Topical Review

Toward nanotechnology-enabled face masks against SARS-CoV-2 and pandemic respiratory diseases

Nazek El-Atab^{1,*} , Rishabh B. Mishra^{1,2} and Muhammad M. Hussain^{2,3,*}

¹ Smart, Advanced Memory devices and Applications (SAMA) Lab, Electrical & Computer Engineering, King Abdullah University of Science and Technology (KAUST), Thuwal 23955-6900, Saudi Arabia

² MMH Labs, Electrical & Computer Engineering, King Abdullah University of Science and Technology (KAUST), Thuwal 23955-6900, Saudi Arabia

³ Electrical Engineering and Computer Sciences (EECS), University of California, Berkeley, CA 94720-1170, United States of America

E-mail: nazek.elatab@kaust.edu.sa and mmhussain@berkeley.edu

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Abstract

Wearing a face mask has become a necessity following the outbreak of the coronavirus (COVID-19) disease, where its effectiveness in containing the pandemic has been confirmed. Nevertheless, the pandemic has revealed major deficiencies in the ability to manufacture and ramp up worldwide production of efficient surgical-grade face masks. As a result, many researchers have focused their efforts on the development of low cost, smart and effective face covers. In this article, following a short introduction concerning face mask requirements, the different nanotechnology-enabled techniques for achieving better protection against the SARS-CoV-2 virus are reviewed, including the development of nanoporous and nanofibrous membranes in addition to triboelectric nanogenerators based masks, which can filter the virus using various mechanisms such as straining, electrostatic attraction and electrocution. The development of nanomaterials-based mask coatings to achieve virus repellent and sterilizing capabilities, including antiviral, hydrophobic and photothermal features are also discussed. Finally, the usability of nanotechnology-enabled face masks is discussed and compared with that of current commercial-grade N95 masks. To conclude, we highlight the challenges associated with the quick transfer of nanomaterials-enabled face masks and provide an overall outlook of the importance of nanotechnology in counteracting the COVID-19 and future pandemics.

Keywords: COVID-19, face mask, nanotechnology, nanomaterials, nanofibers, hydrophobic, electrospinning

(Some figures may appear in colour only in the online journal)

* Authors to whom any correspondence should be addressed.

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1. Introduction

In the last two decades, the world has witnessed multiple pandemic respiratory diseases, ranging from severe acute respiratory syndrome (SARS) and Swine Flu (H1N1) to the most recent coronavirus disease 2019 (COVID-19 or SARS-CoV-2 virus), where the latter showed higher infection rates

with serious medical complications. In fact, the COVID-19 disease was first reported to the World Health Organization (WHO) in Wuhan, China, in December 2019, where it was believed to be originally existing in a bat host and got transmitted to human beings via wet-market [1]. Due to the unprecedented disease transmission rate, almost 90% of the world's countries were affected by March 2020 [2]. While the virus is confirmed to be exceedingly infectious, most of the infected persons are asymptomatic. At the same time, the most common symptoms include high fever, dry cough, weakness and respiratory distress [3]. Existing studies have explained the virus transmission mechanism by respiratory droplets which carry the virus and which are generated when the infected person sneezes, coughs or even speaks. While the main reason for the virus spread is either direct contact with an infected person who is coughing and/or sneezing or transfers the virus to any surface, which might get transferred to another person [4, 5].

The respiratory droplets can be divided into two main categories based on their dimensions, where aerosols are sub-5 μm in size while droplets are larger than 5 μm . Due to the gravitational forces, droplets normally settle within 1–2 m, while the lighter aerosols can travel several meters for extended periods which can lead to a significant increase in the spread of the virus [6, 7]. Moreover, it has been shown that the symptoms can be more or less severe depending on the dose of the virus that the person has been exposed to. The minimum dose of virus that is necessary to make a certain person ill is known as the infectious dose. Thus, a virus having a low infectious disease can be very contagious especially in populations lacking a good immunity. The infectious dose of the COVID-19 virus is uncertain so far, however, it is believed to be low [8]. As a result, the demand on face masks has rapidly escalated due to its capability in achieving a barrier between the person and the floating respiratory droplets. In addition, the face masks contribute to lowering the probability of hand-to-mouth and hand-to-nose activities which can result in directly transmitting the SARS-CoV-2 virus into the human facial entry points. This has led to the worldwide adaptation of three main practices to fight against the pandemic: social distancing, maintaining the good hygiene of the hands and wearing face masks [9, 10].

As per the underlying precaution provided by WHO which recommends the usage of a facial mask to contain the COVID-19 pandemic, several countries have made it mandatory to wear a face mask in public areas [11, 12]. As a result, shortage in commercially available surgical-grade face masks occurred due to this increasing demand [13, 14]. To overcome this problem, people and researchers worked on developing homemade do-it-yourself (DIY) cloth masks to protect themselves even though the performance and efficiency of the different fabrics in filtering the virus are still being studied [15, 16]. To this end, Konda *et al* analyzed the filtration efficacy of different combinations of fabrics against particles with various dimensions [17]. The study showed that using multiple layers of hybrid fabrics such as cotton-silk, cotton-chiffon, etc., can achieve a filtration efficiency up to 90% for particles which are >300 nm in size while the filtration efficiency drops down to 80% for smaller particles (figure 1(a)). The good

performance of such hybrid masks is explained to be due to a combination of mechanical and electrostatic-based filtration. Moreover, the study shows that cotton, the most commonly available material, performs better when the thread count in the material is higher. Nevertheless, the loose-fitting property of the mask is found to reduce the filtration efficiency by over 60% which implies the need to better design future cloth-based masks to achieve proper fitting with the face.

Other researchers have focused on the development of reusable personal protective equipment (PPE) which could provide a protection level from COVID-19 that is comparable to that of the N95 masks and which can be easily sterilized and reused to overcome the shortage issue. To this end, Byrne *et al* developed a silicone-based mask using injection molding on an actual N95 mask. The mask includes two openings where replaceable N95 filters can be embedded as depicted in figure 1(b) [18]. It should be noted, however, that the presented mask requires less N95 filtering material than a conventional N95 mask. The new mask has been tested on 24 healthcare workers in terms of fitting and filters exchange. A 100% success rate is obtained where the mask is shown to be perfectly fitting different face shapes and sizes using an Occupational Safety and Health Administration (OSHA) approved testing technique. Additionally, the researchers studied three different sterilization techniques, including inserting them into a steam sterilizer, an oven, and dipping them into a solution of bleach and isopropyl alcohol. Mechanical tensile tests on the silicone masks confirmed the possibility of sterilizing the masks without mechanically damaging them. On the other hand, Matt Carney and his team developed a reusable silicon-based face mask using the 3D-printing technology [19]. The mask also includes an opening for adding replaceable N95 filters as shown in figure 1(c). The leaders of this project focused not only on achieving a good filtration efficiency, but also a good ability of releasing the residual CO_2 that a person exhales. The first model of the reusable PPE has already entered large scale production in several countries including the US, Colombia, Portugal and Brazil. Nevertheless, it should be noted that 3D printing is considered as a slower and less scalable technique compared with injection molding.

The commercially available face masks provide different levels of protection against the SARS-CoV-2 virus which is found to possess a spherical or elliptic shape with a size of 65–125 nm [20]. While the surgical-grade N95 mask can provide the highest protection level, its filtration efficiency against particles with sizes of 300 nm is 95% under test conditions. This efficiency is shown to get lower for smaller particles sizes. Moreover, most of the existing face masks are hydrophilic and inefficient when wet where their performance in repelling water droplets and allowing appropriate respiration degrades [21]. Another important characteristic of face masks is the breathability. In fact, even though the N95 masks pass the breathability test set by the United States National Institute for Occupational Safety and Health (NIOSH), where the mask must not result in a pressure drop above 343.2 Pa during inhalation and 245.1 Pa during exhalation when tested using an airflow rate of 85 l min^{-1} (corresponding to moderate work rate), however, the N95 mask has been reported to cause

Figure 1. (a) Model of the face masks based a combination of materials. The plots show the filtration efficiencies of an N95 mask in comparison with different clothing materials. Reprinted with permission from [17]. (b) Injection Molded Autoclavable, Scalable, Conformable (iMASC) system for aerosol-based protection. Reproduced with permission from [18]. Copyright © 2020, BMJ Publishing Group Ltd. All rights reserved. (c) Early prototype of the mask system including a silicone 3D printed face-piece, polyjet 3D printed rigid components, and a non-woven polymer filter. Reproduced with permission from [19]. © OSI/Andy Ryan.

difficulty in breathing [22]. The comparison of the different commercially available facemasks is shown in table 1.

Therefore, the transmissibility of the virus via droplets, its ultra-small size in addition to the limitation of commercially available PPE confirm the necessity of developing efficient face masks to fight against this pandemic. As a result of this fact, much of the nanotechnology community has focused their efforts on developing efficient and smart face masks. More specifically, many researchers have focused on developing face masks with filtration, exclusion/repelling and sensing capabilities using nanomaterials and nanostructures. In this review, the filtrations techniques using nano-fibers, nanoporous membranes and triboelectric nanogenerators are discussed, the mask coating techniques which achieve hydrophobicity, anti-viral and recharging capabilities using nanomaterials are reviewed, in addition to highlighting the nano-electronic biosensors which can be potentially integrated on the masks to detect the virus. All these sections/fabrication steps are shown in figure 2 which we discussed, previously.

Finally, the usability of the nanotechnology enabled face masks is compared with that of currently available commercial grade masks.

2. Filtration mechanisms and materials

The penetration of particles through a specific filter greatly depends on their size and requiremnt of application. The

application of thefiltration mechanism is required and based on the needs of customers in different industries such as food, chemical and healthcare industries [23–25]. In general, air filters are divided into two main categories: depth filters and membranes. The depth filtration mechanism utilises the thickness of single or multiple layers to trap the unwanted particles. Whereas, the membrane filters trap the contaminates which are larger than the membrane pore size. Five different mechanisms can occur in depth filtration: gravity sedimentation, inertial impaction, interception, diffusion and electrostatic attraction [24]. The gravitational forces and inertial impaction are dominant in large-sized droplets ($>1\ \mu\text{m}$), where the inertia of particles is too large such that a change in the direction of the particles is induced. As a result, large particles can stray from the airflow, collide with the filter fibers and stick to them instead of penetrating through the filter [26]. The interception is effective in trapping particles with sizes up to $0.6\ \mu\text{m}$ and occurs when the particle is following the main airflow and interacts with the closest fibers which are within one particle width distance. The interception mechanism is not directly determined by the velocity of the particle, however, it becomes more noticeable for smaller particles. One of the main differences between interception and inertial impaction is that in interception, the particle does not stray from the streamline. For particles which are sub- $0.2\ \mu\text{m}$, the random Brownian motion of particles increases the collision probability between the fiber and particles. As the size of the particles is reduced, the diffusion rate becomes

Table 1. Comparison table of commercially available face masks.

Type of face masks	Materials	Filtering efficiency (%)	Advantages	Disadvantages
Medical masks	Non-woven fabric	32.9	Disposable and can be used for hospital outpatient	Poor efficiency
	Blended material in non-woven fabric	96.5	Disposable	Greater resistance to respiration,
N95	Thickened non-woven fabric	94.9	Disposable and offers protection against influenza and microbial particles both	Greater resistance to respiration, not comfortable, not resistant to oil-based particles
R95	Activated carbon	99.9	Resistant to oil-based particles, service time = ~8 hours	Greater resistance to respiration,, expensive and not very comfortable
ITO PM2.5	Thickened non-woven fabric	82.8	Useful for filtering particles size of less than 2.5 μm	Greater resistance to respiration

Figure 2. Outline of the review paper on nanotechnology enabled face mask.

more apparent [27, 28]. Moreover, as the speed of the particles is reduced, their residence period in the filter medium is increased resulting in an amplified probability of collision with the fibers. Nevertheless, nano-sized particles can still slide and penetrate between the fibers, in this case, electrostatic attraction becomes the most efficient trapping mechanism where the virus for instance is attracted to the fiber surface. It should be noted however that the electrostatic attraction can also be used to filter out larger particles [29]. In this technique, electrically charged fibers are included in the filter to attract oppositely charged particles. Higher speeds of particles are also found to reduce the efficiency of the electrostatic attraction based filtration [30–35]. On the other hand, in membranes-based air filters which generally use polymeric materials, the filtering is generally based on the straining mechanism and is size-dependent. In this case, particles of smaller size than the apertures in the membrane get filtered out. It should be noted that in this kind of filters, the fouling or cake-formation on the surface of the membrane becomes a critical issue which needs to be solved, where the filtered particles agglomerate and block the filtering process. Thus, an anti-fouling mechanism is necessary to keep the surface of the membrane clean.

2.1. Depth filters

The most common application of depth filters in the polishing industries are water removal, transformer oil filtration, blood fractionation operation for recovering plasma, haze removal from spirits etc. The biopharmaceuticals industries also utilize the depth filters for cell separations [23]; however, the depth filters are fabricated using three components i.e. wet-led cellulose, filter aid and resin binder. The wet-led cellulose is polypropylene fibres that provide rigidity to the depth filters.

The filter aid component provides an increased surface area which improves the holding capacity of solids and enhances the retention characteristic. The resin binders consists of the positive charges which can commonly be functionalised using charged ligands like amines.

A face mask generally includes a filter medium as an inseparable part of the PPE tool. The level of protection provided by a given mask normally depends on the embedded filter efficiency as well as the face seal property. When it comes to aerosols filtration, using finer fibers in the filter allows for finer particles filtration. Typically, the size of the filter fiber is matched with the size of the particles to be filtered [36]. Thus, for nanoparticles filtration, nanofibers-based filters are needed. Nanofibers do not exist individually and are typically produced on a substrate of nonwoven fabric [37]. In fact, non-woven fabric has been reliably used to generate different-sized synthetic fibers for air filtration purpose. For instance, the melt-blown process has been very commonly used to produce microfibers having a diameter in the range of a few micrometers [38]. Nanofibers, on the other hand, have been most commonly generated using an electrospinning process where the used process parameters dictate the dimensions of the fibers. In fact, different techniques exist for the development of sub-microfibers including phase separation, melt-blown method, template synthesis and plasma treatment [39–42]. However, these techniques are generally time and energy consuming, difficult to control with the need for additional species to be able to separate fine particles. On the other hand, electrospinning became very popular due to its ability to generate nanofibers with controllable dimensions, high porosity, large specific surface area, interconnected porous structure with a low packing density [43–47].

The increased surface area-to-volume ratio available in the nanofibers for capturing particles results in an enhanced filtration efficiency. In fact, nanofibrous media exhibit a high permeability, low basis weight with a small pore size, high specific area (from 1 to $100 \text{ m}^2 \text{ g}^{-1}$), good interconnectivity of pores and possibility of functionalizing them using nanomaterials [48]. As a result, a face mask would require a smaller amount of nanofibers (5 g m^{-2}) compared with microfibers based mask where 30–50 gm of the material is included [49]. Nevertheless, there exist several challenges to overcome when using nanofibers. First, the pressure drop across the filter increases as the diameter of the fiber reduces, following the Davis equation, and as the size of the pores is reduced [50]. A higher pressure drop is undesired since it translates into a lower breathability of the mask where the person will need to put higher effort and consume higher energy to drive the airflow across the filter. Different techniques have been proposed by researchers to achieve a balance between the enhanced filtration efficiency and pressure drop in nanofibrous filters including the use of fiber blend and multi-layers of less dense filters [51]. For instance, Loesecke *et al* reported that adding a nanofibrous coating onto a typical cellulose filter allows the filtering of particles with sizes down to 100 nm, enhancing the filtration efficiency with insignificant degradation in the permeability or air resistance as a result of the considerably smaller size of the fibers in comparison with the substrate itself [52]. In this work, the authors developed a nylon 6-polyacrylonitrile nanofibre-nets binary (N6/PAN NNB) structured membrane with an optimized

Figure 3. (a) FE-SEM images of N6–PAN NNB membranes showing the how filter consists of 2D nano-nets running through the N6 and PAN nanofibers. Reproduced from [52] with permission of The Royal Society of Chemistry. (b) A highly breathable and degradable filter based on cellulose nanofibers capable of filtering sub-100 nm particles including viruses. Reproduced with permission from [53]. Copyright © 2020, Queensland University of Technology. (c) SEM image of the PVDF filter prepared using electrospinning. (d) Efficiency of 2L PVDF nanofiber filter versus aerosol size before and after charging it. Reprinted from [58], Copyright © 2020 Elsevier B.V. All rights reserved.

pore size which dictates the filtration efficiency and packing density which dictates the breathability. The nanofibers were developed using an electrospinning or netting process on a non-woven substrate using optimized process parameters including a 15 cm distance between the jets and the substrate, a 30 kV voltage applied to the tip of the needle, at a temperature of 23 °C with a relative humidity of 25%. The membranes were then dried at 80 °C for 4 h in a vacuum oven in order to get rid of the residual solvent. The obtained filter consists of 2D nano-nets running through the N6 and PAN nanofibers as shown in figure 3(a). The authors extensively analyzed the performance of the resulting filter in terms pore size, packing density, mechanical properties and filtration performance. The results show that the membrane with a low basis weight of 2.94 g m⁻² is capable of achieving a filtration efficiency of 99.99% is possible when 300 nm particles and 90 l min⁻¹ flow rate are used with a high quality factor of 0.1163 Pa⁻¹, which is several times higher than that of commercially available glass fiber and melt-blown polypropylene fiber-based media. Moreover, researchers, from Queensland University of Technology, Australia, reported development of a highly breathable filter and which can filter sub-100 nm particles including viruses [53]. The filter was fabricated using a disposable and biodegradable filter cartridge based on cellulose nanofibers as shown in figure 3(b). Initial testing showed that the resulting filter is much more breathable than commercially available face masks and respirators. Charging the nanofibers is another technique

which assists in improving the filtration efficiency, especially when the particles to be filtered are charged. In fact, traditional respirators such as N95, consist of 4 to 5 layers including non-woven fibers of polypropylene (PPY) [54]. The PPY fibers prepared by electrospinning are micro-porous and may store electrostatic charges during the development process [55]. Thus, in addition to the size-dependent filtration such as inertial impaction, the electrostatic attraction contributes towards the filtration process in such masks, resulting in an efficiency of 95% against 300 nm particles [56]. Nevertheless, it has been reported that the stored charges on the PPY fabrics gets dissipated after 8 h of exposure to the ambient conditions. As a result, the efficiency of the masks considerably degrades after such a short duration. Even though N95 masks are developed for single use, however, in the current situation where the demand has increased and the subsequent scarcity of masks, efforts are being done to allow their reusability [55, 56]. A team of researchers have reported the capability to decontaminate and reuse the N95 masks based on actual data and following procedures provided by the Center for Disease Control (CDC), USA. There after, different approaches to make the N95 masks reusable are being explored. Hossain *et al* have studied possibility to recharge the N95 masks after decontamination which enhanced the filtration efficiency of masks [57]. Before recharging the N95 masks, sanitisation of the masks is performed. As a result, the masks lose most of their charges. Following this step, the recharging of masks is performed. The experimentation

proved that the filtration efficiency after recharging the masks is also dependent on the used sanitisation method. The filtration efficiency (in %) of KN95 masks increased from 90 to 96, 74 to 86 and 75 to 95, after sanitizing using ethanol, boiling water and laundry using a regular washing machine (for 84 min and at 40°C cycle), respectively, and recharging at 1000 V [57].

Leung *et al* investigated the effect of charging the nanofibers in their developed air filters which can be used in future respirator and facemask devices with 90% filtration efficiency on 100 nm airborne COVID-19 virus with a pressure drop of lower than 30 Pa for a good breathability [58, 59]. To achieve this goal, the authors developed polyvinylidene fluoride (PVDF) nanofibers using an electrospinning process and were next charged using a corona charging process where a 15 kV was applied for 1 min using a homemade 5-wire charge head placed 30 mm away from the fibers as depicted in the SEM image in figure 3(c). The obtained filters showed a basis weight of 0.765 g.m^{-2} with an average diameter of 525 nm. The filters were then arranged in 2, 4 and 6 layered modules in an attempt to enhance the filtration efficiency without degrading the breathability characteristic and minimizing the electrical interference between adjacent nanofibers. The authors reported that by charging the nanofibers, the filtration efficiency against 80 nm aerosols can be improved by up to 180% compared to non-charged fibers where the filtration mechanism is mostly mechanical (a combination of diffusion and interception) as shown in figure 3(d). By stacking 6 layers of charged PVDF layers, efficiencies of 88%, 88% and 96% were achieved for 55, 100 and 300 nm ambient aerosols, while efficiencies of 92%, 94% and 98% were reported for monodispersed NaCl aerosols of 50, 100 and 300 nm in sizes. The discrepancy between the reported results for ambient aerosols and NaCl is due to absence of interaction between the NaCl non-charged (neutral) aerosols. As a result, the 6 layered PVDF was qualified for a N98 respirator where it can filter out 98% of the 300 nm particles, however, at a pressure drop of 26 Pa which makes it 10 times more breathable than conventional N98 respirators. In a more recent study, the same group studied the effect of different nanofibers diameters on the filtration efficiency of the multiple-layered charged PVDF filters and dictated the minimum number of layers needed to be stacked to achieve a good protection from the COVID-19 disease [58].

2.2. Functionalised depth filters

Functionalizing the nanofibers has been performed by several researchers in an attempt to enhance their filtration capacity in addition to improving their thermal, mechanical and chemical resistance properties. The surface modification is generally achieved during the development of the fibers or most commonly, the electrospinning process. Previous studies have already analyzed the introduction of active nanoscale materials, including Al_2O_3 , SiO_2 , CuO , and Carbon dots among others, in fibers [60–68]. In addition, instead of performing corona charging of nanofibers, nanomaterials, known as electrets and which are capable of retaining electric charges over extended periods of time and to generate an external quasistatic electric field, can be

added into the nanofibers. To this end, Li *et al* investigated the effect of embedding nanostructured SiO_2 electrets on nanofibers due to their excellent dipole orientation and the charge storage capabilities [69]. Polyetherimide (PEI) was selected as the precursor polymer of the nanofibers due to its high temperature resistance, chemical and hydrolytic stability [70]. The nanofibers were developed using an electrospinning process and where hydrophobic SiO_2 nanoparticle electrets (7–40 nm) were added during the same process, in a single step. The results show that the PEI/ SiO_2 membranes were able to achieve a 99.992% filtration efficiency against particles with 300 nm size and a pressure drop of 61 Pa, thanks to the contribution of the space charges and polarization. The addition of the nanoparticles not only enhanced the filtration efficiency but also provided the nanofibers with a super hydrophobicity with a water contact angle of 152° , achieving nanofibers with a self-cleaning capability in comparison with commercially available polypropylene (PP) filter medium. Moreover, the fibers could be heated up to 200°C for 30 min. Without deteriorating the excellent efficiency, which enables the sterilization and reusability of the filters. The effect of embedding different nanoparticles was also reported by the same authors, including barium titanate nanoparticles (BaTiO_3 , 20–60 nm), boehmite nanoparticles (10–40 nm) and silicon nitride NPs (Si_3N_4 , 10–30 nm) as shown in figures 4(a)–(d). In fact, the electric charge property, morphology, porous structure and efficiency of filtration (figure 4(e)) can all be tuned by controlling the material and concentration of the electrets. When the nanoparticles solutions were added, the nanofibers morphology was changed where nanoscale rough islands were generated on the fibers surfaces which can considerably enhance the effective contact area between the fibers and the particles to be filtered. Moreover, the fiber diameters of the PEI is reported to be reduced when the different nanoparticles solutions were added during the electrospinning process compared to the case of pure PEI fibers. This is explained by the fact that the viscosity of the solution in addition to its conductivity got reduced when the nanoparticles solution was added.

In terms of filtration performance, the PEI- SiO_2 membranes exhibited the highest filtration efficiency compared to three other electreted membranes with a similar basis weight of 1.41 g m^{-2} . This confirms that the SiO_2 particles are capable of trapping a higher density of charges on the surface and in the bulk of the fibers as a result of the permanent dipole orientation characteristic of the particles. This was further confirmed using a study on the charge retention property of the electreted fibers based on an aging procedure where the current peak in the fibers was measured as a function of temperature. In addition, the degradation of the surface potential of the as spun fibers was tested in an attempt to investigate the charge transport in the relevant fibers. While the pure PEI fibers showed a 53% charge degradation over a period of 120 min, PEI- SiO_2 fibers showed a much smaller charge degradation of 10% over a period of 200 min, which confirmed the benefit of electreted fibers. The reported phenomenon was a result of the trapping of charges by permanent dipole orientation, which makes the escaping from the traps more challenging for the carriers. Finally, it should be

Figure 4. FE-SEM images of Polyetherimide (PEI) based nanofibers functionalized with different nanoparticles including: (a) PEI-Boehmite, (b) PEI-SiO₂, (c) PEI-Si₃N₄ and (d) PEI-BaTiO₃ membranes. (e) The resulting filtration efficiency and pressure drop across the filters with the different nanoparticles. Reprinted from [69], Copyright © 2014 Elsevier Inc. All rights reserved. (f) Embodiment of proposed triboelectric self-powered mask. (g) Block diagram of TENG based self-powered mask, where VTU stands for Voltage Tripler circuit unit. Reprinted from [71], © 2020 Elsevier Ltd. All rights reserved.

noted that the filtration efficiency of the electrospun membranes did not deteriorate even when an airflow rate of 90 l min^{-1} was used, which is needed for actual application in face masks.

2.2.1. Nanoparticles based. The study is performed to understand the effect of integrating nanoparticles with nanofibers for a different purpose; instead of being used as electrets, Ag (5 nm), TiO₂ (21 nm) and ZnO (50 nm) nanoparticles were used for their antimicrobial activity [72]. Polyacrylonitrile (PAN) was chosen for the generation of nanofibers due to its chemical stability and mechanical strength. PAN nanofibers were again developed using an electrospinning process and loaded with the nanoparticles in a single step on a polyethylene (PET) substrate with 27 μm fibers. The obtained nanofibers showed an average diameter of 290 nm with an average basis weight of 75 g m^{-2} . The filtration efficiency was tested against NaCl aerosols with particles size ranging from 9 up to 300 nm under an airflow rate of 1.5 l min^{-1} . The TiO₂ fibers showed the best performance with a 100% filtration efficiency while ZnO based fibers showed the lowest efficiency of 95% with a lower pressure drop which could be due to the larger pore size and lower thickness. Nevertheless, it should be noted that the antibacterial property is used in this work, while for fighting against the COVID-19 pandemic, an antiviral property would

be needed instead. In addition, the filter was tested under a very low airflow rate of 1.5 l min^{-1} while for a face mask filter to be qualified, it needs to be able to exhibit excellent filtering at a flow rate of 80 l min^{-1} . It should also be noted that the concentration of the nanoparticles affects the filtration performance. To this end, Zhang *et al* reported the effect of adding different concentrations of boehmite nanoparticles to 90 nm PAN electrospun nanofibers. The addition of 5% weight of nanoparticles is shown to prolong the electrostatic duration time in the nanofibers where the removal efficiency decay was less than 0.03% after 48 h, compared to the pristine PAN nanofibers which showed a 1.02% removal efficiency decay. Thus, the introduction of the appropriate concentration of the boehmite nanoparticles resulted in an extended service lifetime in addition to an enhanced capture efficiency [73].

In order to add the antiviral activity to nanofibers, different nanomaterials have been studied [74, 75]. In specific, copper oxide (CuO) nanoparticles have a small bandgap and exhibit an ability to prevent the growth of viruses and bacteria, as a result, they have been used in biomedical applications including infection control [76]. More specifically, it has been reported that the virus responsible for COVID-19 exhibits a lower stability on copper surface in comparison with different materials such as stainless steel or plastic [77–79]. As a result, using copper oxide in the filtration material helps in preventing antifouling as the microbes or viruses would not be stable on and would not get

attached to the surface of the filter [80]. Very recently, Kumar *et al* reported the capability of copper-zinc-imidazolate metal organic framework (MOF) (ZIF-8) core-shell nanowires in improving the antimicrobial performance of face masks by dip coating the filtration media with the copper based solution. The authors reported an enhanced antimicrobial performance of the copper@ZIF-8 core-shell nanowires based face mask against *Streptococcus mutans* and *Escherichia coli* compared to other face masks which were coated with only copper or ZIF-8. Moreover, the authors assessed the antiviral activity of the face masks against SARS-CoV-2 using virus-infected Vero E6 cells, showing a 55% prevention of virus replication after 48 h using 1 μ g of the core-shell nanowires per well. Finally, the authors tested the cytotoxicity of the nanomaterials against four cell lines including the A549 lung epithelial cells, and their effect on the inflammatory response of the cells, proving good biocompatibility. Nevertheless, while the authors provided an in-depth analysis of the core-shell nanowires antibacterial and antiviral performance in addition to their biocompatibility, however, an actual testing of the filtration efficiency against the SARS-CoV-2 in addition to how the coating affected the breathability of the filtration medium were still missing [81].

2.2.2. Nanomaterials based. Graphene is a monolayer of 2D carbon atoms with a honeycomb lattice and which shows excellent electrical and mechanical characteristics such as high electrical conductivity and high robustness. The functional derivative of graphene i.e. graphene oxide is another material which has been studied. Graphene oxide shows an antibacterial, antiviral and biocompatibility characteristics [82–84]. As a matter of fact, using graphene oxide as a minor component in a composite material, an antiviral property was demonstrated against coronavirus and porcine diarrheavirus [85]. Thus, owing to these promising properties of CuO and graphene oxide, Ahmed *et al* developed a membrane based on a polymeric material: Polylactic acid (PLA) and cellulose acetate using electrospinning [86]. Cellulose acetate was chosen due to its biodegradability, transparency, high elastic modulus, in addition to the capability to generate it from starch and different carbohydrates, in addition to its ability to filter out the SARS-CoV-2 virus. In fact, PLA based membranes have been previously used in many promising air filters [87–89]. As a result, cellulose acetate has been widely used in air filters, in addition to other applications such as tissue engineering, protective clothing and reinforced nanocomposites [90]. The authors developed a prototype of a PLA based face mask with a disposable filter based on electrospun PLA with cellulose acetate which are doped with CuO nanoparticles and graphene oxide. CuO nanoparticles and graphene oxide were embedded within the filter in order to allow it to inhibit the stable attachment of the virus on the surface of mask and and to inactivate it. Finally, the authors reported a financial study and indicated that the cost of the mask would not exceed \$1.88, which could potentially be a competitive manufacturing price compared to the current selling price of the commercially available NIOSH certified N95 respirators. While the design of the mask, the used materials combination in addition to the competitive cost sound very

promising, it should be noted that actual testing has not been performed in the study, and thus the performance of the mask is yet to be assessed and confirmed. Readers interested in additional works on antimicrobial face masks using various materials including polymers and natural materials are referred to the review paper by Pullangott *et al* [91].

The functionalization of face masks has also been reported using an embedded layer of triboelectric nanogenerators enabling an electrocution mechanism for filtering and deactivating the coronavirus. In fact, nanosized particles can easily slide in between the pores in a fibrous network [92], as a result, electrostatic attraction can greatly enhance filtration by attracting the particles or virus to the fibers. Integrating the electrostatic attraction concept with nanogenerators which can provide triboelectric charges from mechanical motions can be a very promising solution for face mask application. More specifically, the triboelectric layers could harvest mechanical energy from a wide range of activities including breathing, talking and different facial movements. It should be noted that triboelectric nanogenerators have been previously studied and their effectiveness in terms of simple designs, noise free output signal, compactness, and cost-effectiveness, have been confirmed in many applications including wearables [93–97]. To this end, Ghatak *et al* investigated a self-powered face mask consisting of 3 triboelectric layers (one tribo-positive and two tribo-negative layers) with an outer metallic mesh layer that produces the electrocution mechanism to deactivate the virus using mathematical modelling [71]. The triboelectric layers provide additional protection to the person wearing the mask in addition to generating the electric potential for electrocution. The electrocution layer generates a large enough electric field due to the stored charges in the capacitor and which is charged by the triboelectric nanogenerator. As a result of the meshing in the electrocution layers, current passes between the metallic contacts whenever the droplet carrying the virus gets in between them. The droplet is considered by the authors as a low resistive path having a maximum resistance of 1 k Ω . A capacitor and a resistor are added in the circuit in order to store charges and to reduce the time constant of the discharge circuit, respectively (figures 4(f), (g)). This allows for using the stored charges over a longer period. Thus, once the current flows in the circuit, the droplet gets evaporated if it is made of water, however, even if the droplet is based on a different liquid, the authors expect that the virus would get deactivated. In order to further enhance the energy density between the electrocution layers and thus increase the triboelectric charges, it is necessary to optimize the used materials for the triboelectric layer, the surface charge engineering and the surface modification [71, 97, 98]. To achieve this purpose, the authors studied various combinations of four different fabric materials for the triboelectric layers including polyimide, polypropylene (PP)-polyurethane (PU), polyvinylchloride (PVC), and latex rubber-PU, the results show that the latex rubber-PU is able to provide the largest effective charge. The needed distance between the triboelectric layers is found to be 0.1 mm at an applied voltage of 1 V in order to generate a high enough electric field

of 104 V m^{-1} across the droplet. The authors have also confirmed theoretically that the generated heat by the triboelectric layers would not be harmful to the wearer as the generated heat is negligible. While the authors have extensively studied the face mask in terms of optimization of materials and dimensions, power management, noise, and safety issues, and showed its potential in fighting against the pandemic, however, all the results are obtained using mathematical modelling while the development of the mask and its actual testing still need to be conducted to further confirm the advantages and the viability of this solution especially against nanoparticles having the dimensions of the virus. Moreover, the cost of the face mask is expected to be high due to the integration of electronic components with the polymeric based triboelectric layers and the meshed metallic electrocution layer.

To overcome this shortcoming, low cost (\$1/mask) solution based on triboelectric nanogenerators has been investigated by Figerez *et al* where the triboelectric layer is used to recharge the face mask and enhance the electric attraction based filtration instead of electrocution based filtration [99]. In fact, developing masks using reusable and rechargeable materials has attracted a growing attention and a couple of chargeable face masks have already been demonstrated by different researchers in the recent past [57, 100]. However, enabling the charging of the mask using a simple mechanical movement after the decontamination process would be an ideal solution for future low cost face masks. To this end, Figerez *et al* developed a mask in which the filter layer included in an N95 mask (i.e. polypropylene) has been modified using a mixture of graphene oxide and hydrophobic PVDF and embedded in between a nylon and a cotton layer [99]. The resulting three-layered face mask is reported to be able to achieve triboelectric recharging using facial movements resulting in a >95% filtration efficiency for particles which are sub-500 nm with a flow rate of $30\text{--}35 \text{ l min}^{-1}$. The modified layer in the mask is found to generate $\sim 2 \text{ nC cm}^{-2}$ with a corresponding voltage of 20 V and with a retention of almost 5 days under ambient conditions. Simple motions and agitations such as bending the hand are found to recharge the mask even at high relative humidity levels (>80%). Finally, the modification of the mask inner layer with PVDF/graphene oxide is found to enhance the hydrophylic nature of the PPY fabric which contributes to a reduced probability of passing the virus from the outer environment. The authors report that the presented PVDF/Graphene-oxide based face mask has better filtering efficiency (>95%) and high Q-factor (about 20 kPa^{-1}) which increases the usability of facemask for single use and even for multiple use after decontamination. The presented solution is low cost (about 1 USD/mask), simple and promising, however, testing of the filtration efficiency for particles in the range of the COVID-19 disease still needs to be performed to further prove the potential of this mask in fighting against the pandemic, in addition to testing the permeability of the mask at higher flow rate (at least 80 l min^{-1} instead of 30 l min^{-1}) to confirm its breathability and viability in face masks application.

2.3. Straining-based membranes

While most of the previous demonstrations have been based on depth filters using different materials for fibers combined with various functionalizing nanomaterials to combat against the COVID-19 pandemic, the nano-porous membranes using the straining based filtration mechanism have also been investigated for application in anti-COVID-19 face masks. In the straining based filtration, the dimension of the pores in a membrane are made smaller than the dimensions of the particles to be filtered, such that only smaller particles can penetrate. Nevertheless, in such filtration mechanism, anti-fouling is important since the filtered particles can agglomerate on the surface of the membrane and prevent the continuation of the filtration process and inducing larger pressure drops.

The reuse of the same N95 mask with disposable nano-porous membranes allows fighting against the shortage of N95 masks worldwide, in addition to enhancing the mask filtration efficiency against the COVID-19 disease. El-Atab *et al* developed a nano-porous polyimide based membrane using a combination of electron beam lithography (EBL) and wet etching for application in reusable N95 face masks [101]. In this article, the authors theoretically studied the breathability of the membrane under realistic conditions similar to those of an N95 mask, and the membrane is reported to be breathable over a wide range of nano-pores dimensions and densities and which allow, at the same time, the filtration of the SARS-CoV-2 virus. Finally, the authors indicated several methods which can be used for attaching the membrane on an N95 mask such as wrapping the membrane over the mask and attaching it using tape from the inner side of the mask in order to avoid the contamination of the hand or mask when removing and replacing the disposable membrane. Nevertheless, actual testing still needs to be conducted to confirm the reported breathability of the membrane, in addition, it should be noted that the cost of the membrane is expected to be high due to the usage of nanofabrication tools such as the electron-beam lithography.

Different viruses such as rabies, HIV-1, influenza and dengue, which have sizes less than 10 nm, can be filtered using the nanofiltration membrane [102–104]. Studies on the different straining based nanofiltration of SARS-COV-2 virus are not extensively explored, however, few studies have proven the removal rate of other viruses on the log scale from 3 to 8 [105–109]. All these studies are well performed and show notable improvement, however, these need to be experimentally verified for the SARS-COV-2 virus.

The ultrafiltration technique has also been explored to filter different viruses since more than a decade [110]. The combination of ultrafiltration and coagulation on pilot scale has been utilized for filtrating wastewater [111], by Lee *et al* After optimizing the pH of secondary effluent from 5 to 6; the improvement is performed to remove/filter MS₂ bacteriophage. In another work from Lu *et al*, [112] grafted zwitterionic polymer hydrogels are used to perform modification of polyether-sulfone membrane. This study analyzed the efficacy of the membranes in filtering MS₂ and HAdV-2 human

viruses. Additional experiments are needed to confirm the capability of the same membranes in filtering the SARS-CoV-2 virus.

3. Coatings

An alternative to using nanomaterials as integral components in the filter of face masks, coatings based on nanomaterials can be added on the surface of a regular mask to enhance its filtration efficiency. In fact, surface treatments which increase the resistance against illness-inducing microbes and viruses, such as antimicrobial and antiviral coatings, can drastically enhance the PPE efficiencies and functionalities. Over the last couple of decades, substantial amount of research has been conducted on the addition of antimicrobial coatings on the surfaces of PPE and medical devices. Taken into consideration the fact that microbes can generate drug resistance, known as antimicrobial resistance, developing antimicrobial coatings using nanomaterials is of particular interest. Ravindra *et al* developed antibacterial cotton fibers with silver nanoparticle based coatings [113]. They achieved this by immersing 1 mm cotton fibers in a solution of silver nitrate with leaf extracts. The polysaccharides of the leaf extracts converted the silver nitrate into silver nanoparticles attached on the fibers. To test the antibacterial activity in the coating, the authors used the *Escherichia coli* (*E. coli*) bacteria and reported an excellent antibacterial characteristic even when the cotton was washed several times. Similar nano-silver based coatings can provide anti-viral characteristics as well. Otto *et al* have recently reported a novel antiviral nano-coating based on polysaccharides with the purpose of deactivating the coronavirus [114]. This study is not within the scope of this review paper, however, interested readers can refer to the paper for more details. Another complementary technique to achieving an anti-viral surface includes converting the surfaces into hydrophobic ones which can repel the viruses, especially when considering the fact that the COVID-19 virus is generally transported using a water droplet carrier. Thus, a water repellant coating can prevent the penetration of the virus. Coating have also been used to make the surface of the face mask charged and thus be able to repel the charged viruses. Combining the properties of these coatings with the mechanical filtration mechanism would definitely improve the filtration efficiency and hinder the penetration of viruses through the mask, thus contributing to containing the spread of the virus. In the following, the reported nanotechnology enabled techniques for achieving antiviral, hydrophobic and recycling capabilities in an attempt to enhance the filtration efficiency of face masks against the COVID-19 disease are discussed.

To achieve an antiviral feature for application in surfaces and PPE disinfection, chemical coatings based on chlorines, alcohols and peroxides have been previously shown and used to fight against several pathogens [115]. While they are effective in sterilizing surfaces, the chemical coatings have several shortcomings including the need for high concentrations for an effective disinfection, having low durability and

being potentially hazardous to the wearer health and the environment [115, 116]. This led to the use of metallic nanoparticles including copper, silver and titanium dioxide as alternatives due to their intrinsic antiviral properties and effectiveness at lower concentrations [117, 118]. For instance, Balagna *et al* reported the antiviral effects of sub-200 nm silver nanocluster/silica composite coatings against the SARS-CoV-2 virus and which were sputter deposited on an FFP3 mask with non-woven fabric as shown in figures 5(a)–(d) [119]. The preliminary results are promising, while further testing is needed to study the effect of the coating on the breathability of the mask, durability and safety. Furthermore, it should be noted that sputtering is unlikely to be feasible to achieve such composite coatings on a mask surface. More likely, solution-mediated approaches will need to be developed to achieve such coatings. Moreover, NanoTechSurface, Italy, reported the development of a long-lasting and sterilizing coating based on titanium dioxide and cell ions [120]. Similarly, FN Nano Inc., USA prepared a coating based on titanium dioxide nanoparticles with photocatalytic activity which allows the decomposition of viruses and the destruction of their membranes when exposed to light [120].

In addition to the anti-viral property, super-hydrophobicity can contribute to protecting the wearer from the disease by repelling the virus. Super-hydrophobic surfaces have lately attracted significant interest due to their excellent water repellant properties with water contact angles beyond 150°. As a result, they enable surfaces with self-cleaning features [121, 122]. Due to these preferable properties, super-hydrophobic surfaces have lately garnered popularity in biomedical applications which allow blood repellency and antifouling properties [123, 124]. For a surface to become super-hydrophobic, it has to be chemically modified in addition to being textured [125]. More specifically, surfaces coated with materials exhibiting a low surface energy in addition to showing nano or microstructures allow the super-hydrophobicity. Even though several methods have been previously demonstrated for achieving hydrophobicity [124, 126], however, they have not been widely adopted in commercial applications due to their complexity and high cost. In order to overcome these shortcomings, a promising method is the development of nanocomposites based coatings consisting of low surface energy nanoparticles embedded within a polymeric solution. Thus, these coatings can then be applied on a surface to convert it into a super-hydrophobic one [127–129]. The selection of the coating constituents relies on the desired surface properties. For instance, flexible, stretchable and transparent polymeric matrices with low surface energy including polydimethyl-siloxane and Ecoflex can be employed on flexible surfaces, while polymeric materials with a larger mechanical stiffness, such as epoxy, can be employed to cover regularly touched rigid surfaces. For application on face masks and respirators which are generally flexible, the coating should also not degrade the breathability of the PPE. When it comes to the nanoparticles component of the coating, silicon dioxide has shown outstanding self-cleaning properties while being cost effective [127–129]. On the other hand copper nanoparticles can be of high importance in the fight against the SARS-CoV-2 virus. As a matter of fact, copper fomites were shown to have

Figure 5. Disposable facial FFP3 mask in non-woven fabric: (a) uncoated (b) coated by means of co-sputtered antimicrobial/virucidal silver nanocluster/silica composite coating. Typical morphology (FESEM images) of the coated mask (Ag₃W) at (c) lower and (d) higher magnification. Bright spots are silver nanoclusters embedded in the silica matrix. Coated textile fibers are visible, with the EDS analysis in at% of Si and Ag relative to the two coatings (Ag₃W and Ag₅W). Reprinted from [119], © 2020 The Authors. Published by Elsevier Ltd on behalf of European Ceramic Society.

antiviral features against Influenza A virus particles [130] in addition to being the quickest in destroying the activity of the SARS-CoV-2 particles in comparison with various materials [79]. Copper-based nanoparticles were also demonstrated having an inactivation role on the H1N1 influenza virus [131], in addition to other virus strands [132].

To this end, Meguid *et al* proposed a coating based on a mixture of silica and copper nanoparticles to achieve self-cleaning and anti-viral properties in order to fight against the pandemic [133]. The nanocomposite was fabricated by initially dispersing the nanoparticles in a silicone based polymeric matrix. To achieve a homogeneous dispersion of the nanoparticles, an acetone solvent is used in the solution which was mixed using an ultrasonicator. The resulting emulsion produced was then spray-coated onto the surface. Subsequently, the covered surface with the solution is cured at a high temperature of 120 °C for 1 h to guarantee its effective polymerization. The nanoparticles concentration in the coating can significantly modify the properties of the resulting surfaces. The authors reported that a concentration of 15 wt% was needed in order to achieve a balance between the super-hydrophobicity characteristic and the durability of the coated surfaces. The resulting surface resulted in a 163° water contact angle on various substrates including fabrics, which would be needed for commercially available face masks, and allowed an impinging droplet to move away from the surface without leaving any traces as shown in figures 6(a), (b). Using SEM images, the hydrophobicity of the surface was explained where the nanoparticles created nano-asperities while micro-structures are achieved due to the atomization effect in the

coated surfaces as shown in figure 6(a). Thus, the subtle balance between the nanoparticles concentration and polymer is necessary for the development of an effectively hydrophobic surface without degrading its durability. Nevertheless, while the authors proposed the incorporation of copper based nanoparticles in the coating to achieve an antiviral feature, however, this was purely based on the previously confirmed antiviral characteristic of the copper material. Thus, additional experiments are needed to confirm this hypothesis. The effect of the mixture between silica and copper nanoparticles with different concentrations on the resulting properties of the coating would be interesting for the researchers to learn about. Moreover, virus testing are needed to ensure that the coating would be able to inactivate the SARS-CoV-2 virus in specific. Finally, the breathability of the material/mask before and after applying the coating is necessary to be known for it to be a viable solution for application in face masks.

In a complementary work, Ray *et al* demonstrated the potential of silica nanoparticles in achieving hydrophobic coatings for face masks [134]. A compressed compressed-polyurethane (C-PU) face mask was selected since they became very popular during the current pandemic as a result of their flexibility, softness, comfort, low cost in addition to their filtration efficiency. Moreover, the face mask can be reused for 2–3 times after washing it. Nevertheless, the surface of the mask is hydrophilic as most of the commercial face masks, as a result, its efficiency drops when it becomes wet. Ray *et al* developed a low cost and environmentally friendly non-fluorinated coating [135] containing silica nanoparticles to achieve hydrophobicity. The mask was initially dipped and sonicated in silica sol for

Figure 6. (a) SEM image of the spray coated fiber. (b) Typical droplet impingement on a coated metallic sample and rebound at different time intervals leaving no traces. Reprinted by permission from Springer Nature Customer Service Centre GmbH: Springer Nature, *Mechanics of Materials in Design*, [133], Copyright © 2020, Springer Nature B.V.

30 min to achieve a uniform coating. The mask was then partially dried under ambient conditions followed by heating it at 75 °C for 5 min. Next, the mask was sonicated in a hexadecyltrimethoxysilane (HDTMS) solution containing silica nanoparticles for an hour [136]. Finally, the mask was dried at room temperature for 12 h following by curing it at 100 °C for 3 min. As a result, the hydrophobization of the mask is completed. The coating is thus achieved using the dip coating technique which is considered as a low cost, simple and reliable deposition technique with high reproducibility [137]. It is based on the dipping of the sample in the solution to be coated, after removing the sample, a homogeneous liquid is developed on the surface. The water contact angle on the face mask was investigated before and after coating it. With the coating, the contact angle increased from 85° to 132°, in addition, the contact angle was maintained after 15 min while it became 0 on the pristine sample which confirms its hydrophilicity. This also confirms the effectiveness of coating in achieving hydrophobization. The stability of the coating was also proved after dipping the mask in deionized water for half an hour and noting the change in the water contact angle. A 1.2% degradation in the contact angle was reported using 11 nm silica nanoparticles while the uncoated mask exhibited high wettability which deteriorates its filtration efficiency. A reliability test was also performed on the face mask to check its reusability after several washes. For this purpose, the mask was dipped in deionized water for 30 min and then squeezed, the obtained water is then chemically tested in terms of silicon content. An insignificant amount of silicon leakage was observed after the drainage test which confirms its efficacy as a physical barrier even after several washes, moreover, SEM images showed that negligible effect is caused by the coating on

the mask morphology which allows for a good permeability. Finally, the authors studied the effect of the size of the silica nanoparticles on the performance of the mask, where larger size particles (4 nm versus 11 nm) have shown an improved water contact angle, stability and drainage resistance. The size of the particles can in fact be easily controlled by tuning the ammonium hydroxide content in the solution where a higher concentration induces the agglomeration of the particles [138]. It should be noted however, that the breathability of the face mask has not been confirmed experimentally. In fact, the reported porosity of the face mask is reduced from 89% to 75% after applying the coating, while the authors indicate that this reduction will have negligible effect on the air permeability, experimental testing needs to be performed to support this claim. In addition, testing the coating on different face masks such as N95 and nanofiber based filters and investigating the interaction between the solution containing the silica nanoparticles with different fabrics and fiber materials would further confirm the potential of nanocomposite.

The recyclability and hydrophobicity of face masks have also been investigated by Zhong *et al* in an interesting study using a graphene-based coating [100]. The graphene layer was developed using a laser ablation process performed on a polyimide based coating. In fact, laser induced graphene (LIG) is considered as a low cost and scalable method for developing graphene from commercial polymeric substrates. The CO₂ laser pulses were optimized (dual-mode) to avoid damaging the underlying nonwoven mask having a melting temperature of 130 °C and to enable a roll-to-roll production which is compatible with the production of current face masks [139]. The resulting graphene layer showed super-hydrophobicity with a 140° water

Figure 7. (a) SEM image of the nonwoven fibers from a pristine surgical mask; the scale bar is 100 μm . (b) Water contact angle measurement of the pristine surgical mask. (c) SEM of the graphene-coated nonwoven fiber within the surgical mask; scale bar is 10 μm . (d) Water contact angle on the graphene-coated mask. Reprinted with permission from [100]. Copyright © 2020, American Chemical Society.

contact angle which causes the water droplet to roll off of the mask without leaving any marks as shown in figures 7(a)–(d), in addition to photothermal properties, where the surface temperature quickly rises up to 80 °C under solar illumination, allowing for the sterilization of the mask from the SARS-CoV-2 virus which has been previously reported to be very sensitive to temperature [140]. In fact, graphene exhibits a high absorption of photons (95%) between 300 and 2500 nm of the solar spectrum unlike the uncoated mask. As a result, the tested mask under 1 sun illumination using a solar simulator showed a quick increase in temperature to 70 °C in just 40 s and further increased to over 80° after a minute while the pristine mask remained at 45 °C even after 5 min of exposure to sunlight. It should be noted that a temperature of over 70° is sufficient to inactivate most viruses [141, 142]. The study reported an interesting technique for achieving hydrophobicity and sterilization or reusability of the face mask using the graphene coating, however, further experimentations are still needed in order to ensure the safety, comfort and durability of the mask. Whether the high temperature of 80 °C would be uncomfortable to the wearer, the graphene coating would affect the breathability across the mask, the coating flakes would fall off when the person is walking or moving, or whether the graphene flakes/particles would penetrate through the mask and cause medical health issues to the wearer are all questions which need to be answered and experimentally confirmed before the adoption of this technique for large scale production.

Supporting to this work, Shan *et al* demonstrated the development of surgical face masks with graphene modified

melt-blown nonwoven fabric filter to achieve sterilization due to the improved photo-thermal and electro-thermal performance of graphene [143]. To achieve this, interdigitated electrodes based on conductive cloth tape are added on a melt-blown nonwoven fabric filter followed by spreading a graphene layer with excellent electric and thermal properties. Next, the resulting filter is embedded in a surgical mask as shown in figures 8(a), (b), which was then tested in terms of hydrophobicity, filtration efficiency, photo-thermal and electro-thermal characteristics, and reusability. The mask showed excellent hydrophobic characteristics where a 103° water contact angle is achieved while the filtration efficiency against the *E. coli* bacteria is shown to remain almost intact (around 95%) before and after adding the graphene modified filter. However, the modified mask showed enhanced thermal properties where its temperature can rise up to 80 °C when 3 V is applied across the interdigitated electrodes and thus kill the attached viruses. The filtration efficiency of the mask did not degrade even after electrifying 10 tests which supports the reusability claim of the proposed mask. The same technique could be applied on different types of face masks such as the N-95 with enhanced filtration efficiency compared to a surgical mask, nevertheless, additional experiments must be performed to prove safe and useful against the SARS-Cov-2 virus. A similar approach is used by Lin *et al* where graphene nanosheets-embedded carbon were distributed uniformly between the melt-blown fibers in a surgical mask [144]. The resulting face masks showed improved hydrophobic properties with a 157.9° water contact angle. Moreover, the filtration

Figure 8. Graphene modified masks with photo-sterilization capability. (a) Development process flow of a surgical mask with graphene modified melt-blown nonwoven fabric which can be sterilized by applying 3 V across the interdigitated contacts. Reprinted with permission from [143]. Copyright © 2020, American Chemical Society. (b) Surface temperature mapping of a pristine (top) and a graphene nanosheet-embedded carbon-based mask (bottom) showing the enhanced photo-thermal performance of the graphene-based masks under sunlight. Reprinted by permission from Springer Nature Customer Service Centre GmbH: Springer Nature, Nano Research, [144], Copyright © 2020, Tsinghua University Press and Springer-Verlag GmbH Germany, part of Springer Nature.

efficiency of the masks was tested using NaCl aerosols with different sizes, the pristine masks showed a particle filtration efficiency of around 35% against 300 nm particles, while this efficiency is enhanced to 95% when the graphene nanosheets were used. This enhancement can be due to both a mechanical filtration effect in addition to the surface electrons trapping in

the graphene nanosheets. Finally, the graphene modified mask showed a promising photo-sterilization capability where it can heat up to 110 °C under the 1769 W m^{-2} solar illumination for 200 s, compared to 44 °C shown on the pristine mask. The proposed modification of the face mask can prove effective for combatting against the COVID-19 pandemic,

however, the same approach must be applied on face masks with better face sealing compared to the surgical masks, in addition to testing and confirming the safety and breathability of the resulting mask. Complementary to these works, LIGC Applications Ltd, USA, reported a reusable face mask, called G-Volt, based on a microporous LIG foam which can adsorb the virus due to its exceptional physical and chemical characteristics and its high surface area to volume ratio [145]. Once trapped, a small electric current is conducted across the graphene layer using a portable battery to destroy the SARS-CoV-2 virus, resulting in a filtration efficiency of 99% against 300 nm particles. The mask can also be sterilized at home by heating it up at elevated temperatures. MOF have also shown potential in inactivating bacteria as a result of their photocatalytic bactericidal properties. More specifically, Li *et al* demonstrated the ability of zinc-imidazolate MOF (ZIF-8) to almost completely inactivate *Escherichia coli* (*E. coli*) with a high inactivation efficiency (>99.9999%) in saline within 2 h of simulated solar irradiation [135]. The dominant disinfection mechanism is found to be due to the trapped photoelectrons on the Zn²⁺ centers. Using the same filtering medium in air filters, the authors reported a >99.99% photocatalytic inactivation efficiency against airborne bacteria within 30 min. While the filters based on ZIF-8 nanoparticles show photocatalytic antibacterial properties, nevertheless the reported filtration efficiency is tested against large particles in the range of 2.5–10 μm [146].

4. Usability

The studies of the physical parameters, usability and fitting ability are key components of any commercially available facemasks. In this current COVID-19 pandemic situation, N95 facemasks are known to provide the best protection against the SARS-CoV-2 virus from entering into the human body [14, 147]. In the current scenario, multiple N95 facemasks, with different shapes and filtering properties, are readily available in the market; which makes the selection of the best among all a quite challenging task. Nanofiber fabricated facemasks have garnered a lot of attention because of ease in development in addition to their low-cost. More specifically, nanofibers have been extensively utilized for environmental, healthcare and energy storage applications due to their advanced functional and morphological properties [148–153].

According to different strategies and experimental analysis, it has been recommended by Suen *et al*, to use nanofiber based N95 facemasks of flat-fold shape after performing a comparison with three 3M facemasks which are of cup size 1860 (small and regular) and flat-fold shape 1870+. The comparison is performed in terms of the masks' different physical properties including weight, fabric thickness, air permeability (air flow rate), moisture management capacity, and filtering percentage [148]. The wearers of the 3M masks were initially asked to sit for 10 min. Next, all the subjects were asked to perform nursing tasks for 10 min which can involve clinical testing, positioning of patient or any other

physical activity which might be challenging to perform while keeping the facemasks fixed. The subjects were taught to record eight different health parameters; such that breathability, speech intelligibility, difficulty in mask maintaining at specific/same place, facial heat and pressure, comfort level on earlobe. After taking rest for 30 min, all the subjects were asked to wear the nanofiber based facemasks and perform similar tasks and went through the same testing (quantitative fit test and seal check process). Finally, the same parameters were recorded after the completion of the test.

The different physical parameters have been analyzed for all three models of 3M and nanofiber facemasks. Moreover, the collected data is analyzed using multiple statistical techniques which involves chi-squared test, paired t-test and Wilcoxon signed rank test for identifying fit-factors (pass if ≥ 100 otherwise fail), to compare best fitted 3M mask with nanofiber masks during nursing processes and to compare the usability of both kind of masks, respectively. After comparing the physical parameters and data provided by the subjects, the authors report that the flat-fold nanofiber facemask shows better usability in terms of weight, thickness, and ventilation resistance. However, it should be noted that the cost-effectiveness, thermal stress of the subject and durability of facemasks are not considered during the experiments which can play an important role in designing any suitable facemasks during the COVID-19 pandemic.

In a complementary study, Ullah *et al* compared melt-blown and nanofiber facemask filters after considering the shortage of N95 facemasks due to this global COVID-19 pandemic [149]. The melt-blown filter facemasks are found to be not very effective for reuse after cleaning them which can degrade their ability of filtering virus/bacteria, whereas the nanofiber filtered facemask are reusable after cleaning as shown in figures 9(a) and (b). To perform the experimental evaluation of reusability, both kind of facemask filters are cleaned by either spraying or dipping them in 75% ethanol. During the spraying treatment process, 75% ethanol is sprayed thrice and placed in the air for 1 h with a repetition of the process up to 10 cycles. While in the dipping process, both types of masks are dipped in the 75% ethanol solution for 5 min to 24 h and then kept under ambient conditions for drying. Next, the performance of both types of cleaned filters are examined in terms of air-permeability, filtration efficiency, surface and morphological efficiency. The air permeability of melt-blown filter before treatment is found to be about 27.2 cfm units without any significant change after ethanol treatment. In the nanofiber filter, the air permeability was initially almost 17 cfm units and a negligible change has been reported after the treatment. This was explained by the fact that the nanofiber filter is mechanically supported on polyethylene terephthalate (PET) which depolymerizes into mono-ethylene or di-ethylene after ethanol treatment. The melt-blown filters do not show any significant change in pressure drop (for analyzing inhaling and exhaling difficulties) after the spraying or dipping treatment. Nevertheless, the nanofiber face masks showed incremental change due to the same reason, i.e. PET depolymerizes, when treated, however, the resulting values are still within the safety limit. To

Figure 9. (a) Schematics of the cleaning procedure of melt-blown and nanofiber filters after treatment with 75% ethanol. (b) After proper evaluation studies, it has been confirmed that nanofiber-based filters are better choice and are reusable after multiple cleaning treatments. The schematics show that melt-blown filters are not an effective choice to protect the wearer from the virus and bacteria after the cleaning treatment. (c) Infrared thermal images confirming that nanofiber filtered facemasks are better choice for air, moisture and CO₂ transmission. Reproduced with permission from [147]. Copyright © 2009, Oxford University Press.

validate the ability of reusing the filters, the filtration efficiency is one of the major parameters which worsens for melt-blown after multiple cleaning cycles however it remains unchanged in the case of the nanofiber filters during both spraying and dipping mechanism of cleaning.

The comfortability of wearing the facemask is also an important parameter which has been analyzed after the cleaning process. The breathability test is performed for both filters and it is found that the nanofiber filter shows better water vapor transmission rate. In fact, the melt-blown fiber loses the static charges on its surface after ethanol treatment which is the major reason of showing low water vapor transmission rate. The practical breathing ability test showed that the nanofiber filtered facemask is better in terms of transmission of air, moisture and carbon dioxide (CO₂) as depicted in figure 9(c). The extensive discussion on the usability of N95 respirator and comparison with surgical face mask is performed by Li *et al* from the Hong Kong Polytechnic University which also shows that the N95 face mask has better filtration efficiency [154]. However, the subjects wearing masks are not able to differentiate the N95 and surgical masks.

5. Conclusion and outlook

Due to the COVID-19 pandemic prevalence worldwide, the demand on face masks has increased drastically, and so have their prices, especially during the beginning of the pandemic.

Even though there exist a wide range of commercial masks in the market, however, selecting the best one to protect the wearer from the transmission of the SARS-CoV-2 virus under all circumstances is not an easy task, especially with the current commercial availability of face masks with the same type but with different shapes and filtering properties. This is owing to the fact that the only line of defense that is currently available to people, and especially those who are immunocompromised, is the use of face masks, hand sanitizers in addition to social distancing. While international researchers are working side by side in order to scavenge a curative remedy to hinder the further spread of the disease, however, there are still several bottlenecks to providing and large-scale producing well-tested and safe vaccines to the world. In fact, according to the WHO, it might take until 2022 for the pre-COVID-19 life to return. Moreover, since this pandemic has shown the incapability and limitations of the world in providing the needed supply of face masks, it is necessary to join forces and develop alternative approaches and techniques to combat against COVID-19 and potential future pandemics. The bright side amid this emergency situation is the reached level and the promises of the achieved technological advances especially in the fields of nanotechnology. Until now, a considerable amount of work has been conducted on the production of nanotechnology enabled anti-viral agents and vaccines to fight against the SARS-CoV-2, however, they are still far away from being publicly implemented due to the extensive, prolonged and strict regulatory affairs. In this review paper, we suggest that nanotechnology could be more

impactful in the current crisis when implemented in the key area of face masks. More specifically, we have shown that nanotechnology can enable safer, more durable, environmentally friendly, reusable, and lower cost face masks which can be developed using simple fabrication techniques. The different sections of this review article covers different fabrication steps which is required to fabricate any nanotechnology enabled face mask.

In the second section, we have reviewed the different approaches to develop face masks where the nanomaterials are integral components of the PPE. These include the use of nanofibers instead of microfibers in order to be able to filter out particles with dimensions closer to those of the fibers, and therefore to be able to filter viruses such as the SARS-CoV-2. In fact, it has been shown in previous studies that nanofibers have higher capture efficiencies in comparison to microfibers in the submicron particle size range of 100–500 nm because of small fiber diameter and increased surface area of the fibers [155, 156]. However, generally, this comes at the expense of an increase in the pressure drop across the filtering medium in addition to the higher cost of nanofibers compared with microfibers. As a result, many studies focused on using a combination of micro/nanofibers to enhance the filtration efficiency while maintaining a low pressure drop. The different nanofibers productions parameters, such as flow rate of precursor solutions, spinneret gauge, type of collector and viscosity parameter of the precursor solutions have not been previously extensively discussed or reported. Researchers are encouraged to report these parameters in their future works to better assess the filters and for a fairer comparison between them.

The nanofiber-based filters are most commonly being developed using an electrospinning process which is a simple and low cost technique and which allows the functionalization of the filters during the development process. The charging of the fibers using nanomaterials such as silica nanoparticles with excellent charge storage capability has shown the potential of enhancing the filtration efficiency where both, the mechanical and electrostatic attraction mechanisms, come into play. The silica nanoparticles exhibited super-hydrophobic characteristics as well, which allowed the repelling of the water droplets carrying the virus. Filtration efficiencies above 95% against sub-300 nm particles have been reported. Different nanoparticles have also been embedded in the fibrous network during the electrospinning process such as silver, titanium dioxide, zinc oxide, copper oxide and graphene oxide to inactivate the COVID-19 virus, thanks to their anti-viral features. It is reported that the resulting face masks can be developed at a cost of \$1.88/mask and lower. The functionalization of face masks by integrating triboelectric nanogenerators has also been reported, where the outer layer of the mask contributes to electrocuting the virus using the energy that is harvested from simple face motions. While this proposal could be economically challenging, a \$1/mask solution has been reported where the triboelectric nanogenerators self-charge an outer layer of the mask and contribute to an enhanced filtration using the electrostatic attraction mechanism. It is also worth to note that such functionalization has been reported in newly developed and currently commercially available face masks. Finally, straining based filtration has

been demonstrated using sub-100 nm nano-porous yet breathable membranes, however, this was developed using a quite expensive fabrication process and alternative approaches are needed to make this filter economically viable.

In the third section, the recent surface treatments using nanotechnology enabled coatings for anti-COVID-19 face masks are reviewed. Three main purposes for the coatings have been discussed: first, coatings based on a matrix of flexible polymer with embedded nanoparticles such as C, Ag and TiO₂ have shown the potential of de-activating the virus due to their anti-viral characteristics. It is worth to note that several companies have adopted these nanoparticles in their commercial anti-viral coatings. Second, super-hydrophobic coatings using silica nanoparticles and LIG have been developed and showed large water contact angles up to 140 °C, allowing the repulsion of the virus carried by a water droplet. Both types of nanomaterials, antiviral and hydrophobic, have also been mixed in the coatings to enable the deactivation and repulsion of the virus. The third goal of the reported nanotechnology enabled coatings has been focused on the recyclability of the mask using a photothermal laser induced coating which quickly heats up to 80 °C under sunlight and thus contributes to destroying the virus and disinfecting the mask for future uses. In the final section, the usability and reusability of nanotechnology enabled face masks are explained. More specifically, extensive studies comparing nano-fiber based masks and commercially available masks such as the N95 confirm the advantages of the nano-based filters which allow for enhanced filtration efficiencies, increased durability, reusability and recycling capabilities, enhanced breathability and comfort with a lighter weight.

While the presented nanotechnology-enabled approaches have shown great potential in providing enhanced protection to the wearer against the SARS-CoV-2 virus using different filtration, repulsion and inactivation mechanisms, however, the reported studies mostly reported preliminary results with missing details. Thus, for the techniques to be viable solutions, further testing is needed in order to investigate not only the filtration efficiency against sub-300 nm particles, but also, the breathability, durability, reusability, comfort level, cost and scalability. In general, the future generation of face masks should integrate nanomaterials which allow multi-functionality such as excellent filtration efficiency in addition to hydrophobicity, anti-viral, and disinfecting capabilities. This could be achieved by integrating several nanomaterials with distinct roles, such as the case when anti-viral CuO nanoparticles were integrated with hydrophobic silica nanoparticles for instance. Moreover, future face masks could potentially become smarter by integrating nano-based biosensors which can detect the virus. To this end, Seo *et al* and Rodriguez *et al* have recently reported the fabrication of graphene based biosensors for promising application in nasal swabs and saliva swabs, respectively, and showed their capability in detecting the SARS-CoV-2 virus with high selectivity [157, 158]. The different approaches for enhancing the filtration efficiency of the face masks which have been covered in this review paper, as well as their advantages and challenges are summarized in table 2.

Table 2. Comparison of different nanotechnology enabled face masks.

Specification	Nanomaterial	Efficiency (%)	Features	References
Rechargeable Mask	Graphene oxide and polyvinylidene fluoride mixture	>95	The cost of the mask is less than 1 USD and is based on electrostatic rechargeability. The researchers modified the melt-spun polypropylene (PPY) available in conventional N95 mask which acts as the filtration layer.	[99]
Photoactive	Copper nanoparticles	99.37	The Mask is reusable, disposable and offers self-cleaning ability.	[159]
Electrothermal	Graphene	99.8	The mask maintains almost the same removal efficiency for repeated electrifying cycles (>10 cycles) which offers reusability.	[143]
Photothermal	Laser-Induced graphene (LIG)	–	Under solar illumination, the temperature of LIG goes up to 80 °C which makes the mask reusable.	[100]
	Laser-Induced graphene (LIG)	99.998	LIG material can be converted into bio-degradable material. About 100% inhibition rate was achieved for 10 min under 0.75 kW m ⁻² irradiation which still doesn't confirm disinfection SARS or SARS-CoV virus diseases.	[160]
Straining-based filtration	Nano-porous polyimide thin sheet	~100 (theoretical)	No further work is performed to further confirm the results. Fabrication is very expensive which requires cleanroom facilities.	[100]
Nanofiber of polylactic acid and cellulose acetate	Copper oxide nanoparticles & Graphene oxide	–	The cost of each mask is less than 1.88 USD however the study does not confirm the efficiency of the mask and no experimental evaluation is performed to test the filter.	[86]
Nanofiber face mask	Nanoporous polyethylene with Ag coating	99.6	The facemask is used for both outdoor and indoor. It shows high reflectance (87%) which allows for warming purpose as well as comfortability.	[161]
	Electrospun Nano-fibers	–	No further work is performed to confirm the results. The filter can be disinfected using suitable methods and protocols which offers reusability without compromising the filtration efficiency.	[162]
Coating on FFP3 facial mask	Silver nanocluster/silica composite	~100 (theoretical)	No experimental evaluation is performed to test the efficiency of filter. The coating composite can be performed on any metallic, ceramic, polymeric and glasses surfaces. This composite coating increases the working life of filtering mask/media.	[119]

Once extensively tested in a laboratory, face masks need to be sent out for certification and approval which is needed for transferring the proof-of-concept masks into commercial ones. In fact, face masks are normally controlled by national medical device regulations. The rules outline the legal obligations, to certify that the masks provide the wearer with the required protection against certain specified risks. The different requirements depend on the classification/category of the mask and are normally explained in the referenced performance standards. It should be noted that the requirements vary based on the country. Nevertheless, the main requirements for certifying a specific mask include the registration and approval of the PPE, the specification of its category and the passing of several tests including particulate and bacterial filtration efficiencies, differential pressure which dictates the breathability, blood penetration resistance, flammability, microbial cleanliness and biocompatibility. Nanomaterials based face masks need to pass these tests in order to achieve their translation into actual components which can be impactful during the current pandemic. Additional regulations which might be unique to nanotechnology enabled face masks could include the testing of the potential health hazards of the used materials. To overcome this, metallic nanomaterials could be replaced with biodegradable lipid-based nanomaterials, for instance, in order to avoid their adverse effects on the wearer health and the environment. Finally, this review confirms that nanotechnology can be significantly impactful in fighting against the current crisis when implemented in the key area of face masks, especially that this area is not associated with some of the stricter protocols normally associated with vaccines, thus quicker technology translation could be achieved.

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Data availability statement

No new data were created or analysed in this study.

ORCID iDs

Nazek El-Atab <https://orcid.org/0000-0002-2296-2003>

Rishabh B. Mishra <https://orcid.org/0000-0003-2609-1644>

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