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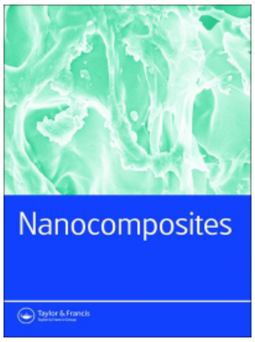

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RESEARCH ARTICLE

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New generation washable PES membrane face mask for virus filtration

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ABSTRACT
Membrane materials might be used for face protection because they can decontaminate the inhaled air from particle pollution and viruses like the SARS-Cov0-2 which damages our respiration system. In this study, plyethersulfone membranes (PES) were synthesized with green solvent at room temperature and its filtration effectiveness was investigated against nano-bacteria (size 0.05 to 0.2 μm) by measuring their Bacterial Filtration Efficiency (BFE) and micro aerosol size (0.3 μm), and Particulate Filtration Efficiency (PFE). The average SARS-CoV-2 diameters are between 50 nm to 160 nm. A series of experiments were performed to accomplish between 0.03 to 0.21 μm PES sponge like diameters so that can be used for SARS-CoV-2 filtration. Results showed that nanofiltration/ultrafiltration could filter 99.9% of bacteria and aerosol from contaminated air the size of the Covid-19 molecule.

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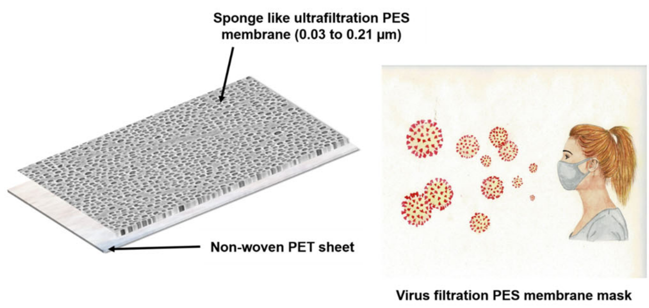
Membrane filtration; PES membrane; Corona-virus; microfiltration; nanofiltration; air filtration; high efficiency; fresh breathe

GRAPHICAL ABSTRACT



Highlights

- A washable innovative PES membrane face mask has been developed.
- The virus removal efficiency of the PES membrane face mask reaches ~99.9%.
- The PES membrane face mask is washable and reusable retaining its efficiency.

1. Introduction

SARS-CoV-2 (the 2019 coronavirus pandemic (Covid-19)) is responsible for causing acute respiratory symptom, high contamination and death. Covid-19 is still a pandemic and continues to spread and mutate becoming a major threat to public health, infecting more than 219 M people worldwide with over 4.55 M deaths as reported on Oct 2021 [1,2]. Covid-19 might be transmitted from spit droplets, airborne, fomite, fecal-oral, blood borne, and animal-to-human contact. Covid-19 may cause acute myocardial injury, severe pneumonia, and chronic damage, resulting in a mortality rate between 1.5 to 2.5% [3,4]. Researchers examined the size and content features of the SARS-CoV-2 particles in addition to the mechanism of transmission. Different studies have produced various findings

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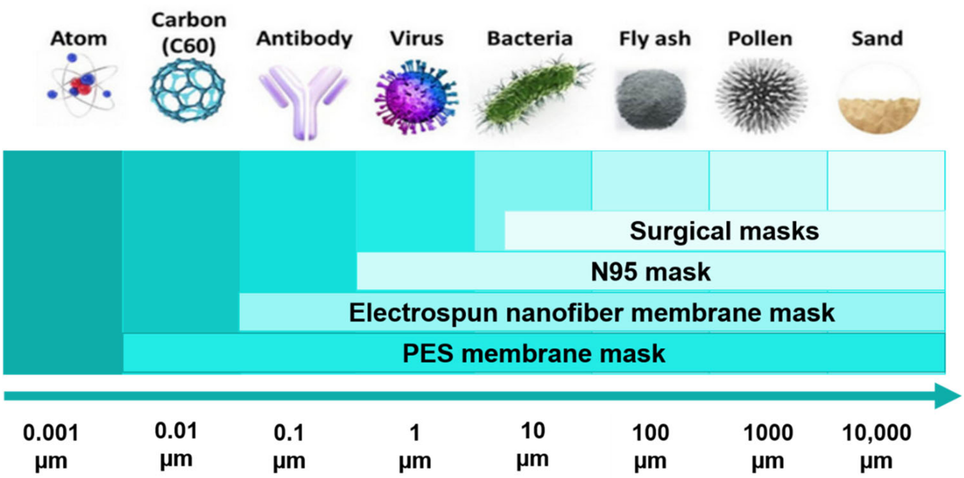

Figure 1. The filtration performance comparison among PES membrane, electrospun nanofiber membrane mask, N95 mask, and surgical facemask.

when using electron microscopy to examine negative-stained SARS-CoV-2 particles, and reported that the virus's diameter ranges between 50 nm to 140 nm [5].

Covid-19 virus primarily causes respiratory sickness, ranging from moderate to severe, affecting the lungs and their function which might be lethal. Whilst some people who are infected never show any symptoms, they can infect others which is difficult to detect, but dangerous for spreading transmission. Spit droplets and aerosols are mostly important for the rapid spreading of Covid-19 [6,7]. An infected person can release the virus *via* coughs and sneezes [8]. According to recent studies, aerosols and respiratory droplets ejected during sneeze/cough can travel up to 12 to 26 feet [9,10], which is substantially further than the (CDC)- 6-feets social distancing guideline [9,11]. In addition, droplets can be stable in the air for an extended period of time due to their micro-meter/nano-meter size and negligible gravitational effect, posing a threat of airborne transmission, especially in enclosed spaces with inadequate filtration systems. It was confirmed by the World Health Organization (WHO) that Covid-19 is characterized as airborne and can remain 8 h in the air, so people are asked to wear face masks to protect themselves and others from contamination in public places.

Before a significant percentage of the world's population is vaccinated, wearing a mask in public places might be the most effective weapon to reduce the spread of the virus. Because of this reason, masks have become a necessity in every day's life. Commercial surgical face masks, and N95 masks typically use melt blown non-woven tissue paper as a virus filter sheet. PET (Polyethylene terephthalate) melt blown nonwoven used mask can be worn only a few fours and then get disposed. [12], as shown in Figure 1. Worldwide face mask shortages were exacerbated by very high demand, as a result of this, while face coverings or homemade masks can help to protect from bacteria, there is no scientific evidence that they are effective against Covid-19 virus, mainly because most of them will have much bigger pore size than the Covid-19 virus. Pore size is one of the most important parameters in the fabrication of masks for Covid-19, so that, the next generation reusable and anti-virus nano pore filtration size masks may be necessary [13]. However, reusable masks should prolong the use of the mask, not at the expense of filtration effectiveness. Anti-virus masks can promptly shield as well as destroying the virus in the filter of the mask, preventing virus retention [13–15]. The invention of such a novel mask would aid us in dealing with epidemics such as COVID-19.

Many researchers have reported on the fabrication of face masks by electrospinning and producing nanofiber membranes. Zhang et al. employed melt-blown Polypropylene(PP) non-woven fabrics with a fiber diameter of 0.5–10 µm [13,16]. Cheng et al. reported on an electrospun polyetherimide non-woven bi-functional material for an innovative face mask [17].

Ultrafiltration or nanofiltration membranes, whose width is measured in micro or nano-meters, are highly regarded for air filtration applications due to the high surface area and sponge-like inter-membrane pore sizes less than 0.1 µm. These ultrafiltration or nanofiltration membranes supported with a non-woven fabric might be a good candidate for mask effectiveness, because it can possess high level of air permeability promoting user comfort and as well reuse [18,19]. Polyether sulfone (PES) is a soluble polymer, and the PES-based ultrafiltration or nanofiltration membranes have shown high chemical resistance, thermal and mechanical stability and hydrophobicity, making them suitable for air filtration due to their nano sponge-like pore size distribution, while virus protection performance by the pure PES membrane can be adjusted by the addition of a catalytic process [20].

PES-based membrane products have an excellent homogeneous sponge-like pore size membrane structure. The sponge-like membrane pore size distribution mechanism is explored to improve virus filtration. The filtration performance of separation membranes with additional gradient structural change has been previously reported [21]. Furthermore, because these virus particles are resistant to inactivation procedures such as low pH, as in the case of the reported parvovirus elimination [22,23]. When these membrane materials are washed and reused several times, and potentially the total membrane virus particle attachment maybe high to null, divergent flow filtering is preferable [22].

This novel research introduces a washable and reusable innovative sponge-like structure with a non-woven supporting PES membrane for face mask use. The non-woven support PES membrane face mask can effectively prevent the aerosol and nano-bacteria/virus sized particles during the inhaling process. This novel material shows the following advantages: (1) uniform pore size, sponge-like pore size distribution, and virus filtration by the non-woven support PES membrane by phase inversion *via* immersion precipitation method; (2) the filtration efficiency of particles (size of between 0.01 to 0.2 μm) and (3) the durability of the PES-based face mask with excellent working efficiency, which can continuously protect from the virus more than 72 h. This novel work reports on a new efficient, washable, and reusable PES membrane for face mask end uses.

2. Experimental

2.1. Materials

This research was carried out using polyethersulfone (PES), ultrason E6020P (average M_w: 6 58 kDa,) from BASF, Germany. Polarclean® was purchased from Solvay Fine Chemical Additives (Qingdao) Co., Ltd., China. polyvinylpyrrolidone, (PVP)-K30, was purchased from Chemical Reagents Co., Ltd, China. The non-woven PET fabric was supplied by Guocheng CO. (Wuxi, China). Sodium hydroxide (NaOH) was obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). All chemicals and reagents were used without further purification.

2.2. Membrane preparation

The membrane solution was prepared by dissolving a certain amount of PES in a Polarclean® solvent bath, and the PES has successfully solubilized into the solvent at 60 °C temperature. The 2 wt % PVP additives were used by continuous magnetic stirring at 60 °C temperature for 1 h or until a completely clear and uniform solution was obtained. The hydrophobicity, surface charge, and roughness of the membrane are dependent on the blending ratio of PES and PVP. Using a casting knife, the polymer solution was then cast onto a glass plate, and the cast film's solvent was allowed to gently evaporate at room temperature overnight. To complete precipitation and membrane development, the glass plate containing the cast film was gently submerged into a water bath for 6 h. Then, pure water is used as the coagulation bath, and the non-solvent induced phase separation method is used to obtain the PES flat membrane by scraping. Prior to UF operation, the membranes were maintained in deionized water. The PES membranes exhibit better antifouling ability and have a more sponge-like pore size structure because of the increase in positive polar charge when the PES and PVP blend get in contact with the solvent.

2.3. Characterization

The solution viscosity of the embrane sample was measured using a Rotational Viscometer (NDJ-8S Digital Viscosity Meter, Novel Scientific Instrument Co., Ltd, China) at ambient temperature. Membrane sample contact angles were assessed using a dropmeter^{TM} (dropmeter^{TM}-A-300-main st vision, Kudos precision Instruments, USA). The membranes morphology was examined using scanning electron microscopy (SEM) (Phenom XL, Phenom world, Thermo Scientific, Japan) at an accelerating voltage of 5 kV. Fourier transform infrared spectrum (FTIR) was recorded from 400 to 4000 cm-1 by using (IR, Interspectrum, low noise DLATGS, FTIR-920, Estonia). The thermal decomposition behavior of the membrane was studied using thermal gravimetric analysis (TGA) (TG 209 F1 *Libra*® Netzsch company, United Kingdom), under a nitrogen atmosphere.

The prepared membrane pore size was determined using prostate-specific membrane antigen-10 (PSMA-10, Nanjing GAO Qian functional Materials Technology Co., Ltd., and China). The pore volume (mL/g) at specific pore sizes (m) ranging from 0 to 0.30 mm was measured for membrane air permeability and selectivity characteristics. Membrane pore sizes were calculated from the smallest to the largest, with the mean flow pore diameter representing the primary pore size. The thickness of the membrane was measured with a digital micrometer Shanghai Liuling Hand-Type Qianfen Thickness

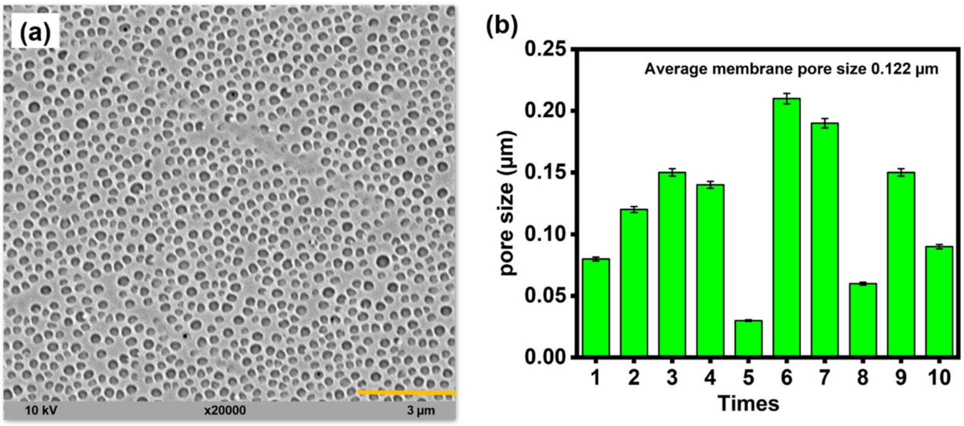

Figure 2. (a) SEM image of PES membrane, (b) equal pore size distribution (sponge-like).

Gauge CH-1-S Plastic Film Sheet Hand-Type Thickness Gauge, which has a precision of 0.001 mm.

2.4. Tensile strength

Talukder et al. reported a procedure of tensile strength for membranes [20]. The tensile strength and elongation of five samples from each membrane measuring 50 mm in length were measured at a constant elongation rate of 20 mm/min up to the breaking points of the PES membrane using a tensile strength tester KD-||| model BA-100m by Transcell Technology, China.

2.5. Filtration efficiency (FE)

The Food and Drug Administration (FDA) issues ASTM standards as the recognized standard in the United States. ASTM F2100-11 (2011) is a fundamental standard that sets the performance requirements for respirators and face mask (3 Tips for Choosing the Right Face Mask) [24]. The ASTM F2100-11 standard outlines the required characteristics and testing methods for the materials used in the manufacture of face mask for use in hospitals, health care, and patient care. In the 42 CFR Part 84 certification process, there are several techniques for measuring filtration efficiency, including particle filtration efficiency (PFE), bacterial filtration efficiency (BFE), virus filtration efficiency (VFE), and NIOSH [25]. Material efficiency is linked to the PFE and BFE techniques, which are employed as a barrier to protect the user from aqueous viral aerosols. The filtering efficiency test is carried out according to the ASTM F2100-19E1 methodology, which uses a nano-size salt aerosol/bacteria [26]. Eq. (1) is used to calculate the filtration efficiency of mask and respirators, where Cu and Cd are the average particle concentrations per each upstream and downstream test specimen [27].

$$\text{Filtration efficiency (FE)} = \frac{Cu - Cd}{Cd} \quad (1)$$

2.5.1. Bacterial filtration efficiency (BFE) experiments

The ASTMF 2101 standard Test Method was used to evaluate the Bacteria Filtration Efficiency (BFE) of the material. Here, biological nano-bacteria were used and, the virus filtration efficiency was tested by obtaining the filtration percentage by comparing between the nano-bacterial control counts and the test article effluent counts. The challenging part in this test are the nano-bacteria (size 0.05 to 0.2 µm). A nano-bacteria mixer liquid solution was passed throw to the filtration medium at a 28.3 liters per minute (LPM) flow rate [28] for 4 h at a 21 ± 5 °C temperature and relative humidity of 85 ± 5%.

2.5.2. Particulate filtration efficiency (PFE)

According to the FDA guideline paper, the PFE of the various devices was tested with unneutralized 0.1 m PSL particles [29,30]. PFE testing was carried out using the whole PES membrane mask material. According to the ASTM 2299 procedure, the test velocity was between 1 and 25 cm/sec [27,31]. Prior to testing, the test samples were preconditioned at 30–50 percent relative humidity (RH) at 21 3 °C [31]. An automatic Particulate Filter Efficiency PFE Tester GT-RA09 from GESTER INTERNATIONAL CO. LTD. (China) was used to investigate the filtration efficiency. Concentrations upstream and downstream of the respirator were monitored at a flow rate of 80 L/min, with 2% accuracy. The user simply needs to insert the filter paper in the fixture and push the button to change the test flow; the system will test the resistance and efficiency automatically *via* the controller. The PFE is calculated by equation (2). For each test material, the upstream count was measured before and after the downstream count. Both upstream and downstream counts were measured three times for one minute each.

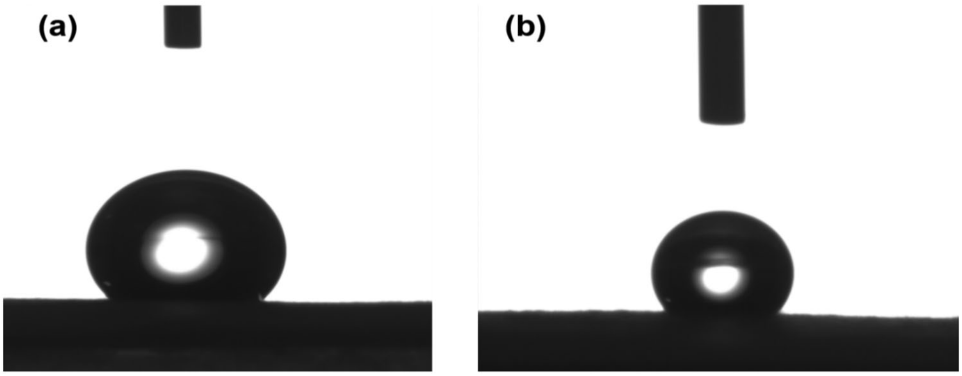

Figure 3. Contact angle of PES membrane for the face mask (respectively 120.1° and 121.5°).

$$\text{Particulate filtration efficiency (PFE)} = \frac{Cu-Cd}{Cd} \times 100 \quad (2)$$

The PFE findings range from 1 to 99.99 percent, where Cu and Cd are the averages of upstream and downstream counts. The greater the percentage, the better the mask filtration. For the PFE test, particle sizes ranging from size 0.05 to 0.2 µm can be measured. When comparing the test results, the particle size (e.g. size 0.05 to 0.2 µm) must be taken into account, since using a particle with a greater size might lead to a deceptive PFE evaluation.

3. Results

3.1. Membrane characterization

The viscosity and electric conductivity of the solutions were 2465 mPa/S, and 1.4 µS/cm, respectively. The morphology of the PES membrane was analyzed using SEM, as shown in Figure 2a. The PES membranes have a uniform structure with an average membrane thickness at approximately 0.5 mm and 0.3 mm, as shown in Figure 2a. The SEM image shows the surface roughness of the PES membrane. As shown in Figure 3, the PES membrane was shown to exhibit hydrophilic behavior, with contact angles of 120.1° and 121.5°, as shown in Figure 3a and b.

3.2. Pore size

Sponge-like pore size distribution of PES membrane is depended on membrane diameter and on membrane fabrication environment such as temperature, humidity, coagulation bath temperature. The withdrawing air pressure through the PES membrane was 145 to 200 kPa and shown a pore size distribution range of 0.03 to 0.21 μm. The pore size distribution experiment was reported 10 times and repeated an average pore size of 0.122 µm, as shown in Figure 2b.

3.3. Sponge-like structures adjustment control

As an excellent porogen, PVP can effectively increase the porosity of the filter membrane and at the same time increase the hydrophilicity of the membrane. It is widely used in the preparation and modification of membrane materials. The addition of non-solvent additives, such as small inorganic molecules and small organic molecules, has become an important method for adjusting the structure of membrane materials [20,32]. Additives can effectively affect the mass transfer rate of solvents and non-solvents, thereby forming different pore structures. This experiment uses PVP as an additive to control the structure of the polyethersulfone membrane material.

3.4. Adjustment of membrane pore size and its performance

The pore size and its distribution are important indicators for the application of membrane materials, which determine the filtration performance and application fields of membrane materials. In this experiment, the effects of the solid content of PES and the content of solvent in the casting liquid on the structure and pore size of the membrane were investigated. Under the condition of ensuring certain content of solvents and additives in the casting liquid, the effect of solid content (PES mass fraction) on the pure water flux of the PES membrane was investigated. The results are shown in Figure 2. It can be seen from the figure that as the solid content increases, the viscosity of the casting liquid increases, the force between the polymer molecules becomes larger, the movement space between the polymer molecules becomes smaller, the double diffusion speed between the solvent and the non-solvent becomes slower, the liquid phase separation rate becomes slower, and the structure of the prepared PES separation membrane tends to be more dense [33], which leads to a decrease in the pure water flux. And as the solid content of PES increases further, the viscosity of the material liquid becomes too

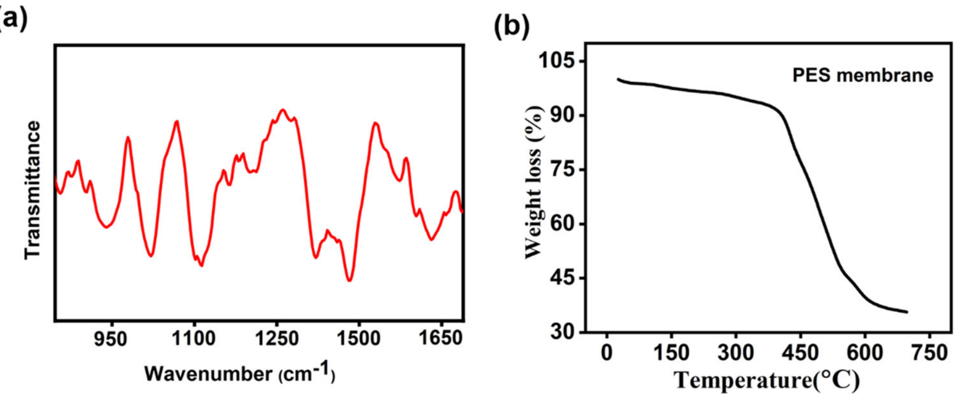

Figure 4. (a) FTIR spectra of PES membrane, (b) TGA results of PES membrane.

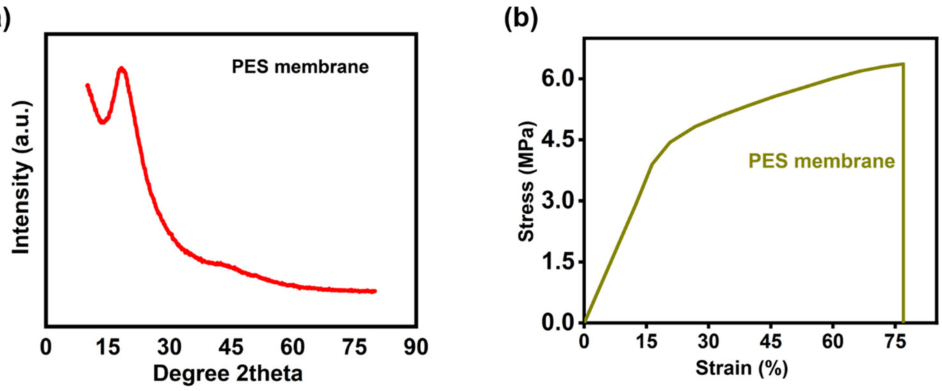

Figure 5. (a) XRD results of PES membrane, and (b) Tensile strength of PES membrane.

large and the uniformity of the film formation becomes worse. Therefore, the solid content is maintained at 16% to 18%, which can ensure that the PES membrane has good film-forming properties and pure water flux. Using Polarclean® as a solvent for membrane material preparation, and under the premise of ensuring the consistency of solid content and porogen, using acetone as the pore size regulator, the effect of Polarclean® content on the cross-sectional structure, pure water flux and rejection rate, pore size and pore size of the PES membrane was investigated. The results of the influence of porosity are shown in Figure 2.

The FTIR spectra of the PES membrane is shown in Figure 4a, and its functional group was analyzed on different spectra. Four major peaks may be seen in the functionalized self-made membrane. Repeated ether and sulfone linkages alternate between aromatic rings in the PES. As a result, the stretching vibrations of S = O symmetric and S = O asymmetric may be ascribed to the bands at 1145 and 1261 cm^{-1}, respectively [34]. And, the bands at 1665 cm^{-1} represent PVP's amide group (OH). The spectrum of the PES/PVP membrane, on the other hand, reveals that PVP was maintained in the membrane structure. Another unique absorption peak at 1305 cm^{-1} and 1663 cm^{-1} is present in the C = O functional group vibration stretching [35]. These peaks indicate the presence of PVP in the PES membrane which is connected to PVP residue. PVP stretching vibration increased with increasing of PVP ratio from 1 wt% to 10 wt%, as illustrated in Figure 4a.

According to the TGA curves, the thermal decomposition behavior temperature for the PES membrane was relatively stable up to 420 °C (5% weight loss) [36]. As shown in Figure 4b, its thermal degradation was demonstrated a one stage weight loss. The PES membrane was stable up to 420° C without significant weight loss, as compared to 450 °C for the pure PES membrane, concluding that adding PVP influences thermal degradation on the PES membrane [37,38].

The crystallinity of the PES membrane was analyzed by WAXD, which shows the crystallinity properties of the PES membrane. As shown in Figure 5a, the membrane was characterized as a typical amorphous structure with peaks at 2θ a.u. values of 16–19°.

3.5. Tensile strength

The tensile strength of the PES membrane is anessential fact for face mask application, with its stress-strain properties shown in Figure 5b. The tensile properties and breaking elongation rate are enhanced because of the specific intermolecular interactions by the additives in the PES membrane.

The elongation at breaking point and tensile strength values are summarized in Table 1, with 6.4 MPa tensile strength, and 77% elongation at break, [39] being higher than the ENM mask [40]. This rise could be attributed to the increase in PES membrane surface area.

3.6. Virus filtration test

The BFE method was modified, and Staphylococcus aureus (S. aureus) was replaced by nano-bacteria (the hypothesized nano-bacteria are mostly 0.05 to 0.2 µm in size) as the test specimen [41], which is roughly similar to the SARS-CoV-2 diameter [42]. This precautionary change was applied to this experiment, so that the chosen nano-bacteria can represent SAR-CoV-2 [21]. 100 L of 8 10^5 PFU/mL nano-bacteria is used in sterile water at a flow rate of 28 L/min through the membrane (shown in Figure 6) under normal respiration range and cascade impact or constraints [43]. The filtration pressure was kept at 35 kPa throughout the suspension.

Nano-bacteria was passed through the membrane face mask on E. coli (Escherichia Coli) plates within the 6 stage cascade impactor. The E. coli plates were incubated overnight at 37 °C. The control plaques could enhance the performance of the PES membrane, and the positive hole correction of multiple-jet impactor was counted and recorded [44]. The positive hole was calculated for each of the 6 stages and added together. The average number was counted. To assess the number of viable nano-bacteria generated, the positive control system was employed without a PES membrane. On the triple side, the test system was performed, and the negative control system was completed without the nano-bacteria. And then, the negative system was done by air sample in aerosol chamber on the triple part [45]. The average nano-bacteria size was 0.10 µm, which is the average size of Covid-19 virus droplets produced by coughing. Bacteria filtration efficiency (BFE) was calculated by comparing the average positive hole number corrected of nano-bacteria captured after the PES membrane mask, compared with the positive control. The BFE for each PES membrane mask was also calculated without the largest size nano-bacteria, yielding an average nano-bacteria size of 0.1 µm [24]. This more accurately portrays the amount of inhaled aerosol reaching the lower respiratory system and alveolar region of 0.3 µm.

Table 1. Tensile strength and elongation (%) of PES membrane.

Membrane type	Tensile strength (MPa)	Elongation (%)
PES membrane	6.4	77
ENM mask [40]	4.1	38

3.7. Particulate filtration efficiency (PFE)

The particulate filtration efficiency was tested by Nacl testing methods with adjusting the rotary flow rate 20 mg/m^3. When the upstream concentration is stabilized, it automatically changes into the downstream concentration test, and the curve is observed. When the downstream concentration is stable, the experiment starts counting until the end. The results reveal an upstream concentration of 20.900 mg/m^3 and downstream concentration 0.243 mg/m^3, producing 99% filtration efficiency, as shown in Figure S1.

3.8. Hepa PES membrane mechanism

A HEPA filter is designed to collect very small particles and does not operate like a normal membrane

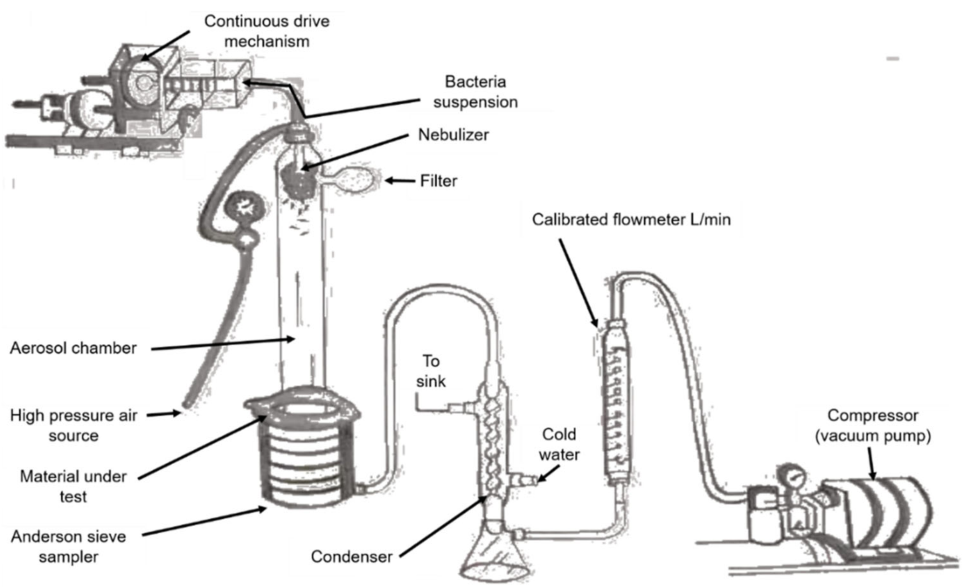

Figure 6. Bacteria filtration efficiency process and apparatus according to ASTM.

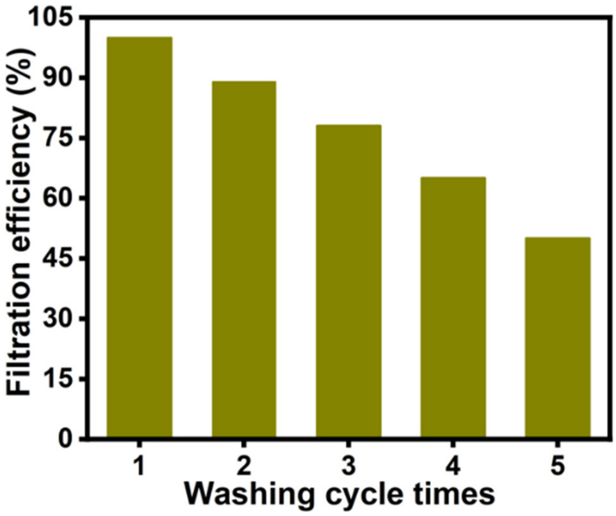

Figure 7. PES membrane virus filtration efficiency against washing cycles.

filter, which captures particles bigger than the filter's pore size. Instead, HEPA filters capture particles using a mixture of three methods. Interception is the first process, in which particles are transported in the airflow around the filter fibers attached to the filter. To be collected, particles must be within one radius of the filter fiber. The second process, impaction, is frequently used to collect larger particles. Because of their size, these particles are unable to respond to abrupt variations in airflow around the filter, and instead rush towards and embed themselves in the fibers of the filter. Diffusion is the last process, which happens as a result of how tiny particles travel and interact with surrounding molecules. Brownian motion describes how molecules move in a random, zig-zag manner as they clash with neighboring molecules.

3.9. Washable and reusable

Ethanol is an extensively used disinfectant, and has been shown to lower coronavirus infectivity by a factor of four or more [46]. Although masks cleaned by soaking in 70% ethanol for 2 h had no effect on ΔP, they did exhibit a significant drop in BFE [47]. The filter efficiency distribution across particle sizes appeared to be affected by ethanol treatment, with the MPPS changing from 0.03-0.21 µm to 0.05-0.23 µm pre- and post-treatment [47].

The reusing conduct of the PES membrane was researched during five progressive reuse cycles to explore PES membrane execution. The used PES membrane was recuperated by shaking in 1.0 mol/L NaOH arrangement at 50 rpm for 0.5 h, followed by centrifugation. The recovered adsorption limit of the PES membrane has appeared in Figure 7. The reused PES membrane capacity stayed flawless for the initial two cycles and somewhat dropped after five cycles. After five recovery cycles, the Filtration Efficiency of the PES membrane decreased by 50%, showing that the PES membrane can be used after washing.

4. Discussion

Handling, appearance and skin comfort of the PES membrane are good because it is soft, smooth, dust free and not irritating to the nose and face. Fabricated PES membranes show hydrophobic behavior, which could be used for face masks. The average coronavirus diameter ranges between 50 to 160 nm and those sizes could be filtered *via* the sponge-like pore size (0.03. ton 0.21 µm) of this PES membrane. Moreover, its tensile strength is two or three times higher than any ENM [40,48]. After weighing, the membrane was found to provide enough virus protection. More importantly PES membrane is capable of filtering aerosol and bacteria with 99.9% efficiency, by its ability to filter particles from air streams. They could be essential component in respirator and face mask filter materials.

5. Conclusion

With the Covid-19 epidemic, face masks and respirators ware becoming an everyday necessity potentially for years to come. PPE is subjected to a range of tests to assess its performance and suitability under various conditions. The PES membrane has soft handle and smoothness and does not irritate the skin around the face. This study has demonstrated the high filtration efficiency of the PES membrane. The PES membrane was used in a direct flow configuration. The filtering surface of asymmetric membranes served as the downstream membrane surface. To achieve optimum rejection of viral particles and passage of product species, the membranes must have a very narrow pore-size distribution. When constructing viral filters to exclude small parvovirus particles, this is very crucial. This PES membrane can be used for face masks and has the potential of being certified for use to protect against contamination.

Disclosure statement

No potential conflict of interest was reported by the authors.

Authors' contributions

MET has conceived the idea, conducted the experiments, and written the initial draft. MET, FA, MNP, WJ, FH, GKS, VN analyzed the data and prepared the final draft.

HS has administered the project. All authors have read and approved the manuscript.

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