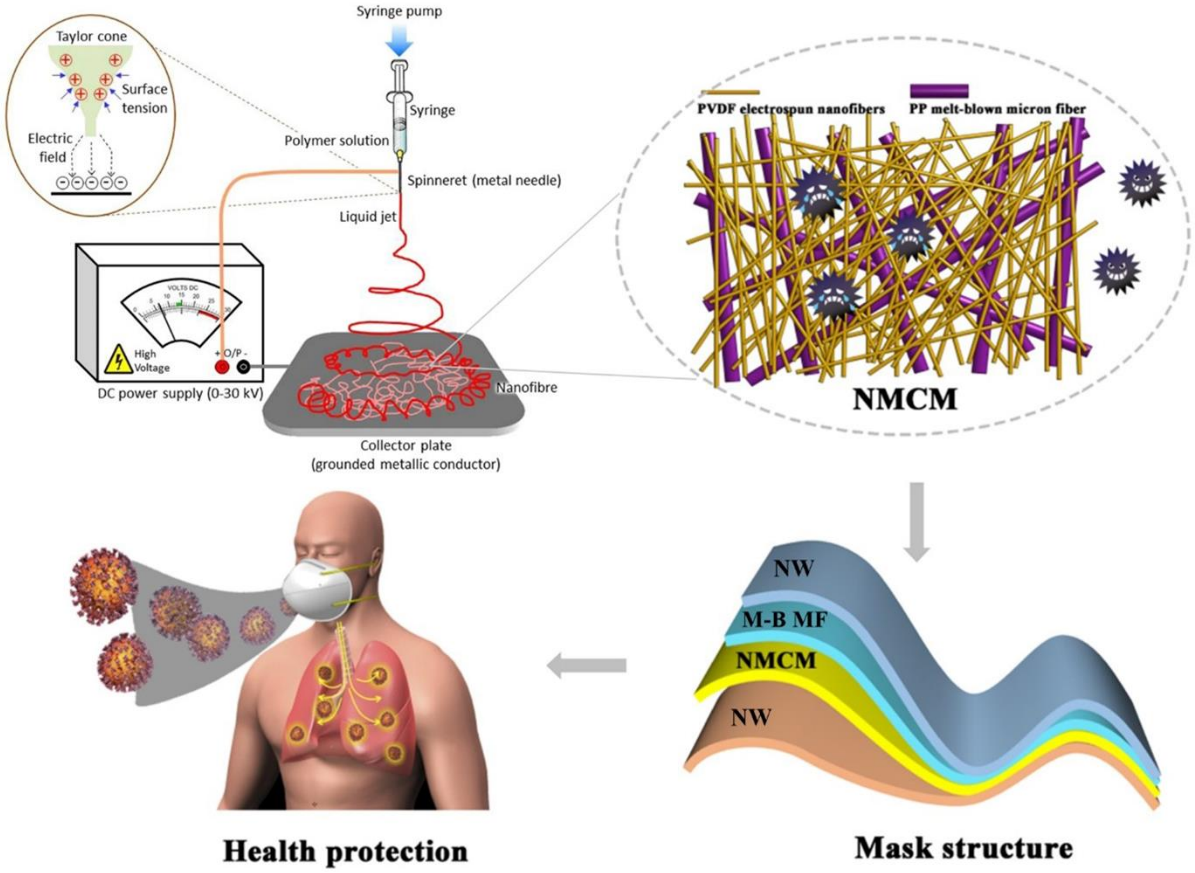

Figure 3. Electrospinning apparatus set-up and nanofiber/microfiber composite membrane (NMCM) spun on polypropylene melt-blown micron fibers (M-B MF). The filtration mask structure contains additional protective nonwoven layers (NW), and its final application shows protection against viruses. Reproduced under terms of the CC-BY

ELECTROSPUN NANOFIBER-BASED FACE MASKS

Passive filtering membranes

Different face masks provide different levels of protection for the wearer, depending on the material of which they are made and their engineering design. The level of protection is closely related to the ability to filter pollutants and bacteria or viruses of varying sizes.[65–69] The porosity of the fibrous membrane plays a crucial role in the passive particulate matter filtration mechanism. High filtration efficiency is achieved by using thick layers of densely packed nanofibers. Materials with small pores catch smaller particles than materials with large pore sizes, such as cotton and synthetic fabrics.[70] Electrospinning is the most frequently used technique for producing nanofiber membranes of this type.[71]

Despite their high filtration efficiency, conventional face masks of melt-blown microfibers usually make breathing difficult.[72–74] Electrospun nanofibers are adequate substitutes for this masks because they provide high filtration efficiency, even close to 100%,[75] without causing respiratory distress.[76]

Essential parameters for assessing the user's comfort with a given filter medium are pressure drop and air or water vapor permeability.[77] Electrospun membranes have randomly arranged ultrafine fibers and a high surface-to-volume ratio. Moreover, they are nanoporous, vapor permeable and have good mechanical properties, so their use for personal protection face masks against minute particles, bacteria, and viruses can be particularly beneficial.[78–82] Moreover, nanofibers are considered especially effective in filtering submicron and nanometric contaminants, while having only a slight influence on the pressure drop.[83] The diameter of fibers and pores of the materials obtained by electrospinning polymer solutions can be easily controlled and regulated. The diameter of nanofibers can be 10-100 times smaller than conventional melt-blown microfibers.[83]

Possible particle trapping mechanisms (Figure 4a) explain how the high surface-to-volume ratio of electrospun fibers improves the filtration efficiency. Filtration can be achieved in five different ways, depending on particle size. These mechanisms include sieving, interception, inertia impaction, diffusion, and electrostatic attraction.[76,83]

If the particle size is larger than the pore size of the filter membrane, they are captured by sieving. When the polymer nanofibers are charged, they can attract particles having an opposite charge – according to the electrostatic attraction mechanism. Other, smaller particles are filtered via the other three mechanisms. Particles with a size of 300-600 nm move with airflow. Since they are heavier than air, they are affected by an inertial impaction[84,85] and cannot separate in different directions, as air does when entering the fiber pores, so they hit them and deposit on their surface. Medium-sized particles are too small to have inertia and too large to diffuse in an airflow. Therefore, they are filtered by direct interception when the air stream deflects in the spaces between fibers.[83] The smallest particles, i.e. those with a size smaller than 300 nm, can diffuse in the airflow.[84,85] Moving randomly, they collide with the fibers on which they deposit. Therefore, as shown in Figure 4a, the smaller diameter of the electrospun filament corresponds to the higher filtration efficiency on particles of a specific size.

All the properties described above have led electrospun nanofibers to be increasingly used in the design and production of face masks all over the world.[42,86] Up to now, the following materials have been studied for use as passive filtration membranes in nanofiber mats: nylon-6.6,[87] nylon-6,[88–90] polycaprolactone,[91] poly(ethylene terephthalate),[92] silk fibroin,[92] polylactic acid,[93] poly(methyl methacrylate),[83] polybutylene succinate,[94] polyacrylonitrile,[76,86] polyvinylidene fluoride.[42,95]

Polyamides

Polyamides are long-chain polymers in which the basic units are bound together through amide bonds (-C(O)-NH-). These compounds are classified as crystalline materials with a high melting temperature, stiffness, mechanical strength, and chemical resistance. However, they show considerable susceptibility to moisture. Moreover, polymer nanofibers with polar functional groups, e.g. nylon-6,[88,89] have a strong affinity for particulate matter (PM). This makes polyamides highly effective for eliminating pollutants with high efficiency at a low-pressure drop and sufficient air permeability.[88]

Zeraati *et al.* optimized nylon-6.6 electrospinning parameters so that the obtained nanofibers had a specific diameter and made it possible to prepare a membrane useful as a filter against the SARS-CoV-2 coronavirus.[87] To determine the conditions of this process, researchers used artificial intelligence, including gene expression programming (GEP) and genetic algorithms (GA), as shown in Figure 4b. The results proved that the application of a 26 kV voltage makes it possible to obtain high-directional nanofibers. In addition, by electrospinning a 16% solution using a working distance of 18 cm and a flow rate of 0.2 $ml \cdot h^{-1}$, nylon-6.6 nanofibers with a diameter of 55.8 nm can be obtained. It is worth mentioning that coronaviruses have a size of 60-140 nm, and their aerosols measure 0.3-2 μm. Scanning electron microscopy (SEM) measurements indicate that the surface of the obtained nanofibers is smooth, and they are arranged in a structure resembling a spider's web. Their porosity was 84.3%. The filtration efficiency (following ASTM standards) was 98.6%. The obtained diameter of nanofibers, their porosity, and the fact that they can attract viruses due to electrostatic interactions make it possible to use the developed structure as a filtering layer in face masks.[87]

Considering the importance of the thermal comfort of face mask users, Yang *et al.* developed a filter layer based on nylon-6 that is highly efficient in particle trapping and effective radiative cooling.[88] Electrospun nanofibers were transferred to a nanoporous polyethylene substrate (nylon-6/nanoPE fibers), thus increasing their mechanical strength. The nylon-6 nanofibers produced were characterized by a small diameter (<100 nm). Because they also had a large dipole moment, $PM_{2.5}$ particle filtering tests showed >99.0% efficiency for fibers with density reduced to about 85%. Particle trapping was accompanied by a low-pressure drop that characterizes air permeability. The polyethylene substrate is highly transparent to infrared radiation. Therefore, its use permitted an effective radiative cooling, ensuring thermal comfort for users on warm days. An additional advantage is that both materials used in this study are low-cost and readily available.[88]

Recently, another research group modified a nylon-6/PE membrane by electrospinning nylon-6 (PA) into a polyethylene non-woven fabric.[90] The new substrate proved better for face mask applications than pre-punched nanoporous PE. In addition, the filter developed by Xu *et al.* had a two-level pore size of the electrospun mat providing gradient filtration. PA nanofibers formed larger pores (~143 μm), while PA microfibers formed smaller pores (~6 μm). Due to the gradient filtration and the high dipole moment of nylon-6, the composites showed high efficiency in the filtration of a particulate matter (>99%) with a low-pressure drop (<100 Pa) and were also highly transmissive to mid-infrared radiation, thus being able to remove heat effectively.[90]

Polyesters

Polyesters are polymers with multiple ester bonds (-C(O)-O-) in their main chain. They are characterized by mechanical strength and elasticity. Due to their properties, they are used primarily in the textile industry and for the production of plastics.

Polycaprolactone (PCL) is a biocompatible material,[96] and its nanofibers are successfully used in tissue engineering,[97] as well as in air filtering devices. However Khandaker *et al.* investigated for the first time their use as a filter medium for use in face masks.[91] In this study, PCL nanofiber mats were fabricated by electrospinning on a drum collector for 3 or 10 minutes (PCL-3, PCL-10, respectively). By selecting the parameters for electrospinning, the pore diameter could be controlled, which made it possible in the end to obtain an average pore value of 1.4±0.3 μm for the PCL-10 sample. For comparison, the filtering layer from the Henry Schein Earloop surgical mask (as a control sample) was also tested. It was shown that, in this case, the average pore diameter was 5.7±0.6 μm, which is almost 50 times the size of the average virus, whose diameter is approximately 120 nm. The values of the contact angles showed that all the mats – PCL-10, PCL-3, and the control sample – were highly hydrophobic. The contact angle, however, was the largest for the control sample, while PCL fabrics absorbed water droplets to a lesser extent. Moreover, they were resistant to extreme temperature conditions, and their properties did not change in the presence of a solvent. The PCL-10 fabric turned out to be the strongest mechanically, resistant to a pressure of 18 Psi, and the filtration efficiency test proved that the double layer of the spun mat placed between fabrics is impermeable to particles smaller than 120 nm. Based on the results obtained, it can be said that PCL electrospun fibers may be a better filter medium for surgical masks than those currently used.[91]

Poly(ethylene terephthalate) (PET) is a semi-crystalline thermoplastic polyester,[98] characterized by flexibility, high mechanical and thermal strength, and good chemical and electrical insulating properties,[99] enabling its wide application. Opálková Šišková *et al.* prepared a composite membrane based on PET with the addition of silk fibroin (SF), which could serve as a filter medium protecting against air-polluting particles, while ensuring a comfortable use.[92] Importantly, such a filter makes it possible to reuse plastics. Since SF can be obtained from natural silk cocoons, its production cost becomes negligible and hassle-free due to the easy availability of the necessary materials. In this study, electrospun membranes based on PET obtained from used bottles together with different SF contents were produced and tested. Micrographs taken with SEM showed that the fabricated mats were smooth, without defects, and the fibers were continuous and randomly oriented. SF-containing nanofibers were thinner than pure PET fibers, and the pore size of composite membranes was smaller than the pore size of the mats made of their pure components. The increase in silk fibroin content in the composites increased air permeability. The most effective filter from all those tested (with a filtration efficiency of 90.2%) belonged to the FFP1 class following the EN149 classification. Belonging to the FFP1 class makes it possible to use a filter as personal protective equipment in the form of a face mask.[92]

Polylactic acid (PLA) is a thermoplastic polymer belonging to aliphatic polyesters. This polymer is biocompatible, non-toxic, and safe for the environment. In addition, it has good mechanical properties, for which its use is very widespread. PLA is used in packaging for food, furniture and clothes, and in the medical and pharmaceutical industries.[100] Medical sutures, stents, and hygiene products for women are made from this polymer. Buluş *et al.* used the electrospinning technique to prepare a polylactic acid membrane and composites containing activated carbon as reinforcement.[93] A significant advantage of activated carbon, in this case, is the ability to adsorb gaseous pollutants in its deposits. In this way, it is possible to purify the air of volatile organic compounds, other gaseous pollutants, and unpleasant odors such as, e.g., the smell of cigarette smoke. Unfortunately, this material is insufficient for filtering particles such as dust and mold. In this study, four mats with the composition of 10% PLA, 10% PLA-1% AC, 10% PLA-5% AC, and 10% PLA-8% AC were obtained. Their structural, morphological, and mechanical analyses were then performed, and their filtration abilities were tested. The research showed that when the concentration of active carbon was increased, the electrical resistance of the solutions rose, the nanofibers obtained were thinner, and their mechanical strength was better. Their diameter was in the range of 80-240 nm. The best fibers were obtained in a composite containing 10% PLA and 8% AC, for which the tensile strength was 60 MPa. For all four membranes, the filtration efficiency of both bacteria and submicron particles was greater than 98%. Therefore, it has been proven that PLA-AC composites have ideal properties for use in the production of personal protective equipment and filtration applications.[93]

NanoLayr Ltd. developed a new type of filter membrane based on electrospun nanofibers. For this purpose, the company used poly(methyl methacrylate) and ethylene vinyl alcohol. The product is currently sold under the trade name FilterLayr™.[83] Poly(methyl methacrylate) (PMMA) is a thermoplastic polyester with high transparency[101] and stiffness, commonly known as acrylic glass. Moreover, it is highly resistant to UV radiation. Ethylene-vinyl alcohol (EVOH), on the other hand, is a copolymer of ethylene and vinyl alcohol and is characterized by low oxygen permeability. For this reason, it is widely used in the production of food packaging. Karabulut *et al.* investigated the filtration efficiency of nanofiber materials similar to those marketed as FilterLayr™.[83] Scientists produced more than 75 samples by electrospinning a PMMA/EVOH solution onto a polypropylene (PP) non-woven fabric. Tests were performed according to the following international standards: ASTM Test Method F2299 (for surgical masks), ASTM Test Method D3502 (for barrier masks), and NIOSH 42CFR84 (for respiratory masks, N95). In this study, researchers used various types and sizes of particles and variable air velocity for their tests. Results showed that nanofibers are highly homogeneous, and that as filters, they meet the requirements of each of the standards. Following the standard for respiratory masks, the filtration efficiency was 98.1% (pressure drop of 226 and 290 Pa at a rate of 85 and 120 $l \cdot min^{-1}$, respectively). According to the standard for surgical masks, the filtration efficiency was 99.9% (pressure drop of 44 Pa at a rate of 8 $l \cdot min^{-1}$ with a filtered particle size of 100 nm), and for barrier masks, 99.7% (pressure drop 133 Pa at a rate of 60 $l \cdot min^{-1}$). Therefore, it has been shown that the materials based on electrospun PMMA/EVOH nanofibers produced by NanoLayr Ltd. meet all the requirements of face masks set out in three major, albeit quite different, international standards.[83]

Polybutylene succinate (PBS) is a thermoplastic polymer resin from the polyester family. This semi-crystalline polymer is a biodegradable material. Choi *et al.* developed an electrospun PBS filter which wholly degrades in compost soil thanks to its biodegradability.[94] The produced filter consisted of two integrated mats – microfiber and nanofiber – obtained by the electrospinning method and covered with chitosan nanowires. These researchers used PBS solutions with different concentrations and, consequently, viscosity to obtain mats with varying fiber diameters. In this case, the particle capture mechanism combines physical sieving and electrostatic adsorption due to cationic sites and polar amide groups in chitosan. The efficiency of the obtained filter was comparable to the commercial N95 filter. It removed 98.3% of the 2.5 μm diameter solids ($PM_{2.5}$). PBS nanofibers screened contaminants, and microfibers ensured high breathability and low-pressure drop (59 Pa). Moreover, the resulting filter is reusable and fulfills its function even when wet, while the efficiency of the N95 filter drastically drops due to moisture, limiting its use to one time. Additionally, chitosan is also a biodegradable material, and studies have shown that the obtained filter is wholly degraded in compost soil within one month. A comparison of conventional non-woven filters and the integrated filter developed in this study is shown in Figure 4c.[94]

Polynitriles

Polynitriles are vinyl polymers composed of units derived from hydrogen cyanide; their structure contains a nitrile group (-C≡N), which is attached to the rest of the compound via a carbon atom.

Polyacrylonitrile (PAN) is a polar, stiff, and brittle duroplastic polymer. Nanofibers obtained from polymers such as PAN have reinforced polar groups on their surface.[89] They are often used as precursors to produce carbon nanofibers.[102] Lee and Jeon have developed a membrane made of PAN electrospun nanofibers which, apart from high filtration efficiency, also made it possible to monitor the user's breathing.[76] This study examined a pure polymer PAN mat, while a mat containing an electrically conductive metal-organic (MOF) – Ni-CAT-1. $Ni(OAc)_2$ framework was synthesized directly on PAN nanofibers by a two-step hydrothermal reaction where the linking molecules were 2,3,6,7,10,11-hexahydroxytriphenylene (HHTP). The growth of Ni-CAT-1 crystals on the surface of nanofibers significantly influenced the latter's morphology, adhesion strength, and electrical resistance. As the concentration of MOF crystals increased, the diameter of the obtained fibers also increased, while the electrical resistance decreased as the thickness of the conductive armature increased. Filtration efficiency tests were conducted with the use of air polluted with dust particles $PM_{2.5}$ (~500 $\mu g \cdot m^{-3}$) and $PM_{10-2.5}$ (~1000 $\mu g \cdot m^{-3}$) and incense smoke containing $PM_{2.5}$ (~1500 $\mu g \cdot m^{-3}$) and $PM_{10-2.5}$ (~1000 $\mu g \cdot m^{-3}$). The particulate filtration efficiency from the air for both membranes was similar: >99%. The composite membrane, however, was a better filter for particulate matter from incense smoke than the pure PAN mat. In this case, the former's capture efficiency was 79% for $PM_{2.5}$ particles, and 97% for $PM_{10-2.5}$, while for the pure PAN membrane, it was 59% and 90%, respectively. This was due to the fact that the oil droplets from incense burning had a higher affinity for MOF than for PAN nanofibers. Additionally, the electric resistance of the composite membrane changed with the change in the humid air flow rate, which makes this membrane serviceable for breath monitoring.[76]

The usefulness of such face masks should be as high as possible, so it would be good if their protective and filtering properties lasted longer and not were limited to a single use. However, for a given mask to be used again, it must be adequately prepared in advance to rid it of previously captured impurities. Mamun *et al.* verified whether the masks based on PAN nanofibers were reusable after washing them in a home washing machine.[86] Scientists produced electrospun PAN mats on a polypropylene substrate, which were then covered with another layer of PP, as is the case in a standard mask, and then tested their filtering capacity following the EN143 standard for respiratory masks. Filtration efficiency was retested after the composites were washed in a domestic washing machine at different temperatures [40 °C (short program), 40 °C, 60 °C, and 95 °C]. Air permeability and resistance to evaporation were also tested. Images from a Confocal Laser Scanning Microscope (CLSM) showed that washing actually did not change the surface of nanofiber membranes at all. It was found, however, that this process affects air permeability and resistance to evaporation. As the washing temperature increases, air permeability increases, while the resistance to evaporation decreases. These changes, however, are so small that the effect of washing on air permeability and evaporation of the mats is negligible, thus making them reusable. Before washing, PP/PAN nanofibers/PP composites showed a filtration efficiency of 93.9%, while after washing at 60 °C and 95 °C, filtration efficiency was 86.1% and 88.6%, respectively. Therefore, their performance drops somewhat, but still remains high. There is a need for further research in this area, but it seems that washing masks in a domestic washing machine is an effective and promising method for achieving reusability.[86]

Polyfluoroolefins

Polyfluoroolefins also represent vinyl polymers, i.e. polymers formed due to combining monomers containing -C=C- double bonds. Their backbones consist of carbon atoms linked by single -C-C- bonds and hydrogen and fluorine atoms.

Polyvinylidene fluoride (PVDF) is a thermoplastic polymer with a low chemical reactivity. Due to the smooth structure of nanofibers, uniform pore structure, and easy bonding with PET nanofibers, Ullah *et al.* chose PVDF to evaluate the reusability of filter membranes after an ethanol cleaning process.[42] These researchers compared a typical melt-blown (MB) filter with PP found in N95 masks and a filter made of PVDF nanofibers (NF) supported by PET (weight PET/weight PVDF=98:2). The changes after cleaning in the filtration efficiency, morphological properties, and air permeability of both filters were assessed. Two different cleaning procedures were followed: in the first, membrane samples were immersed, and in the second were sprayed, with 75% ethanol. The porosity of the MB filter (~96%) was higher than that of the NF filter (~80%), which probably resulted in twice better air permeability of the MB filter than that of the NF filter, before and after alcohol cleaning. Nevertheless, both membranes met the requirements for face masks in terms of air permeability, pressure drop, and morphology. When used once, they also showed a high and comparable filtration efficiency. However, the MB filter efficiency after treatment with ethanol proved to drop to 64%, while the NF filter efficiency was kept high at ~97-99%, making its reuse possible (Figure 4d).[42]

Leung and Sun decided to develop a filter membrane composed of PVDF nanofibers capable of filtering COVID-19 virus particles.[95] Using the electrospinning technique, the Authors produced mats from electrostatically charged PVDF. The obtained samples were arranged in 2, 4, and 6-layer stacks, which increased the grammage of fibers in individual filtration membranes. Filtration efficiency tests were conducted using aerosols with a size of 20-300 nm, with the 100 nm aerosol being a representative example of an aerosol made of virus particles and carriers. The 50 nm particle size aerosol simulated the smallest possible aerosol event, and the 300 nm aerosol was selected for testing based on the standards set by NIOSH. The 6-layer membrane of charged PVDF nanofibers achieved a filtration efficiency of 88%, 88%, and 96% for the 50, 100, and 300 nm aerosols, and 92%, 94%, and 98%, respectively, when tested with monodisperse NaCl aerosols. The pressure drop was 26 Pa in each case, so the obtained filter was ten times more breathable than standard N95 masks. As the 300 nm NaCl aerosol test was 98% effective, the resulting filter could be qualified as an N98 respirator mask.[95]

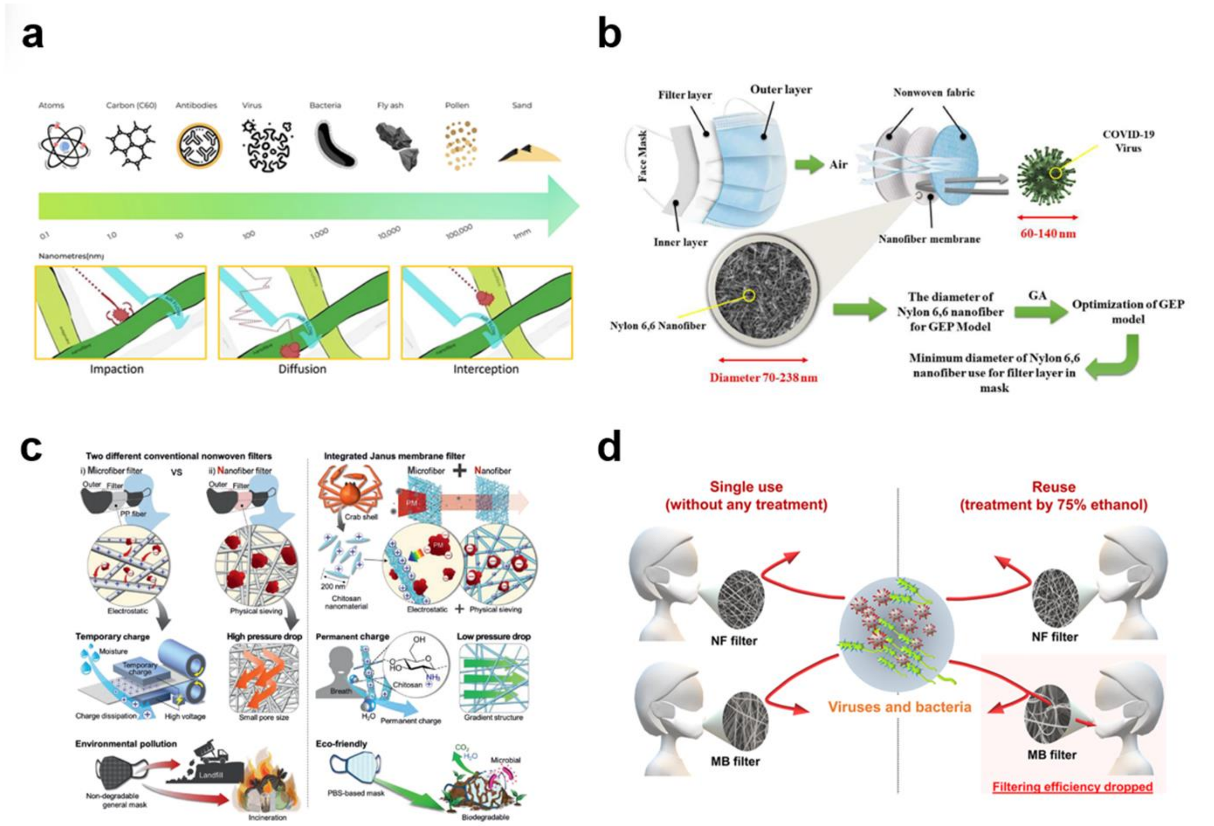

Figure 4. Passive filtering membranes. a) Comparison of the size of naturally occurring particles and illustration of possible filtration mechanisms in membrane systems made of electrospun nanofibers. Reproduced under terms of the CC-BY license.[83] Copyright 2021, The Authors, published by MDPI. b) Flowchart of the study for optimizing the nylon-6 electrospinning process using gene expression programming (GEP) and genetic algorithms (GA). Thus, the obtained nanofibrous membrane can capture coronavirus particles. Reproduced under terms of the CC-BY-NC-ND license.[87] Copyright 2021, The Authors, published by Elsevier. c) Comparison of particle capture mechanisms, user comfort, and environmental impact of conventional non-woven filters and the integrated Janus filter developed by S. Choi *et al.* The use of conventional filters (left) results in a temporary charge or high-pressure drop and, as a disposable product, pollutes the environment. The filter based on PBS nanofibers with chitosan (right) has a durable charge, causes low-pressure drops, and is biodegradable. Reproduced under terms of the CC-BY license.[94] Copyright 2021, The Authors, published by Wiley-VCH. d) Change in the filtration efficiency of the melt-blown (MB) filter and the

nanofiber filter (NF) before and after ethanol treatment. The filtration efficiency of the NF filter does not change with cleaning, making it possible to reuse it, while the MB filter can only be used once. This article is made available via the ACS COVID-19 subset for unrestricted RESEARCH reuse and analyses in any form or by any means with acknowledgement of the original source. These permissions are granted for the duration of the World Health Organization (WHO) declaration of COVID-19 as a global pandemic.[42] Copyright 2020, American Chemical Society.

ACTIVE NANOFIBROUS STRUCTURES FOR FACE MASK APPLICATION

Substantial efforts to produce higher-performance masks and respirators have been reported a number of times over the past few years. These include developing novel technologies to produce new or modified filter pieces and improving manufacturing protocols. Recently, researchers in the field of protection masks are working to include additional features in face masks, enabling an active filtration in protective equipment. This section focuses on the development of novel active filtering membranes that maintain constantly active properties over time.[103]

Active thermal dissipation for wearability and comfort optimization

Wearing face masks remains the most effective protective measure against COVID-19 infection even after mass vaccination, but an inadequate comfort and low antibacterial/antiviral activities accelerate the frequency of surgical mask replacement, resulting in large amounts of medical waste. Several researchers have developed electrospun nanofiber layered masks with excellent wearing comfort to help solve this problem.

Xiong *et al.* proposed fabricating masks from electrospun fluorinated carbon nanofibers/carbon fibers.[103] These fibers were used to replace commercially available polypropylene nonwovens (PP) as the core layer in face masks. The through-plane and in-plane thermal conductivities of commercial PP nonwovens were only 0.12 and 0.20 $W \cdot m^{-1} \cdot K^{-1}$, but the fabricated nanofiber membranes achieved 0.62 and 5.23 $W \cdot m^{-1} \cdot K^{-1}$, representing an improvement of 380% and 2523%, respectively, providing better heat dissipation (Figure 5a).[103]

Yang *et al.* fabricated a highly breathable and thermally comfortable face mask combining the asymmetric wettable skin layer with the nanofiber membrane via the electrospinning technique.[104] In electrospinning, the electret process and fiber construction could be integrated into one process, as the Authors demonstrated. Thus a desired composite face mask with a hierarchical structure and decent filtration performance was fabricated by combining electrospun nanofibers and nanoparticles. However, practically no effort has been made to investigate the wearing comfort of an electrospun composite membrane, and especially the relationship between membrane structure and wearability. To create a multilayer structure for filter application, Yang and *et al.* synthesized an asymmetric wettable skin layer made of an electrospun PAN/polyetherimide membrane with high $PM_{2.5}$ removal efficiency, desirable air permeability, and good radiation cooling properties. A low air permeability resistance and a uniform directional moisture transfer were observed for the composite membrane with a wetting gradient throughout its thickness. In addition, the multilayer structured nanofibrous membrane with outstanding radiative cooling is expected to significantly improve heat dissipation under extreme environmental conditions.[104]

Xiong *et al.* fabricated a novel face mask with excellent wearing comfort consisting of PP ultrafine fiber nonwovens and antibacterial functionalized nanoparticles of hexagonal boron nitride (hBN).[105] After the antibacterial functionalization of h-BN, a simultaneous improvement in the thermal conductivity and antibacterial activity of PP ultrafine fiber nonwovens was observed. Organic chains of quaternary ammonium salt (QAC) with an extended spectrum of antibacterial activities were immobilized on the surface of the hydroxylation of h-BN nanoparticles by forming covalent bonds. The activated PP ultrafine fibrous webs were then impregnated with the QAC/h-BN nanoplatelet suspension. QAC/h-BN/PP nanocomposite fiber membranes overcome the poor thermal comfort and antibacterial activity issues inherent in commercial PP nonwovens. The Authors claimed that the new QAC/h-BN/PP nanocomposite fiber membranes could find application in a broad range of personal health protective equipment.[105]

Charge surface improvement for higher filtration performance

Electret technology mitigates the conflict between filtration efficiency and pressure drop by providing fibers with an electrostatic effect that contributes to particle adsorption while generating negligible airflow effects. Compared to current electret technologies, electrospinning can perform the *in situ* charging and fiber formation, by making it possible to produce electret fibers in one step.

Li *et al.* fabricated hybrid electret PS/PVDF electrospun nanofibers by investigating the supplementation of electrical responses between polymer hybrid nanofibers.[106] The crossbreeding of the electrical polarization performances of PS and PVDF, two different polymers with low and high dielectric constants, led to the hybrid PS/PVDF nanofiber architecture, which showed an improved performance of electret effect and higher porosity (Figure 5b). The fabricated face masks, including PS/PVDF hybrid nanofibers as the central layer in the N95 facepiece, had a higher filtration performance of 99.8%, a lower pressure drop of 72 Pa, and a longer mean life.[106]

Wang *et al.* developed a hybrid face mask consisting of PAN electrospun nanofibers for air filters, by refining the design of the existing surgical masks.[107] The structure of the proposed face mask consisted of a PAN nanofibrous layer over the face mask, to be covered by a bilayer of PEDOT:PSS and polypropylene. As a result of their sandwich structure, the obtained masks achieved high filtration efficiency, with a filtration performance increase in the particle with a size range between 11.5 nm and 2.5 μm. In addition, with the aid of a tribo-charge applied via the PEDOT:PSS coated PP layer, capture efficiency increased by 7%. Stability and significantly extended mean life were validated by interception efficiencies of 94% ($PM_{0.25}$) and 99% ($PM_{2.5}$), without deterioration after two days. Lastly, an effective sterilization of the face mask surface could also be achieved with the aid of the generated tribo-charges.[107]

The human body is a great energy source, and the mechanical energy generated by body movements such as walking and inhalation is sufficient to power many wearable devices. Flexible electret nanogenerators with the primary operating mechanism of electrostatic induction caused by excess charges in the electret materials are characterized by considerable output power and a wide range of applications.

Cheng *et al.* presented a self-driven smart face mask consisting of a sandwich structure using electrospun polyetherimide fibers as an active electret material.[108] As a result of the excess charges retained in the nonwoven polyetherimide fabric, this smart face mask effectively removes particles during breathing and collects the energy generated during the outbreath. The fabricated electrospun layer had a high filtration efficiency for submicron particles (99.6%) and a correspondingly low-pressure drop (10 Pa). The smart mask can act as a stand-alone sensor to detect the breathing rate; the durability of the smart face mask is outstanding, being able to work continuously for 40 hours.[108]

Antiviral activities of functional membranes

The rapid advancement of disinfecting materials and their incorporation into personal protective equipment is a critical necessity that will significantly contain the spread of the SARS-CoV-2 virus and other pathogens. Extensive research has been conducted to prepare antiviral materials for face masks, such as inorganic or organic nanoparticles incorporated into electrospun nanofibers.[16,45,109–113] Antimicrobial face masks activated by photo/electrothermal or photosensitizing agents have shown a high and functional inactivation of viruses and long durability. There are several different methods for effectively disinfecting material surfaces to prevent the transmission of pathogenic microbes, especially viruses, during pandemics. Most of the available disinfection procedures today require several steps, and cannot be easily applied to the face masks that are currently available.

Abulikemu *et al.* investigated *in situ* assemblies of antiviral metal nanoparticles on a rigid surface and commercial nonwoven and woven textile face masks (Figure 5c).[45] By taking advantage of blade coating, Ag nanoparticles were synthetized in-situ on a glass substrate and then transferred onto the surface of face masks with a roll-to-roll technique. The results showed a disinfection performance of 99.98% on the face masks and 99.99% on glass substrates against a surrogate virus of SARS-CoV-2.[45]

In another study, Hashmi *et al.* focused on electrospun nanofibers of PAN using copper oxide for antiviral applications in respiratory filters.[109] Copper(II) oxide is hydrophilic and has potential applications for magnetic storage purposes. Authors claimed the first-time use of copper oxide nanoparticles for antiviral respiratory masks. The membranes were fabricated by electrospinning a nanocomposite solution of PAN and copper oxide nanoparticles in different concentrations. The fabricated nanofibrous mat showed an antiviral activity as well as release properties. MTT analysis also showed that more than 50% of the total cells survived after 120 h of incubation. Copper oxide nanoparticles also improved the tensile strength (8.45 MPa) of the produced nanofibrous membrane. However, the Authors did not provide data on virus removal efficiency and pressure drop.[109]

Khanzada *et al.* demonstrated the antiviral activity of aloe vera (AV) incorporated within polyvinyl alcohol (PVA) electrospun nanofibers for protective clothing.[110] This research focused on developing electrospun AV/PVA nanofibers using electrospinning to form a cross-linked PVA/AV nanofibrous membrane. The study of the AV release supported the Authors` claim regarding the serviceability of the protective clothing produced. Furthermore, the antiviral activity of the electrospun nanofibers was used to study the effect of the proposed nanofibrous membranes. The results showed a valid antiviral activity in different concentrations of AV incorporated into PVA.[110] The Authors did not report data on antiviral efficacy and pressure drop.

Nanostructured face masks with antibacterial properties

Research on nanofibrous materials for active face masks has improved various possible features. One of the methods for increasing the effectiveness of face masks is to reduce the activity of the bacteria filtered through the layers of face masks actively over time.

Pardo-Figuerez *et al.* fabricated face masks using PAN nanofibers in the middle layer of masks to improve their filtering capacity.[114] The implementation of ZnO in the PAN matrix produced nanocomposite fibers, resulting in solid antimicrobial properties of face masks against *E. coli* and *S. aureus*. The highest reduction (R>3) against both types of bacteria was observed in the case of the PAN-ZnO at 3 wt.%. As for filtration capacity, ZnO nanoparticles did not affect filtration performance, and although they slightly increased breathing resistance, the values obtained remained low and in line with the required European standard EN149 for FFP2 type PPE.[114]

Abbas *et al.* proposed a degradable multifunctional hybrid composite consisting of three electrospun nanofiber layers as a filter material, offering the potential to solve some of the challenges presented by face mask filters.[115] The outer active layer of the filter, a composite of TiO_2 nanotubes incorporated into the electrospun nanofiber matrix of chitosan (Cs)/PVA, acted as an antibacterial agent. The middle layer is (Cs)/PVA, which also serves to achieve natural air filtration and the inactivation of pathogens. The inner layer of the face mask filter is made of silk/PVA nanofibers, which improve the filter mechanical properties and heat dissipation, while offering the wearer greater skin comfort. The filtration efficiency of these face masks increased by about 20% compared to commercial competitors. Furthermore, *in vitro* tests showed the efficient role played by TiO_2 nanotubes, and Cs nanofibers in eliminating pathogenic bacteria.[115]

He *et al.* fabricated nanofibrous membranes with an enhanced filtration performance and solid antibacterial properties using bromosalicylic acid (BSA)/polyvinyl butyral (PVB) and electrospinning technology.[116] Figure 5d shows a diagram of the design and construction of an antibacterial face mask. The incorporation of BSA in the membrane effectively eliminated 99.1% of both gram-positive and gram-negative *E. coli* and *S. aureus* in less than eight hours. The Authors also investigated the germ-killing activity of the membrane produced, which showed to have a 99.9% germ-killing efficiency. It is noteworthy that the Authors used BSA and PVB as green polymers and selected eco-friendly solvents and techniques.[116]

Hiragond *et al.* enhanced the quality of commercial face masks in terms of their antibacterial performance, by treating masks with silver (Ag) nanoparticles.[117] First, the Authors synthesized a starch-stabilized colloidal stock solution of Ag nanoparticles. Surgical masks were then treated with colloidal Ag nanoparticle solutions. The antibacterial activity of treated and untreated masks was tested against both gram-positive and gram-negative bacteria via the inhibition of bacteria growth method.[117]

Salam *et al.* developed antiviral and antibacterial electrospun nanofiber membranes by incorporating HeiQ Viroblock (VB) and ZnO nanoparticles into a polyacrylonitrile (PAN) electrospinning solution. The encapsulated electrospun nanofibers can be used for protective clothing against bacteria (both gram-negative and gram-positive).[118]

Patil *et al.* fabricated a cotton/PLA-based biodegradable mask with improved antibacterial and respiration properties.[119] Their study focused on the fabrication of a three-layer mask whose upper and lower layers were made of cotton, and the middle layer was made of an electrospun nanofibrous PLA functionalized with traditional Indian herbal extracts. The use of traditional herbal extracts effectively neutralizes the effect of bacteria. It is known that they are sources of various substances, many of which have antimicrobial and radical-scavenging properties. The computational analysis of phytochemicals using the advanced scoring function showed the improved performance of herbal extracts in neutralizing bacteria. The results obtained from bacterial filtration efficiency (BFE) showed a noticeable efficiency of 97.9%. Furthermore, the achievement of 35.78 Pa·cm^{-2} in the differential pressure of the masks confirmed the study's success.[119]

Zhang *et al.* fabricated a nanofiber membrane based on water-soluble PVA loaded with drugs to endow it with drug-loaded antibacterial properties using electrospinning.[120] These membranes successfully eliminated *S. aureus* and *E. coli*. The membrane's filtration performance was improved by 80-87%, depending on the width of the electrospun layer compared to commercial surgical masks.[120]

Alshabanah *et al.* developed an antimicrobial membrane of electrospun biodegradable and synthetic nanofibers that could be used as a functional layer in PPE.[121] The developed nanofibers were made of PVA and thermoplastic polyurethane – TPU, loaded with Ag nanoparticles, and characterized and evaluated for their antiviral and antibacterial activities. Tests were performed using several viruses, including SARS-CoV-2, and several strains of bacteria, including *S. aureus, E. coli, Acinetobacter,* and *Klebsiella-Pneumoniae.* The results confirmed the successful development of antimicrobial materials.[121]

Qin *et al.* produced an antibacterial electrospun membrane with a mixture of PVB and berberine hydrochloride (BH).[122] The Authors investigated the effects of BH concentration on the fabricated membranes, including morphology, antibacterial properties, and filtration efficiency. The reported filtration efficiency of the membrane reached 96.4% for $PM_{0.3}$ and 100% for $PM_{2.5}$, with a low-pressure drop of 108 Pa. The antibacterial examination of membranes for *S. aureus* showed an effective inhibition zone of the membranes against bacteria.[122]

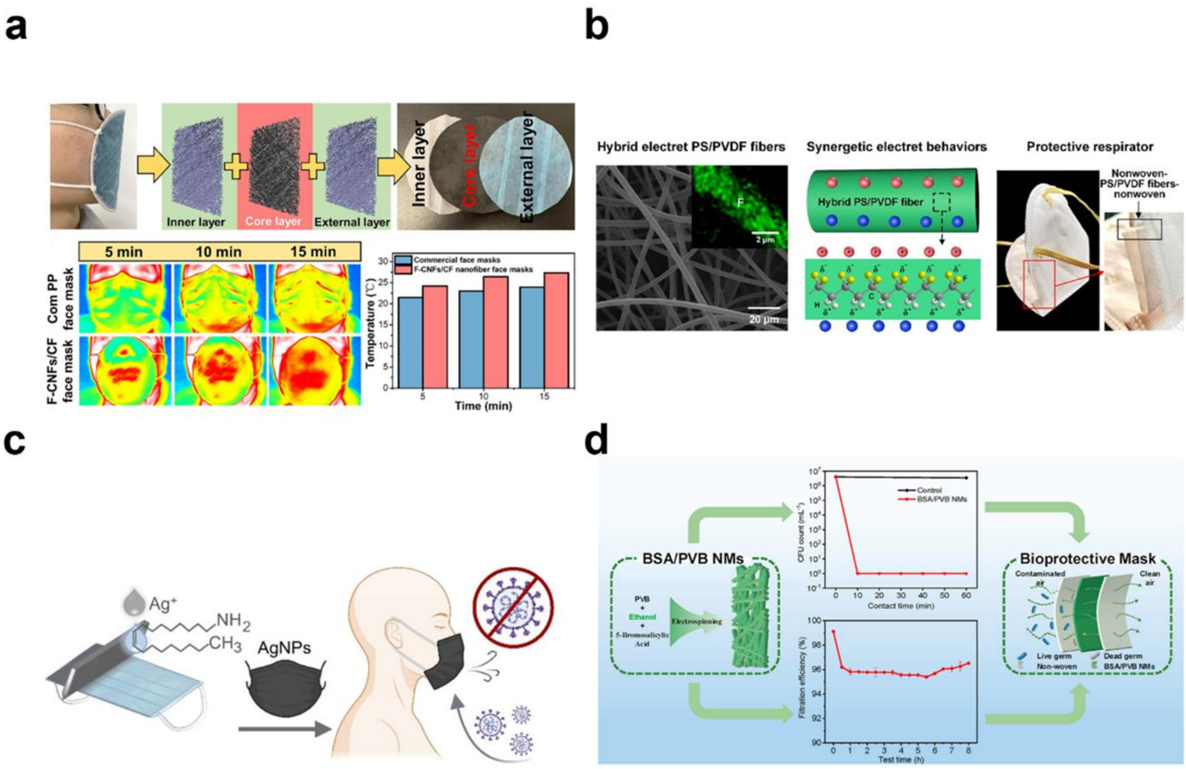

Figure 5. Various strategies applied to broaden the applications of respirators by enhancing and/or adding novel features. a) Structure of nanocomposite fiber masks made of commercially available PP surgical masks and F-CNFs/CF nanocomposite fibers as well as the thermal analysis results obtained for the fibrous masks. Reproduced with permission.[103] Copyright 2022, Elsevier. b) SEM image of the electret PS/PVDF-2 (the inset shows an elemental distribution map of fluorine atoms into a single electrospun fiber collected by energy-dispersive X-ray spectroscopy). Schematic representation of the contribution of PVDF to the electret effect. An N95 respirator with PS/PVDF-2 fibers as its core material. Reproduced with permission.[106] Copyright 2020, Elsevier. c) Schematic representation of the coating procedure on a face mask with functionalized silver nanoparticles to inhibit viral infectivity and the final PPE application. Reproduced with permission.[113] Copyright 2021, American Chemical Society. d) Schematic illustration of the design and fabrication of BSA/PVB NMs. Bactericidal activities of control and BSA/PVB NMs against *E. coli* bacteria. Long-term filtration performance of the

BSA/PVB NMs and a schematic representation of the composite mask. Reproduced with permission.[116] Copyright 2021, Elsevier.

STIMULI-RESPONSIVE AND MULTIFUNCTIONAL FACE MASKS

Stimuli-responsive materials (SRMs) can be fabricated or integrated to enhance the function of multifunctional devices that possess desirable properties such as alertness to favorable stimuli.[123] When SRMs are exposed to external stimuli (e.g. temperature, light, and electromagnetic field), the molecular conformation, polarity, connectivity, or solubility changes from the initial state to a responsive form, resulting in a change in the materials' properties.[124] SRMs have been extensively studied as components in wearable devices, including sensors, actuators, drug delivery systems, and self-repairable devices. Despite their ability to improve protective properties, the study of SRMs for use in smart face masks is still in the early stages, as their development and applicability face many challenges.

Light-responsive face masks

Disposable surgical masks can prevent environmental pollutants and other people's respiratory droplets from entering our respiratory system and help reduce infection risk. However, there are limitations to the existing masks. A non-trivial issue is that although the surfaces of surgical masks are hydrophobic, water droplets containing dangerous viruses may remain on them.[125] To overcome this concern, employing SRMs to provide a self-cleanable surface can be an excellent strategy (Figure 6a). Consequently, easily sterilizable and reusable face masks will offer a new deterrent against spreading pathogens and aid in the reduction of the influence of pandemic situations in the future.[126]

SARS-CoV-2 could be inactivated if exposed to a temperature of 56 °C for 15 minutes. One of the most widely available sources of energy to humans is solar radiation, a portion of electromagnetic radiation containing infrared, visible, and ultraviolet light. By taking advantage of the broad-spectrum absorption properties of SRMs, it is expected that the temperature necessary to inactivate pathogens can be achieved by irradiating photothermal responsive material with sunlight. Soni *et al.* produced a spray-coated layer of carbon nanotubes (CNT) on the outer layer of the melt-blown face mask.[127] The produced layer formed a superhydrophobic coating which increased the water contact angle of the surgical face mask from 113.6°±3.0° to 156.2°±1.8°. CNT-coated surgical masks also showed an outstanding photothermal response by increasing their surface temperature upon irradiation. The reported ΔT was higher than 55 °C within 30 seconds after exposure to sunlight. This increase in temperature leads to 99.9% elimination of *E. coli* compared to pristine conditions. These results confirm the ability to self-sterilize face masks by using SRMs.[127]

A self-sterilizing, recyclable, and high-performance face mask based on electrospinning was fabricated by Xiong *et al.*[128] The Authors developed a needleless-electrospinning/spraying-netting technology to create a nanofibrous network. The proposed nanoarchitecture consists of a PAN nanofiber skeleton as a framework to maintain a network made of CNT@polyvinylpyrrolidone (FVPV) as the active part (Figure 6b). This structure enabled a consistent and rapid photothermal sterilization (in 5 min) under solar radiation and an electrothermal self-sterilization (in 2 min) in sunless environments. The results demonstrated the potential to reduce both the consumption of resources to combat disease transmission and the severe environmental impact of mask waste.[128]

Li *et al.* enhanced a prototype face mask by the ability of photocatalysis to sterilize bacteria on the surface of the mask by light irradiation.[129] Instead of PP, the Authors initially used PVA, PEO, and nanocellulose, which are biodegradable polymers. The biodegradability of these polymers can reduce the environmental pollution caused by present-day masks made of synthetic plastics. The next step taken by the Authors was to use electrospinning to produce composites with porous structures, which give the mask improved breathability and filtration. The Authors later claimed that nanocellulose abundantly formed hydrogen bonds with PVA and PEO, while PEO served as a plasticizer in the electrospinning dopes. Both effects significantly improved the electrospinnability and mechanical performance of the resulting facepieces. Afterwards, by introducing TiO_2 nanoparticles into the structure of the nanofibers, photosensitivity was also achieved in the face mask prototype. The resulting nanofibrous face masks showed exceptional disinfection features against *E. coli* and *S. aureus* under sunlight, a considerable advancement in the technologies for producing the next generation face masks.[129]

Li *et al.* created a nanofiber membrane containing the electroactive polymer of poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) as the matrix for electrospinning and the photosensitizer Rose Bengal (RB) as the dopant.[130] The multilayer porous structure of nanofibers with dopant promoted the trapping of pathogenic droplets and aerosols. Upon sunlight irradiation for 5-10 min, the fabricated membrane generated a considerable amount of reactive oxygen species (ROS). The developed device showed a powerful inactivation by sunlight against various microbes (e.g. *S. aureus, E. coli, C. Albicans,* and *S. cerevisiae*).[130]

The impact of the rise in particulate matter index levels in the urban environment has driven the development of face masks toward novel targets. In addition, another environmental aspect, ultraviolet radiation (UV), is considered a significant risk factor; indeed, the UV index has been increasing continuously in recent years, with harmful effects on human health. Nevertheless, none of the filter materials or face masks have taken this problem into account.

To combat fine flying ashes and UV radiation, Chen *et al.* explored a class of electrospun nanofibers employing SRMs.[131] The Authors constructed a filter made of electrospun PAN nanofibers filled with TiO_2. The induced antibacterial photocatalytic effect of the masks produced is achieved by UV light irradiation; bacteria such as *E. coli* and *staphylococcus* decomposed within a few hours of radiation. Thus the prevention or reduction of UV irradiation and the deodorizing and/or antibacterial ability were simultaneously achieved by simply adding

TiO_2 into the filter material, where TiO_2 has the function of decomposing bacteria and blocking UV rays. Moreover, the presence of TiO_2 beads demonstrated a beneficial impact on filtration properties, in addition to photocatalytic and antibacterial activity.[131]

The disposable face masks currently available can neither inactivate pathogens nor eliminate viruses. Therefore, easily sterilizable and reusable masks with antiviral properties could be an effective prevention tool against the spread of pathogens and, especially, respiratory diseases.

Horváth *et al.* developed filters and facepieces based on TiO_2 nanowires (TiO_2 NWs).[126] The proposed PPE is made of a layer of TiO_2 NWs of a desirable thickness, affording different physical and chemical properties. The high efficiency of the fabricated filters is achieved thanks to the photogeneration of ROS under UV illumination. The Authors estimated that a light-sterilizable mask based on the mentioned TiO_2 NWs could be reused more than 1,000 times.[126]

De Sio *et al.* conceptualized a next-generation face mask with photoactive properties (Figure 6c).[15] By drop-casting, a water dispersion of AuNRs can be used to create a uniform layer of plasmonic nanoparticles on the surface of face masks. AuNRs can absorb near-infrared light, which subsequently lead to an electron excitation of AuNRs. The proposed layer over the face mask can be quickly activated on demand by light, to destroy pathogens and desorb the water entrapped between the human skin and the face mask, thus enhancing the equipment's wearability.[15]

Impact of charge on the smart features of responsive nanofibrous face masks

To avoid using expensive and non-portable disinfection devices, surgical masks with functions such as a comfortable fit and rapid self-disinfection have been developed by changing the structure of surgical masks.

Wang *et al.* fabricated an electret electrospun polyethersulfone/barium nanofibrous composite membrane with enhanced breathability and cooling properties.[132] The adjustment of morphology, porous structure, air penetrability, and water vapor transmittance rate (WVTR) was tuned by the concentration of nanoparticles. The comprehensive filtration measurements of the resulting membrane showed outstanding breathability. The polarization of $BaTiO_3$ nanoparticles conferred high injection energy and enhanced charge storage stability to the membrane, making it possible for the electret filter to trap $PM_{2.5}$ by electrostatic attraction. The performance of the membrane was unchanged even after being treated at 200 °C for 45 min. In addition, the composite nanofibrous membrane with remarkable cooling properties improved the thermal radiation dissipation even at abnormal temperatures. Furthermore, the membrane showed a high filtration efficiency (99.9%) and a lower pressure drop of 67 Pa with the proposed structure.[132]

Piezoelectric materials have been introduced into the structure of nanofibrous filters to improve the filtration efficiency without the polarization of nanofibers or using the energy of the quasi-permanent charge. Several research groups have designed nanofibrous membranes by electrospinning piezoelectric polyvinylidene difluoride (PVDF) to benefit from electric charges on the filter surface.

Kang *et al.* also studied an electrostatic air filter for face masks that exhibited self-electrostatic charge generation and retention properties under cyclic air blowing and moist air infiltration.[133] Bending and vibration can even enhance the electrostatic charge density of the nanonet structure on the filter. By taking advantage of triboelectric charges, the proposed nanofibrous filter was used for fine dust filtration. A membrane was fabricated by electrospinning PVDF on a nylon network layer to preserve the nanofibrous structure (Figure 6d). The number of nylon networks of the filter membrane was adjusted to increase the PM filtration efficiency and improve the pressure drop. A three-layer electrospun filter structure has improved electrostatic charge generation in an environment that mimics breathing, a beneficial feature for face masks. The Authors mentioned that a filter mask could generate electrostatic charges during breathing due to the unique multilayer structure of the membrane, overcoming the current limitation in PM filtration of face masks.[133]

Le *et al.* developed a novel smart face mask based on the concept that poly-L-lactic acid (PLLA) electrospun nanofibers can generate a piezoelectric charge when an airflow (such as human breathing) is applied.[134] This piezoelectric charge creates an electrostatic shielding layer that prevents the penetration of particles or pollutant droplets. The piezoelectric effect improves the removal of particulate matter by enhancing electrostatic charge adsorption and enables the PLLA piezoelectric nanofiber filter to overcome the existing problems of conventional face masks, such as moisture susceptibility, non-degradability, and poor reusability. The PLLA nanofiber filter can also be sterilized by standard means such as an ultrasonic bath or autoclave, making it possible for users to disinfect and reuse the filter. In addition, Le *et al.* created a prototype of a biodegradable face mask made of the piezoelectric PLLA nanofibrous membrane. This filter has a high filtration performance (99% for $PM_{2.5}$ and 91% for $PM_{1.0}$), while offering a promising pressure drop (≈91 Pa at regular inhalation rate) for human respiration, thanks to the piezoelectric charge that is naturally stimulated by breathing through the face mask. The Authors also mentioned that the electrospun PLLA piezoelectric membrane could be cost-effective compared to N95 respirators and surgical masks due to the reusability of the masks.[134]

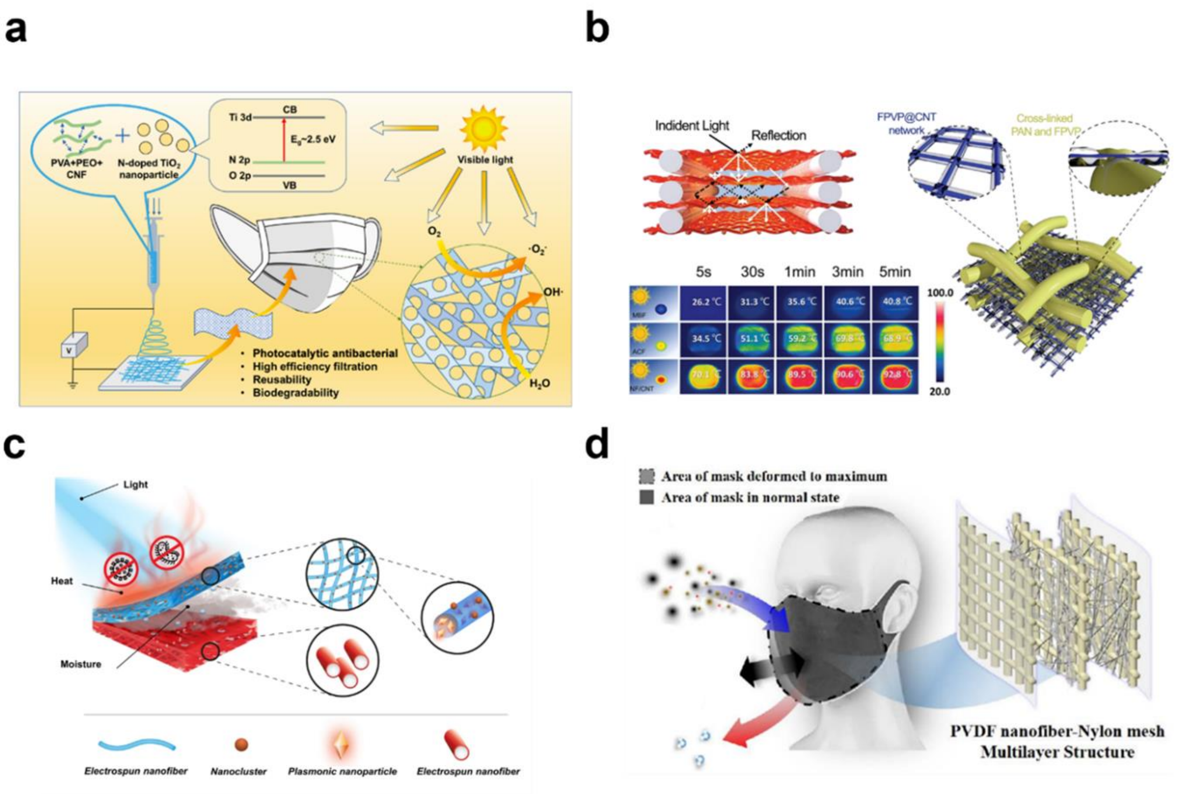

Figure 6. Different approaches developed for fabricating stimuli-responsive face masks. a) Reusable masks with a functional design responsive to visible light. The nanofiber structure fabricated by electrospinning affords excellent breathability and particle filterability. N-TiO_2 nanoparticles efficiently activate the mask upon irradiation with IR light to destroy bacteria. Reproduced with permission.[131] Copyright 2021, American Chemical Society. b) Schematics of the incident light propagation through the nanofibrous network and the architecture of the proposed nanofibrous network. Thermal responses of the nanofibrous masks under different radiation conditions. Reproduced with permission.[128] Copyright 2021, Wiley-VCH. c) The sketch shows the morphology of a multifunctional light-responsive electrospun membrane for smart filtration. Light irradiation can trigger the active layer on demand to destroy pathogens and dissipate the trapped moisture, while the electrospun layer is an effective filtering and desiccant membrane. Reproduced with permission.[15] Copyright 2021, Wiley-VCH. d) Scheme showing the electrostatic charge retention membrane features used by an electrospun PVDF

layer inserted into a filter membrane multilayer structure. Reproduced with permission.[133] Copyright 2021, MDPI.

FUTURE PERSPECTIVE AND CHALLENGES

Face masks have played a pivotal role in the recent COVID-19 pandemic, and we have learned that although they are crucial to protecting people's lives, they also have a variety of downsides.[135] Before a new pandemic arrives, it is absolutely necessary for fundamental/advanced research and the relevant industries to engage in synergistic cooperation, in order to develop innovative solutions that can be tested, scaled up, and manufactured in time.

In the past two years, scientists worldwide have begun to focus on ideas for creative and ingenious face masks that can offer novel and exciting solutions. Innovative textile materials, bio-polymers, nanostructured materials, 3D bioprinting, artificial intelligence, and stimuli-responsive nanoparticles are now considered the prime ingredients for tackling the challenge of producing intelligent, reusable face masks in the coming years. In all preliminary studies, it appears that this new direction can offer brilliant opportunities that must be investigated further .[136]

Cross-disciplinary expertise, international research projects, young researchers' exchange programs, and bi- and multilateral agreements between universities and research centers seem to be strong, galvanizing approaches that can contribute to speeding up innovation. However, scientists and researchers cannot be left alone in this race. The support of governments, international organizations, and industrial partners is still a crucial aspect for supporting and scaling up the most promising new technologies developed so far.

Global climate change, including global warming, is already affecting and modifying our environment and biodiversity. It is time to do an about-face and start adopting meaningful actions to preserve our health and that of the next generations. For these reasons, it is necessary to rethink the materials and the environmental impact of the production of innovative biomedical devices such as protective face masks. From this standpoint, biodegradable and compostable materials[137] must be adopted as a ”gold standard” for making lightweight, comfortable, eco-friendly face masks. The elastic loops connected to face masks to hold them in place produce severe ear fatigue and rub the skin. Ear protection mask extensions made of innovative, flexible and hypoallergenic materials are needed to reduce or minimize face mask discomfort. Advanced and high-precision technological processes such as 3D printing,[138] electrospinning,[139] and nanofabrication[140] must be considered among the preferential pathways for providing face masks with high filtration power, comfort, and innovative designs. Digital twin technology for intelligent manufacturing can offer original ways to solve challenges connected with customization. Indeed, optimized facial comfort can be improved by combining 3D printing and Direct Digital Manufacturing.[141] For example, specific designs can be obtained from a library of 3D heads from 3D scans of the heads and necks of healthcare professionals.

Green and exotic nanomaterials, primarily derived from biowaste, have to be extensively explored to include stimuli-responsive capabilities such as photo-thermal and photocatalytic disinfection, thus providing long-term reusability. Colorimetric and ultra-sensitive biosensors that can measure the presence of harmful pathogens in the environment and are able to send remote alert signals to competent authorities can play a crucial role if adequately integrated into the newly produced face masks. Indeed, this innovative function can help identify and isolate specific areas where risks are much higher. Moreover, the integration of multiple sensors can turn new-generation face masks into smart devices for health monitoring.

The socio-economic impact of COVID-19 on young people has produced countless moral and material damages, forcing them to live in an isolated and virtual world. The coming generations deserve a better world, and the role that science and technology can play now is vital to prevent future pandemics from producing the same effects. Envisioning innovative face masks that can meet all the mandatory protection, customization, capabilities, and sustainability requirements is a fundamental step in the right direction. This technology can be easily extended to other valuable PPE for healthcare professionals and the general population, such as smart gloves, reusable gowns, and recyclable sanitary caps.

CONCLUSIONS

The use of personal protective equipment, including face masks, plays a vital role in numerous aspects of our lives, since an evident increase is taking place in the level of air pollution caused by particulate matter or commonly present bacteria and viruses. During the COVID-19 pandemic, face masks became an integral part of everyone's daily routine. New products of this type can protect us from the next pandemic or significantly reduce its spread worldwide.

The need to develop face masks with outstanding filtration efficiency, excellent mechanical properties, comfortable breathing, and reusability has become a primary target. Moreover, developing devices with unique user-friendly properties activated in response to specific stimuli has been recognized as the next step in the development of the next generation of face masks.

This review has classified and summarized the efforts of scientists to develop filters and masks based on electrospun nanofibers. It has been shown that nanofiber filters typically perform better than the previously-used melt-blown filters and, are therefore their promising alternative. Masks fabricated by electrospinning can be used many times, maintaining high filtration efficiency even after cleaning. In addition, and most importantly, these devices can be the building blocks of "smart" respirators thanks to the versatility of electrospun nanofibers.

The specific properties of various electrospinnable polymers make it possible to produce passive filters with particular abilities, such as controlled heat dissipation and breathing monitoring. However, this paper highlights the fact that researchers have proposed a new generation of face masks that perform better than the previous ones, because they can provide more benefits than does passive filtration.

Scientists working in the field of protective masks are focusing on including other components in filters in order to obtain active working filtration. This type of particle separation can be divided into two classes. In the first class, active nanofibers are used; therefore, there is no need for additional external intervention to force their specific action, but the performed activity is continuous, non-specific, and not triggerable upon request. In the second class of filters, their activation takes place on demand, i.e. under the influence of an external stimulus. It has been proven that enriching nanofiber materials with other particles can make them become active filters, increasing the usability of masks with their new properties, such as antibacterial or antiviral ones. Particular types of face masks can have the ability to store the energy generated by the wearer while breathing. This research provides a new high-efficiency "smart" air filter and demonstrates a unique and straightforward strategy for developing self-powered and multifunctional healthcare devices. "Smart" multifunctional filters can also make it possible for us to create features in addition to that of killing bacteria and viruses, such as sterilizing face masks after using them and responding to environmental or external stimuli, including light, temperature, pH, magnetic and electric field, and/or mechanical stimuli.

As reported in this review, scientists have recently become increasingly interested in studying filtration membranes that show specific effects in response to stimuli. Nevertheless, this topic has not been thoroughly investigated; therefore, more research is needed in this field to discover new possibilities of nanofiber filters based on the modification of their properties. Much effort must also be spent to translate fundamental research results into an actual application of the materials designed. The described approach is innovative and offers excellent, vast opportunities; therefore, it is possible for the new types of face masks to become commercial products of large-scale availability.

"Smart" face masks will benefit society as a whole, providing it with adequate protection against all kinds of pollution, while reducing the costs incurred up to now for purchasing disposable products. These materials will also significantly impact the environment because they do not need to be replaced often and are biodegradable.

ASSOCIATED CONTENT

Competing Financial Interest

The authors declare no competing financial interest

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ACKNOWLEDGMENTS

This work was supported by the National Science Centre (NCN) SONATA BIS Project No. 2020/38/E/ST5/00456. The Authors are grateful for the support provided by the National Agency for Academic Exchange (NAWA) grant no. PPI/APM/2018/1/00045/U/001. P.N. and F.P. acknowledge the financial support from the Polish Ministry of Science and Higher Education through scholarships for outstanding young scientists.

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