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PAPER

Chitosan-coated electrospun waste Polystyrene microfiber as mask filter

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ABSTRACT

Waste-expanded polystyrene is one of the significant contributors to global waste. Its disposal has been a problem due to its low density, causing too much space taken up in landfills. This study utilizes waste-expanded polystyrene as a mask filter through the electrospinning process. It is annealed and coated with chitosan to improve mechanical strength and bacterial filtration efficiency. The annealed fiber mat has an average fiber diameter of 2.80±1.24 µm while the annealed and coated fiber averaged 3.52±1.87 µm. The bacterial filtration efficiency test shows 99.84±0.22% efficiency for the coated filter and 64.58±8.84% efficiency for the non-coated filter. The results imply that incorporating chitosan significantly improved the filtration efficiency of the filter. Moreover, the addition of chitosan increased the ultimate tensile strength from 0.72±0.08 MPa to 9.86±1.24 MPa and turned the hydrophobic EPS microfiber (89.83±1.48° contact angle) to a hydrophilic microfiber (51.61±4.47° contact angle). On the other hand, there is no significant difference in the differential pressure between the coated (5.40±0.17 mm H2O per cm2) and non-coated (5.58±1.54 mm H2O per cm2) mask filter. Adding chitosan does not increase the pressure drop across the filter. Lastly, the produced chitosan-coated waste-expanded polystyrene mask filter qualifies for ASTM Level 3 for medical and surgical masks regarding bacterial filtration efficiency and differential pressure. Thus, the electrospun EPS microfiber mask filter can potentially be applied to standard surgical masks while presenting a simple and environmentally friendly waste EPS upcycling.

KEYWORDS

Waste Expanded Polystyrene, D-limonene, Chitosan, Electrospinning, Mask Filter

1 INTRODUCTION

Due to its excellent insulating and protective properties, polystyrene (also known as EPS Foam or Styrofoam) is a widely used plastic packaging material in food, furniture, electronic goods, and appliances. Moreover, it is also used to manufacture valuable products such as disposable cups, trays, cutlery, cartons, and cases [1]. Despite polystyrene's attractiveness, municipalities and organizations are grappling with a growing problem with packaging and product disposal since polystyrene is a non-biodegradable material [1], [6]. Polystyrene is produced by over 15 million metric tons annually worldwide [2]. Around 4 to 12 million metric tons of plastic packaging are swept down rivers in Southeast Asia and China and end up in the oceans [3]. With the statistics growing, serious efforts were made to inhibit polystyrene. There have been state-wide and citywide polystyrene bans in some states of the U.S., wherein to-go foam containers are prohibited in restaurants, caterers, coffee shops, and grocery stores [2]. However, these legislative efforts have yet to be enforced and will take effect at most four years from now. The Philippines, in particular, produces around 200 tons of waste foamed polystyrene every month, 70% of which is from Metro Manila [4]. Since the decomposition of this plastic takes hundreds of years [3], this poses a severe threat to the marine environment.

Currently, most plastic waste is released into the environment in some way or disposed of in landfills at an estimated rate of 12,000 tons per year, occupying 240,000 cubic meters of space [5]. Polystyrene recycling and reuse are thus desirable and necessary to reduce the massive amount of PS waste [6]. Efforts to utilize and recycle waste polystyrene are done and made possible in various fields such as air [7] and water filtration [8], construction [9], insulation [10], [15], nanotechnology [11], etc. There are three common ways to recycle waste polystyrene: mechanical, chemical [12], and thermal recycling [13]. Moreover, there were attempts to convert waste polystyrene into nanofiber using electrospinning [7], an attractive method due to the improved properties of its products. Its application is increasing [14], [15]. However, no studies utilized electrospun waste polystyrene in face mask filters.

The application of waste PS in face mask filters is a novelty. This upcycling method is very timely because of the pandemic we face today. Covid-19 has infected 216 million people and is responsible for around 4.5 million deaths worldwide [16]. With the spread of the virus, protective face masks became in demand and were considered national essentials [17].

Polystyrene is considered one of the suitable polymers for the production of medical-grade surgical masks [18]. One study used electrospun polystyrene with cellulose nanocrystals to produce filter materials exceeding N95 standards [19]. A fiber membrane from high-impact polystyrene waste was also used as a filter medium for PM2.5 with a filtration efficiency of 98.75% [20]. These studies show the potential of polystyrene as an alternative material to polypropylene, which is currently the most common material for producing 3-ply surgical masks [21] and N95 respirators [22]. Meanwhile, chitosan has long been keeping its significance in antimicrobial and antiviral applications. Chitosan nanoparticles were also studied due to their advantage on the larger surface area, thus increasing their binding effect on the bacterial surface [23]. Some studies include the modification of chitosan with bromine ion to improve antiviral activity against Newcastle disease virus [24], chitosan as a component of an influenza vaccine [25], immune system stimulator against H7N9 virus [26], and many more.

Facing the threat of this health crisis, the idea of fabricating a chitosan-coated microfiber mask filter made with waste-expanded polystyrene using the electrospinning technique materialized. This poses a potential for a sustainable source of material for mask filters and helps battle the problem of plastic waste management. Moreover, compared to other ways of producing polystyrene microfiber through electrospinning, it was synthesized with d-limonene, an organic, less harmful, and widely available solvent. Additionally, to create a mask filter with improved antibacterial properties, electrospun polystyrene was coated with chitosan.

The study aims to upcycle waste-expanded polystyrene through electrospinning as a mask filter material. The objectives include testing the mechanical and physical properties, bacterial filtration efficiency, and calculating its theoretical pressure drop to ensure conformation with ASTM standards.

Moreover, the focus of this study mainly revolves around the upcycling of polystyrene and the fabrication of an electrospun mask filter out of it. The produced mask filter is not intended to compete with the existing mask filters but simply to comply with the qualifying standards set by ASTM. To summarize, using a simple and environmentally friendly synthesis process, a mask filter was produced from both considered safe and resource-friendly materials.

2 MATERIALS AND METHODS

The waste EPS- d- limonene and chitosan solutions were prepared separately (Fig. 1). the EPS solution was then converted into a microfiber using the electrospinning technique. Annealing of the sample for both coated and not coated and characterization with SEM followed. Lastly, the chitosan-treated EPS filter material was subjected to a Bacterial Filtration Efficiency (BFE) test, and its breathability was determined theoretically.

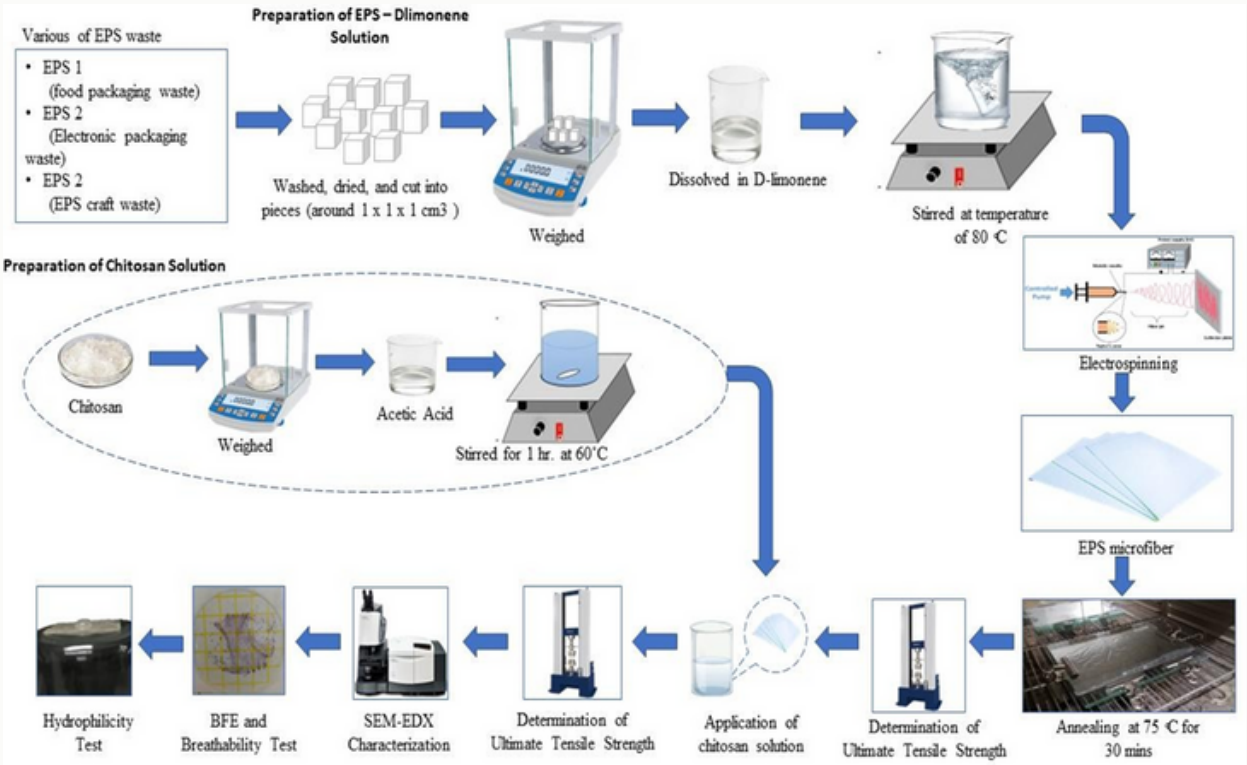

Fig. 1 Schematic diagramon the procedural steps of the study

2.1 Materials and resources

Industrial grade chitosan (≥85% degree of deacetylation, crustacean shell source) was used for the coating solution of the produced microfiber and D-limonene (≥95% purity, Xi'An Pincredit Bio-Tech Co., Ltd., China) as a solvent for waste expanded polystyrene.

Waste-expanded polystyrene from fish boxes, electrical consumer goods packaging, and building insulation panels were collected from households in Davao City, serving as the precursor solution's primary polymer. Glacial acetic acid (≥99.7%, Panreac Quimica Sau, Spain) was used as a solvent for a chitosan-dilute aqueous acetic acid solution. All chemical reagents were used without further purification.

2.2 Preparation of waste expanded polystyrene

Collected waste expanded polystyrene was washed with deionized water thrice, sun-dried, and cut into (1 cm3) pieces [28]. The expanded polystyrene was then rewashed thrice and sun-dried.

2.3 Preparation of expanded polystyrene-d- limonene solution

Prepared polystyrene was dissolved in d- limonene at 29% wt. 71 g of d-limonene was weighed and placed in a 500-mL beaker. Then, 29 g of the previously prepared waste EPS was dissolved. To obtain a homogeneous solution, the EPS solution was stirred on a hot plate (80˚C, 30 min) [27].

2.4 Electrospinning process

Expanded polystyrene solutions were electrospuned using the electrospinning equipment (Biobase HK-400). The 10 mL syringe (23-gauge needle) was filled with the precursor solution. 0.8 mL/hr flowrate was used, and 15 kV of power was supplied to the tip of the needle, 10 cm away from the drum collector, with a length of 25 cm and a diameter of 3 cm, and at room temperature [28]. The thickness of the microfiber filter material was determined by the thickness produced by one syringe only. After electrospinning, the microfiber was left in the collector for 5 min. for further evaporation of the solvent.

2.5 Annealing

The microfiber sheet was placed between the aluminum foil and two (2) transparent glasses, clipped on the sides for even compression. It was then placed in an oven at a temperature of 75˚C for 30 minutes. This was done to augment the mechanical properties of the fibers produced.

2.6 Preparation and application of chitosan- dilute acetic acid solution

Chitosan (1%, 3%, 5% w/v) solutions in (2% v/v) acetic acid were prepared. Chitosan and dilute acetic acid suspension were stirred for one hr. at 60˚C to achieve a homogenous solution. The expanded polystyrene microfiber was soaked in the chitosan solution for 2 min. while shaking [29].

2.7 Determination of Mechanical Properties

Microfiber samples were cut 1 cm wide and 7 cm long [30] and weighed in preparation for the mechanical property testing. The Digital Electronic Tensile/Compression Testing Machine (Laryee Instruments, UE1501) was used for this test to determine the ultimate tensile stress of the samples.

2.8 Determination of bacterial filtration efficiency

Bacterial filtration efficiency is one of the most critical parameters for filter materials for medical use, according to ASTM F2101 and JIS T 9001. This study employs a test similar to the one described elsewhere in detail [31]. The test used an aqueous solution of Staphylococcus aureus (recommended by the ASTM F2101 standard) to be converted to an aerosol (3.0 µm) by a nebulizer. At a constant flow rate of 28.3 L/min, the aerosol was directed toward the filter material covering a Petri film. A test without the filter was also performed to differentiate the number of bacterial colonies in the Petri film. The bacterial filtration efficiency was calculated (Eq. 1), where C and F are the numbers of bacterial colonies in the Petrifilm without and with the mask filter, respectively [31].

$$R = \frac{C - F}{C}(100\%) \quad (1)$$

The counting of the bacterial colonies was done employing a direct counting method. For each BFE test, peptone water with 90% peptone content was used as the growth medium. Then, the Petri film was incubated at 37 ˚C for 24 hours [32]. The viral filtration efficiency test recommended by the ASTM F2101 standard uses aerosol particle size similar to the bacterial filtration efficiency test described here. For this reason, only the BFE test was conducted. The bacterial filtration efficiency was compared to the medical mask standards of ASTM F2100-19 (Tab. VI).

2.9 Determination of pressure drop

According to EN 14863: 2019 Annex C [33], the breathability test was done by measuring the pressure across the filter material when passing air at a constant flow rate of 8 L/min. In this study, the pressure drop was theoretically determined by Davies' Equation of pressure drop (Eq. 2) for fibrous pads [34].

$$\Delta P = \frac{\mu Q L}{d_f A}\{64(1-\varepsilon)^{1.5}[1+56(1-\varepsilon)^3]\} \quad (2)$$

where μ (Pa·s) is the fluid viscosity, Q (m3/s) is the volumetric flow rate of the fluid, L (m) refers to the thickness of the fibrous pad, A (m2)- filter area, df (m) is the mean fiber diameter, ε is the porosity, and ΔP (Pa) is the pressure drop.

Moreover, the porosity will be solved using the following equation [35]:

$$\varepsilon = 1 - \frac{V_g}{V_a} \quad (3)$$

where ε is the porosity, Vg (weight/density) is the volume of the filter excluding void space, and Va is the total volume of the mask filter.

The theoretical pressure drop value was compared to the medical mask standards of ASTM F2100-19 (Tab. IX).

2.10 Characterization of polystyrene-chitosan mask filter

The electrospun waste expanded polystyrene-chitosan microfiber had to be characterized in terms of its fiber diameter and surface morphology. The microfiber was characterized using the scanning electron microscope (ThermoFisher – Quanta 250).

2.11 Hydrophilicity test

The mask filter produced was tested for its hydrophilicity to determine whether it is hydrophilic (water-loving) or hydrophobic (water-fearing) in nature. It was studied using contact angle measurement via drop shape analysis by dropping eight μL of water onto the filter surfaces, then measuring the angle at the liquid/solid interface[44] with imaging software (Image J).

3 RESULTS AND DISCUSSION

3.1 Microfiber Characterization

The EPS microfiber showed a smooth surface, whereas the chitosan-coated (CC) EPS has a rough surface (Fig. 2) due to the coating. The EPS microfiber and the CC EPS have an average diameter of 2.80±1.24 μm and 3.52±1.87 μm, respectively (Fig. 3). The extra layer made by the chitosan coating apparently increased the average diameter of the coated microfiber, mainly because of the high concentration (5 w/v %) of chitosan in the coating solution. Chitosan-coated polyacrylonitrile obtained the same results [41].
The incorporation of chitosan is confirmed by the absence of Nitrogen in the non-coated microfiber (Fig. 4a), and its presence at 20.89 % wt. in the coated microfiber (Fig. 4b). Nitrogen i

is present in the amino groups of chitosan and no other chemical reagents that may contain nitrogen are added to the polystyrene microfiber. Hence, chitosan was successfully coated into the microfibers.

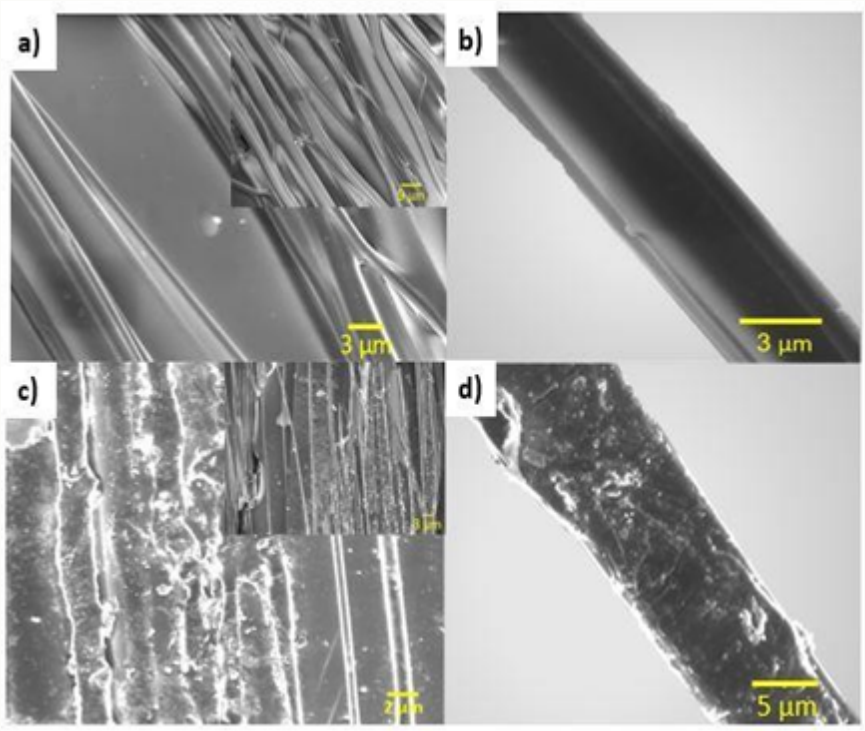

Fig. 2 SEM micrographs of (a,b) EPS microfiber and (c,d) CC (5 w/v %) EPS microfiber

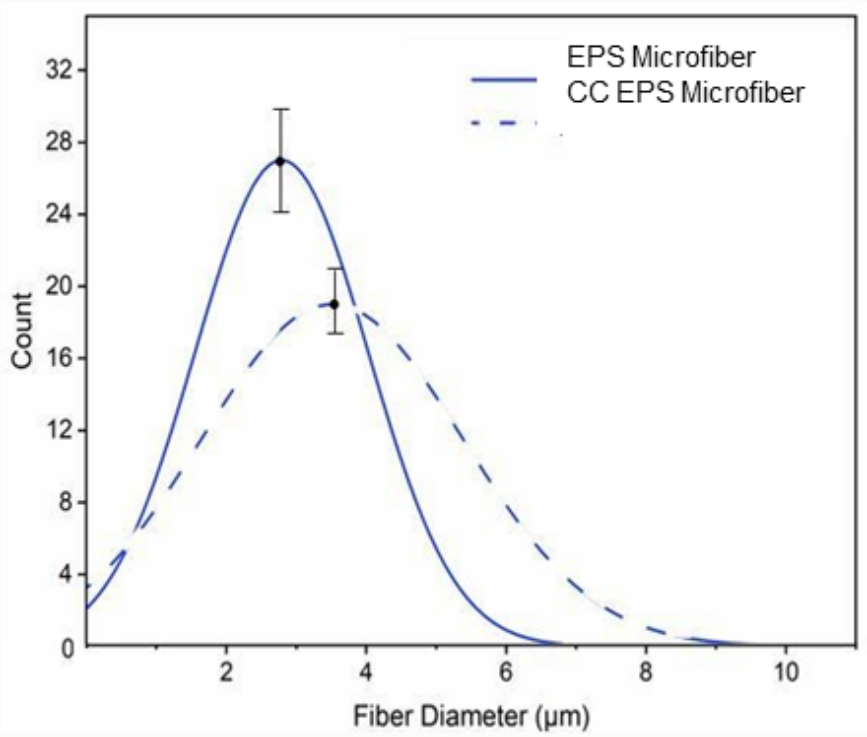

Fig. 3 Normal distribution curve of CC EPS and EPS microfiber diameter

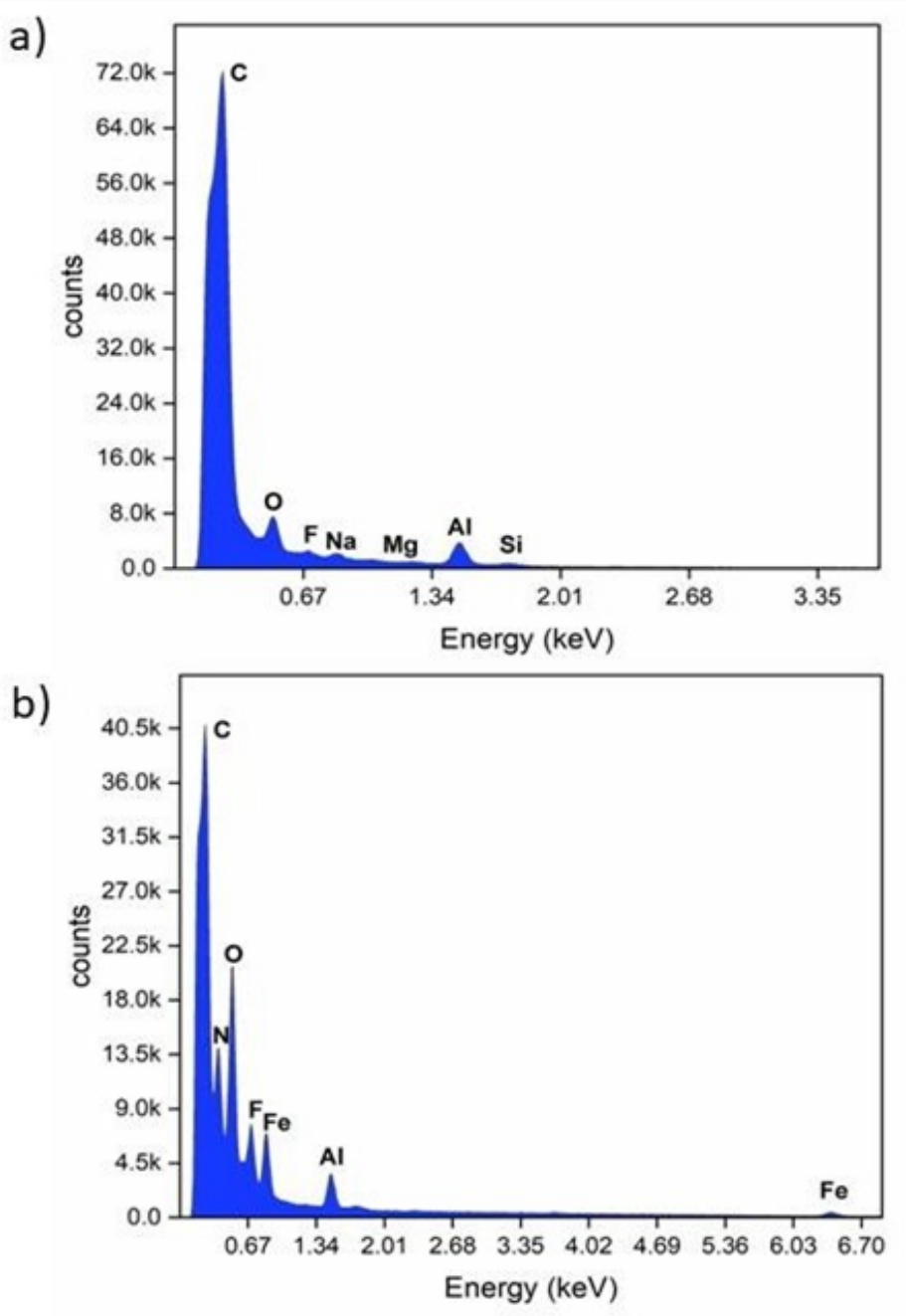

Fig. 4 Elemental analysisof (a)EPS microfiber and (b)CC (5% w/v) EPS microfiber

3.2 Effect of Annealing on Ultimate Tensile Strength

The tensile strength of the microfiber is an essential factor in its applicability as a mask filter. The produced polystyrene fiber through electrospinning is not highly durable. Thus, it was annealed and tested for its ultimate tensile strength. Results showed that the annealed polystyrene microfiber samples were approximately eight times stronger than those not. Annealing as post-treatment increases fiber tensile strength dominantly due to improved fiber-fiber fusion[43]. Another possible mechanism for the increase in tensile strength is the improved degree of crystallinity and fiber alignment
after thermal annealing [36].

3.3 Effect of Chitosan Coating and Concentration on Ultimate Tensile Strength

Due to the improved tensile strength, the annealed polystyrene microfiber was used for coating. The ultimate tensile strength of coated microfiber was compared to non-coated ones. The results (Fig. 5) show that the coating process significantly increased the ultimate tensile strength of the fiber. The same results were obtained when using nano-fibrillated chitosan as a nanofiller for a polycaprolactone scaffold [37] and a blend in a corn starch film [38], attributing the effect to the formation of intra-and intermolecular hydrogen bonding between chitosan and the other polymer in the composite. Increasing the chitosan concentration from 1 w/v % to 3 w/v% increased the ultimate tensile strength to 3.5 times the former value. Moreover, further increasing the concentration from 3 w/v % to 5 w/v % led to an increase of 1.25 times the former value. On the other hand, increasing the concentration from 1 w/v % to 3 w/v % increased the thickness by 14% and 20% from 3 w/v % to 5 w/v % (Fig. 6). While an increase in ultimate tensile strength is desirable, the increase in thickness may add to the filter stiffness which may limit its structural compatibility in the standard surgical masks and may also increase the differential pressure across the filter. Hence, the filter coated with 5 w/v % chitosan solution was used for the bacterial filtration efficiency test and differential pressure calculation compared to the non-coated filter.

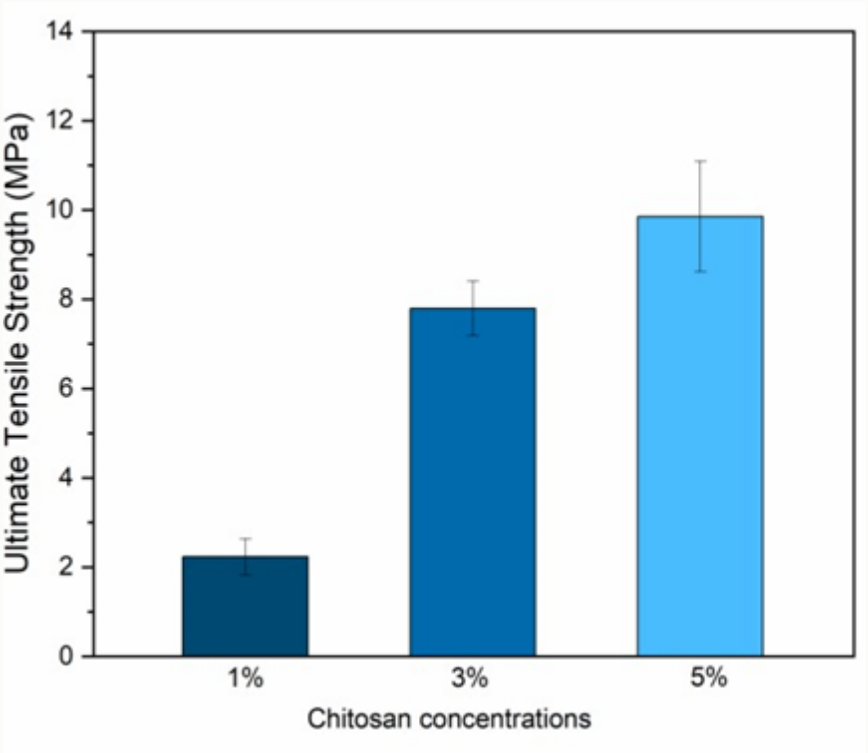

Fig. 5 Ultimate tensile strength of CC EPS microfiber at varying chitosan concentrations

3.4 Theoretical Pressure Drop Values

Breathability is another parameter to be evaluated in respiratory protection filter materials. This study theoretically solved the pressure drop for the coated and non-coated mask filter using the Davies equation (2). The average differential pressure of the coated microfiber (5 w/v %) is 5.40±0.17 mm H2O per cm2, while that of the non-coated is 5.58±1.54 mm H2O per cm2. The values are extremely close, suggesting that adding chitosan did not cause an additional pressure drop across the fiber mat.

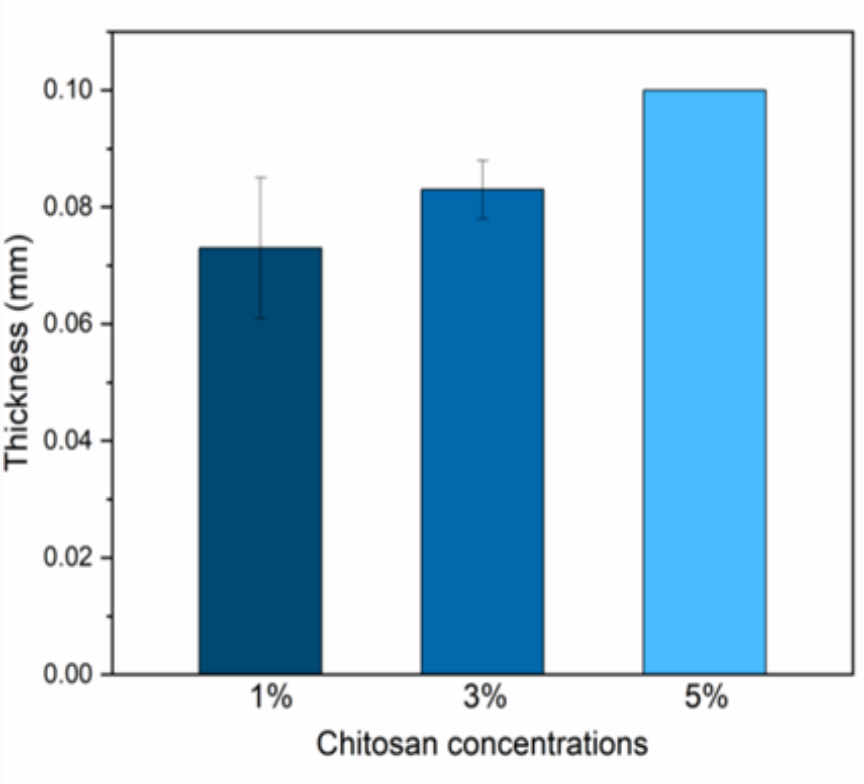

Fig. 6 EPS mask filter thickness at varying chitosan coating solution concentrations

3.5 Effect of Chitosan Coating on Bacterial Filtration Efficiency

A face mask should have at least 95% bacterial filtration efficiency to qualify as a medical or surgical mask and more than 98% as a high-protection mask [39]. The bacterial filtration efficiency of the microfiber mask filter was assessed using an alternative setup described in the methods. Based on the outcome of the test, the mask filter coated with chitosan solution (5 w/v %) showed 99.84±0.22% bacterial filtration efficiency, while the one without the coating showed 64.58±8.84%. The incorporation of chitosan in the microfiber (confirmed in the elemental analysis, Fig. 3) improved the bacterial filtration efficiency of the electrospun microfiber mask filter. Among the many proposed anti-bacterial mechanisms of chitosan [40], the most reasonable explanation for the effect of chitosan in bacterial filtration is the electrostatic interaction of the positive amino group of the polymer and the negatively charged cell wall of S. Aureus (and most bacteria due to the presence of peptidoglycan). It could be that the bacteria in the air adhered to the filter surface, thus minimizing the number passing through it. On the other hand, an increase in average fiber diameter is empirically found to cause an increase in average pore size, which may reduce filtration efficiency [42]. However, in this study, the increase in average fiber diameter of the coated microfiber did not affect the bacterial filtration efficiency as much as the coating effect did.

3.6 Hydrophilicity test

Another essential parameter to be considered in a standard face mask is the hydrophobic nature of its innermost and outermost layers, which prevents aerosols emitted by human activity (e.g., coughing and sneezing) from penetrating its central filter component [44]. While the application of the produced microfiber in this study is as a filter medium for standard medical grade face mask, the hydrophilicity of the microfiber filter was still tested due to empirical findings of increased particle filtration efficiency of hydrophilic fabrics under humid conditions [45].

The EPS microfiber has an average contact angle of 89.83±1.48° (Fig. 7). The contact angle of the water droplet after 4s (88.33°) is almost the same as that of the initial value (91.80°), which suggests hydrophobicity and is due to the hydrophobic nature of polystyrene itself. Meanwhile, adding chitosan (5 w/v%) in EPS microfiber decreased the water contact angle to an average of 51.61±4.47°. The contact angle decreased to 46.41° after 4s, suggesting that the hydrophobic EPS microfiber became hydrophilic upon adding chitosan. This change is also evident when chitosan was applied to an electrospun polysulfone (PSF) nanofibrous membrane; the pore size was decreased, and the highly hydrophobic PSF membrane was changed into a super hydrophilic one [46]. The mask filter's hydrophilic nature could help absorb the bacteria-carrying droplets, thus improving filtration efficiency.

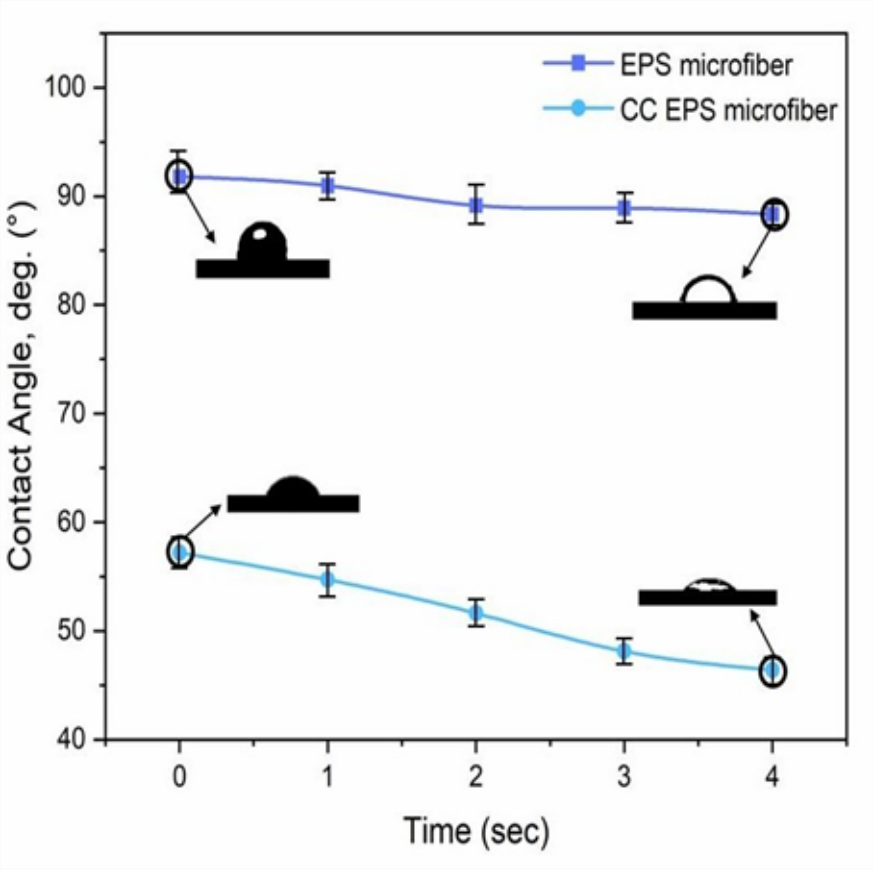

Fig. 7 Contact Angleof EPS microfiber and CC (5 w/v%) EPS microfiber through time

The researcher conceptualized the building's design, which served as the source of the material inputs for the life cycle assessment of the building. Walk-ups and the Row Housing Development can be found here. To conduct an objective comparison, the houses were created to have the same level of functionality. A summary of the building materials

3 CONCLUSIONS AND FUTURE WORKS

In this study, a mask filter microfiber was fabricated from waste-expanded polystyrene through the electrospinning process. It was coated with chitosan solution, and the ultimate tensile strength, bacterial filtration efficiency, and theoretical pressure drop were evaluated. The results determined that incorporating chitosan improved the filter's bacterial filtration efficiency from 64.58 % to 99.84 %. Moreover, there is no significant pressure drop difference between the coated (5.40±0.17 mm H2O/cm2) and non-coated mask filter (5.58±1.54 mm H2O/cm2), implying that the coating would not cause discomfort for the users. The CC EPS microfiber also exhibited a hydrophilic property with an average contact angle of 89.83±1.48°, which presents another factor for improving the bacterial filtration efficiency by adding

Chitosan coating. Most importantly, the mask filter qualifies for ASTM Level 3 for medical and surgical masks in terms of bacterial filtration efficiency (≥98%) and differential pressure (<6.0 mm H2O/cm2). On the other hand, it is recommended to conduct testing of particle filtration efficiency. Consideration of studies and methods on disinfecting and reusing the filter are encouraged to maximize the waste polystyrene upcycling. Furthermore, designs on how the mask filter could be combined with other materials to produce a complete three-layer surgical mask must be studied.

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