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Novel composite multilayer face masks for protection against airborne microorganisms

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This research focuses on the development of polyurethane (PU)/silica (SiO_2) nanofibrous membranes for the filtration of PM2.5 and bacteria, which has been further incorporated in three-layered face mask comprising cotton fabric, nanofibre membrane and polyester fabric. The surface morphology, PM2.5 filtration efficiency and bacterial filtration efficiency of PU/SiO_2 nanofibrous membranes have been investigated to find out applicability of nanofibrous membranes in the filtration application. PU/SiO_2 nanofibrous membranes (0.5%) are found to have a pressure drop of 126 Pa with filtration efficiency of 95.37% and bacterial filtration efficiency of 99.3% against *E. coli* bacteria strain. Two- and three-layered fabric structures are developed comprising PU/SiO_2 nanofibrous membranes and their contact angle, water vapor transmission rate and air permeability are studied to observe the breathability and waterproof behaviour of the fabric structure. Three- layered PU/SiO_2 nanofibrous membranes have contact angle, water vapor transmission rate and air permeability of 145±1°, 109 g/m^2/day and 8.56 $cm^3/s/cm^2$ respectively.

Keywords: Airborne microorganism, Bacterial filtration efficiency, Face mask, Filtration efficiency, Layered fabric structure, Nanofibrous membranes, Polyurethane, Silica

1 Introduction

In this ongoing situation of the coronavirus pandemic, one of the most effective protective measures is the active use of face masks which protects against the viruses and bacteria affecting the respiratory tract [1,2]. The face masks are being tremendously used to lower down the rate of transmission of cross-infection spreading through the release of respiratory droplets during cough, sneeze or breath of infected to healthy individuals [3]. Respiratory droplets on the basis of their size are classified as (i) size lesser than 5 µm known as aerosols and (ii) size greater than 5µm are known as droplets with short range transmission [4]. The droplets of larger size are known to settle down while those smaller in size remain suspended in air for longer durations, further resulting in spread of infection [1]. Thus, a face mask with comfortability and higher filtration efficiency for microorganisms and air-borne particles is the need of the current situation. The properties of the filters utilized, such as its chemical composition, thickness and the packing density, and external factors including airflow, humidity, temperature, influence the filtration efficiency of the masks [2,5–7].

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Electrospun nanofibrous mats have been developed as promising filter media for fine particles [8,9]. The coating of electrospun fibre having antiviral agent on the nonwoven layer of facemask material has the potential to capture and kill the microorganisms [10]. Metal oxide nanoparticles, such zinc oxide, silver oxide and cuprous oxide, have shown antimicrobial and antiviral properties against the tested microbes [11]. Silicon and silica oxide have been reported to give super hydrophobicity [12]. Silica is an attractive material for the coating of fabrics because of its low cost, chemical inertness, thermal stability, controllable pore structure and well-defined surface quality for modification [13–16]. The textiles modified using silica nanoparticle coating show excellent water-repellent property and self-cleaning behavior [17]. The nanosized silica acts as a reinforcing agent to improve the hardness, strength and thermal stability of the polymers [18–20]. The modification of PU leather coatings with nanosilica improves the breathability and comfort of PU leather [21]. Waterproof and breathable membranes have been developed using silica incorporated polyurethane nanofibres [22,23]. Fluorinated PU decorated with silica nanofibrous membranes with waterproof and breathable properties have been developed for use in protective garments, tissue engineering, catalyst carriers, water purification etc [24].

Yu *et al.* [17] prepared the superhydrophobic surfaces by the sol-gel method based on nanosilica and perfluorooctylated quaternary ammonium silane coupling agent, to be used for the preparation of superhydrophobic cotton fabric surfaces. An excellent superhydrophobicity (>170°), is generated by applying amino functionalized silica nanoparticles on epoxy functionalized cotton fabric [25]. Xue *et al.* [26] produced durable and robust nano-roughness on the fabric surface because of the strong bond created between the particles and the fibre during the fuctionalization of silica and cotton surface. Tightly woven microfilament fabrics are durable and lightweight due to their compact structure and small pores. They have high waterproofness and drapeability but lower breathability. Microfibres contribute for further increase in durability and strength of synthetic fibres. Nylon and polyester fibres are made up of microfibres. Production of microfibres is possible using melt, dry and wet spinning methods, and ultrafine fibres are produced by direct and conjugate spinning methods [27].

The face masks currently in use lack the property of comfort and filtration of airborne particles simultaneously. The transmission of the pathogens occurs via respiratory droplets which are not completely repelled by the hydrophobic layers of the majority of the face masks in use. To the best of our knowledge waterproof and breathable electrospun PU/silica nanofibrous membranes for capture of air-borne particles and bacterial filtration from the atmosphere have not yet been reported. So, in this study we developed electrospun PU/silica nanofibrous membranes for the efficient filtration of airborne particles and microorganisms. The addition of silica to the membranes imparts superhydrophobicity which is responsible for the repulsion of aerosol particles. The addition of superhydrophobic coating and antimicrobial properties using metal oxide nanoparticles could provide a higher protection to the wearer. Functional or antiviral protective masks shall be capable of trapping and killing the microorganisms including virus. The nanofibrous membranes developed have been analysed for the filtration of particulate matter, PM2.5 (to study the aerosol capture efficiency) and bacterial filtration efficiency. PM is generally a mixture of water droplets present in air, dust, smaller organic and inorganic particles generated through vehicular emissions and incomplete burning of fossil fuels. PM2.5 are the particles with size lesser than 2.5 µm [28]. Three layered fabrics with first layer of cotton which imparts comfort and third layer of polyester have been developed and its water vapor transmission rate and air permeability have been studied.

2 Materials and Methods

2.1 Materials

Polyurethane (PU) was purchased from the local market of Delhi. N, N-Dimethylformamide (DMF) was purchased from Merck. Silicon dioxide (silica) nanodispersion Type A having an average particle size of 20 nm (40 wt% in water, colloidal dispersion, 40 wt%) was procured from SRL, India. Cotton and polyester 2/2 plain weave fabrics of weight 111 g/m^2 and 92 g/m^2 respectively, used to develop three layered structure were obtained from local markets of Delhi.

2.2 Electrospinning of Nanofibrous Membranes

PU dope solution (12 % w/v) was prepared in DMF. PU pellets were dissolved in DMF by heating at 70 °C with constant stirring for 1 h. 0.5% and 1.0% silica nanoparticles (SiO_2) were added to the PU dope solution for preparation of 0.5% PU/SiO_2 and 1.0% PU/SiO_2 dope solutions.

The nanofibrous mats were developed using electrospinning machine (Royal Electro Spinner, India). PU nanofibrous mats were collected on a collector plate covered with a layer of cotton fabric on aluminium foil at a voltage of 15 kV, spinning distance of 15 cm and a flow rate of 0.6 mL/h. PU/SiO_2 nanofibrous mats were prepared similarly at a voltage of 15 kV, spinning distance of 15 cm and a flow rate of 0.6 mL/h.

2.3 Characterization

The surface morphology of the nanofibrous mats was analysed using field emission scanning electron microscope (SEM, Hitach S-3700N SEM, Germany) at a voltage of 15 kV. The samples were sputter coated with gold prior to SEM analysis. The diameter of the nanofibres was measured using ImageJ software by analysis of 30 fibres for each sample at different positions.

The contact angle of the nanofibrous membranes was measured using DSA 100 Goniometer from Kruss Inc. Germany. The measurements were carried out using distilled water as test liquid at 25 °C, 65% relative humidity and 6 µL drop volume. Three independent measurements were taken for each sample and average value was recorded.

The water vapor transmission rate (WVTR) was measured using the upright cup method [29]. Four tests of each sample have been carried out and average value was recorded. WVTR was calculated, using following equation:

$$WVTR = \frac{M}{At} \quad \ldots(1)$$

where WVTR is the water vapor transmission rate ($g/m^2/day^1$); *M (g)*, the difference in weight reduction in 24 h; *A*, the area of sample (m^2) in contact with air; and *t*, the duration of testing in hours.

The air permeability of the nanofibrous membranes was measured using WIRA air permeability tester (IS:11056-84 (RA 2006)). The water wicking test of the nanofibrous membranes have been performed to measure the movement of moisture from source and through the fabric. A beaker containing 50 mL of distilled water was taken and sample measuring 3×3 cm was retained on the water surface. The time of wicking was recorded.

The PU/SiO_2 nanofibrous membranes were tested as filters for their efficiency to capture PM2.5 from the atmosphere. The filtration efficiency of PU/SiO_2 nanofibrous membranes was determined using Environmental Particle Air Monitor (EPAM-5000, HAZ-DUST, USA). The test was performed using filter of 2.5 μm to capture PM2.5 at a flow rate of $4 Lmin^{-1}$ and a sample rate of one second for a duration of 6 h. The particle concentration of PM2.5 (mgm^{-3}) was calculated as the difference between the weight of the nanofibrous membrane initially and after the test. The filtration of the nanofibrous membranes used as filter was evaluated using the following equation:

$$\text{Filtration efficiency (\%)} = \frac{A-B}{B} * 100 \quad \ldots(2)$$

where A and B are the particle concentration (mg/m^3) of PU/SiO_2 nanofibres and PU nanofibres (control) respectively.

2.4 Bacterial Filtration Efficiency Test

The bacterial filtration efficiency (BFE) test has been performed in accordance with ASTM F 2020-01 with the use of *E. coli* bacterial strains as biological aerosols. During the test method, the bacterial culture was diluted using 1.5% of peptone water. The aerosols of a mean particle size of approximately 3 μm were formed by taking bacterial suspension in nebulizer. The samples were placed on the Anderson sampler. The test was carried out for both the control (without sample) and Test sample (PU/SiO_2 nanofibrous membrane). The aerosol droplets were impinged over the agar plates based on the size of each aerosol. The plates were further incubated at 37°C for a time period of 48 h. The bacterial colonies formed were counted and the BFE was calculated using the following equation:

$$\text{BFE (\%)} = \frac{X-Y}{X} * 100 \quad \ldots(3)$$

where *X* and *Y* are the number of bacterial colonies of the control and test sample respectively.

3 Results and Discussion

The SEM images of the nanofibrous membranes developed using the dope solutions are shown in Fig. 1. The PU nanofibrous membranes having a smooth surface of an average diameter of 221.6±12.5 are obtained. The addition of SiO_2 nanoparticles to PU increases the surface roughness of the nanofibrous membranes developed. As the concentration of the SiO_2 nanoparticles is increased from 0.5% to 1.0%, an increase in the average diameter of PU/SiO_2 nanofibrous membranes from 82.8 nm to 130.6 nm is observed (Fig 1). As the concentration of SiO_2 nanoparticles increases, polymeric beads having spindle like shape are formed as shown in Figure 1 (c). In this study, 0.5% PU/SiO_2 nanofibrous membranes are developed having good distribution of SiO_2 nanoparticles on the surface of the nanofibrous membranes while aggregation of the nanoparticles are observed at a concentration of 1.0%. The addition of SiO_2 nanoparticles has been reported to increase the surface roughness of the fibers [22,23].

The water contact angle of the PU/SiO_2 nanofibrous membranes decreases from 138±1° to 130±2° as the concentration of SiO_2 increases from 0.5% to 1.0%. Li *et al.* [23] developed PU/SiO_2 membranes with a contact angle of 131° in comparison to a contact angle of 125° for PU nanofibres. The increase in water contact angle is due to the increase in concentration of SiO_2 nanoparticles on the surface of fibres and a decrease in average diameter of the fibres [22,23]. An increase in SiO_2 nanoparticles could decrease the surface energy and also increase the surface geometry of the nanofibrous membranes, which further result in high water resistance [30]. The addition of SiO_2 nanoparticles to the PU nanofibrous membranes increases the hydrophobicity of PU/SiO_2 nanofibrous membranes [31].

3.1 Filtration Efficiency of PU/SiO_2 Nanofibrous Membranes

The filtration efficiency of the nanofibrous membranes was evaluated using environmental particle air monitor under environmental conditions.

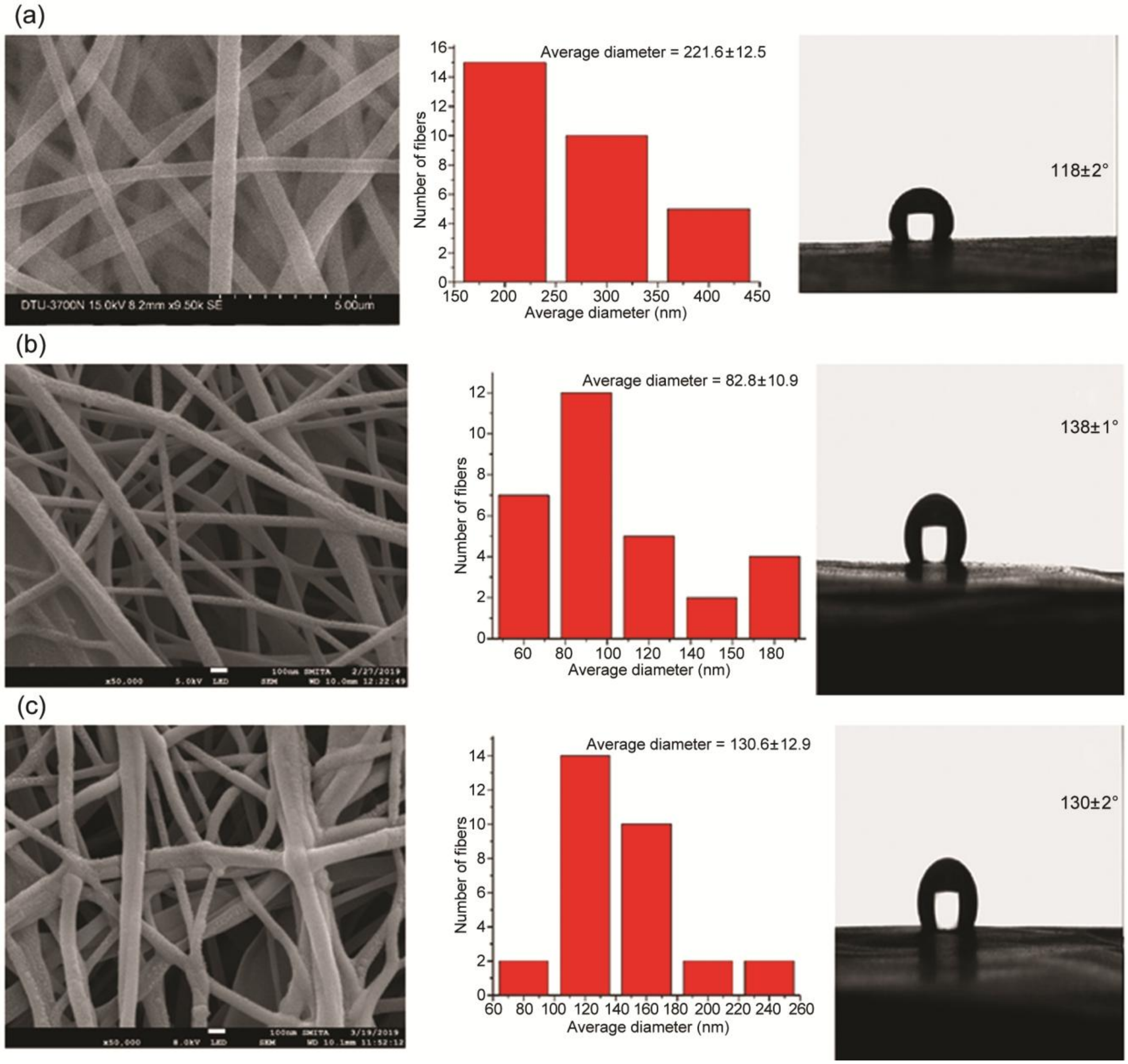

Fig. 1 — SEM images, average diameter and water contact angle of (a) PU, (b) 0.5% and (c) 1.0 % PU/SiO_2 nanofibrous membranes

The filtration efficiency of the nanofibrous membranes was analysed by capturing PM2.5 particles for 3.5 h in the HAZ DUST EPAM 5000 machine kept along the main entrance of Delhi Technological University, India. The concentration of the captured PM2.5 per second for the developed nanofibrous membranes is shown in Fig. 2. The concentration of PM2.5 captured by 0.5% PU/SiO_2 nanofibrous membranes is found to be 0.79 mg/m^3/s while only 0.40 mg/m^3/s of PM2.5 is captured by 1.0% PU/SiO_2 nanofibrous membranes (Fig 2). The particle concentration captured by 0.5% PU/SiO_2 nanofibrous membranes as evident from respirable suspended particulate matter (RSPM) test is 50.23 mg/m^3, while only 30.54 mg/m^3 and 43.65 mg/m^3 of PM2.5 are absorbed by PU and 1.0% PU/SiO_2 nanofibrous membranes. The filtration efficiency is decreased from 95.37% to 68.26% as the concentration of SiO_2 nanoparticles increases from 0.5% to 1.0% (Table 1). The addition of SiO_2 nanoparticles results in formation of nanofibrous membranes with an increased surface roughness and smaller fiber diameters having a lower fiber contact area which is responsible for enhancing the filtration of the nanofibrous membranes [32]. The electrospun nanofiber web possesses high surface area to volume ratio, and this has significantly contributed for enhancing filtration. The stacked structure of

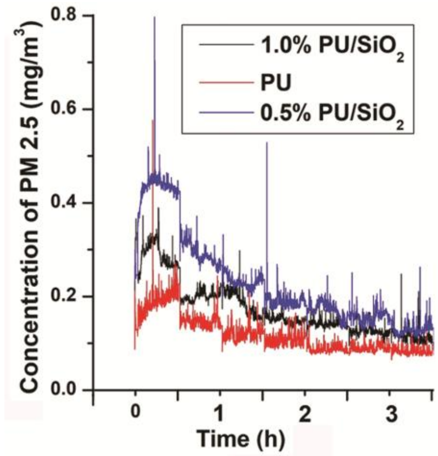

Fig. 2 — RSPM test of nanofibrous membranes

Table 1 — Filtration efficiency and BFE of nanofibrous membranes

Nanofibrous membranes	Particle concentration mg/m^3	Filtration efficiency %	Average number of colonies	BFE, %
Control	-	-	156	-
PU	25.71	-	122	21.8
0.5% PU/SiO_2	50.23	95.37	1	99.3
1.0% PU/SiO_2	43.65	68.26	25	83.9

polyacrylonitrile/silica nanoparticle has been reported to have a filtration efficiency of 99.98% in comparison to monolayer membranes [32]. PU/Si_3N_4 membranes show a filtration efficiency of 79.36% [33]. The robust silica nanofibrous membranes exhibited an excellent filtration efficiency of 99.99% towards NaCl aerosols [34].

Since 0.5% PU/ SiO_2 nanofibrous membranes are developed with lower fibre diameter and higher filtration efficiency, their surface morphology is studied after the RSPM test (Fig. 3). The PM2.5 particles are found to be deposited on the surface of the nanofibrous membranes and as the concentration of PM2.5 particles increased aggregation of particles occurred.

The pressure drop of the nanofibrous membranes decreases from 250 Pa in PU nanofibres to 126 Pa for 0.5% PU/SiO_2 nanofibres, whereas an increase in pressure drop is observed for 1.0% PU/SiO_2 nanofibrous membranes (182 Pa). The quality factor increased from $5.9*10^{-4}$ Pa^{-1} to $1.5*10^{-3}$ Pa^{-1} as the concentration of silica nanoparticles increases in PU/SiO_2 nanofibrous membranes. The roughness of the nanofibrous membranes is attributed to the enhanced filtration efficiency of the nanoparticles, which results in air flow penetration across the

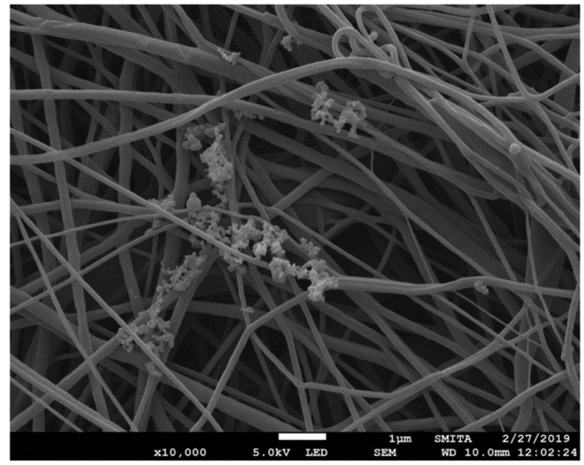

Fig. 3 — SEM images of 0.5% PU/SiO_2 nanofibrous membranes after RSPM test

membranes [32]. Wang *et al.* [35] reported high- efficiency filtration of sodium chloride aerosol particles (300-500 nm) using electrospun polyvinyl chloride/PU fibers with a filtration efficiency of 99.5% and a pressure drop of 144 Pa. The addition of fluorinated PU to polyacrylonitrile/PU membranes enhances its superhydrophobicity with a water contact angle of 154° and a filtration efficiency >99.9% for oil aerosol particles [36]. The addition of silica nanoparticles to PAN nanofibrous membranes is reported to increase the roughness of the surface. The multilevel structured PAN/silica nanoparticle nanofibrous membranes exhibits a filtration efficiency of 99.989% and a pressure drop of 117 Pa in comparison to monolayer structure [37].

3.2 Bacterial Filtration Efficiency (BFE)

The average number of bacterial colonies on the agar plate without test sample (control) and with PU/SiO_2 nanofibrous membranes is reported in Table 1. The average BFE values calculated using Eq (3), are found out to be 21.8%, 99.3% and 83.9% for PU nanofibres, 0.5% PU/SiO_2 and 1.0% PU/SiO_2 nanofibrous membranes respectively. Thus, the PU/SiO_2 nanofibrous membranes are effective against the bacterial strains as compared to PU nanofibres. The nanofibrous membranes have a smaller pore size in comparison to the size of the bacteria [37]. The general mechanisms for capturing of particles using a filter medium are inertial impaction, sieving, diffusion, interception and electrostatics. The mechanism involved in passage of too larger particles via fibre pores is sieving, while the electrostatic filters attract and bind charged particles of opposite charges while traversing the media [38].

In this case, electrostatic mechanism exists predominantly. The smaller diameter of the nanofibrous membranes provides enough surface for the attraction of bacterial particles, resulting in a higher BFE for 0.5% PU/SiO_2 nanofibrous membranes. In comparison to 1.0% PU/SiO_2 nanofibrous membranes, the average diameter of 0.5% PU/SiO_2 nanofibrous membranes is smaller, so these 0.5% PU/SiO_2 nanofibrous membranes have higher BFE.

3.3 Development of Three-layered Fabric Structure

From the above studies, it has been observed that 0.5% PU/SiO_2 nanofibrous membranes had the lowest fibre diameter of 82.8 nm and a filtration efficiency of 99.37%, so a three-layered fabric structure (Fig. 4) comprising 0.5% PU/SiO_2 nanofibrous membranes as filters for PM2.5 has been developed to study the comfort of the fabric as a filter. The cotton, polyester, cotton+PU, cotton+ 0.5% PU/SiO_2 and polyester+0.5% PU/SiO_2+cotton fabric structures are denoted as C, P, CU, CS and PSC respectively. The contact, angle, WVTR and air permeability are tabulated in Table 2.

The air permeability and WVTR are the two basic properties which determine the breathability of the fabric. It is observed that the plain fabrics of cotton and polyester have the highest air permeability of 41.26 cm/s and 99.27 cm/s while the electrospun fibres applied on the fabrics reduces the air permeability to 6.12 cm/s, 8.63 cm/s and 8.56 cm/s for CU, CS and PSC fabrics respectively (Table 2). The air permeability is related to the available open pores and is decreased for electrospun fabric as compared to the control [24]. WVTR also decreases as the electrospun nanofibrous membrane is inserted compared to the plain fabric (Table 2). The air permeability and the WVTR of the three-layered and two-layered fabric decreases in comparison to single-layered fabric due to an enhancement occurring in the adhesion amongst the fibrous structure [30]. The electrospun PU/hydrophobic silica gel are found to have higher WVTR and air permeability of 8.05 kg/m^2/day and 9.25 L/m^2/s respectively for waterproof and breathable applications [39]. Superhydrophobic and breathable electrospun SiO_2/PU porous membranes developed for oil-water separation has an air permeability and WVTR of 4.6 mm/s and 8.4 kg/m^2/day [31] and the fluorinated SiO_2/PU nanofibrous membranes has a moisture permeability of 10.4 kg/m^2/h [31] respectively.

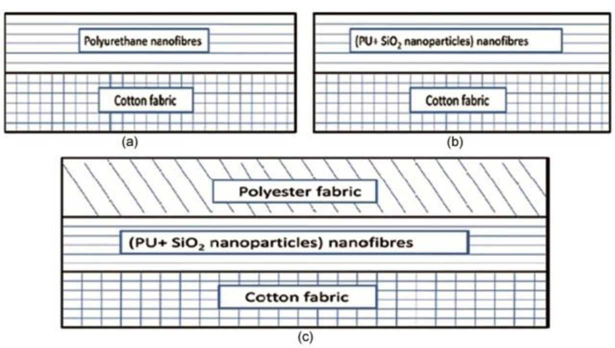

Fig. 4 — Development of layered structures

Table 2 — Properties of developed fabric structure

Fabric structure	Fabric	Contact angle, deg	WVTR, g/m^2/day	Air permeability cm^3/s/cm^2
Single-layered	C	80±1	115	41.26
	P	104±2	118	99.27
Two-layered	CU	134±3	114	6.12
	CS	148±2	115	8.63
Three-layered	PSC	145±1	109	8.56

4 Conclusion

Waterproof and breathable PU/SiO_2 nanofibrous membranes have been developed for effective filtration of aerosol particles especially PM2.5 and bacteria. The addition of SiO_2 nanoparticles to PU enhances the surface roughness of the nanofibrous membranes, which, in turn, makes it effective for filtration. The increase in concentration of SiO_2 nanoparticles from 0.5% to 1.0% results in decrease in contact angle of PU/SiO_2 nanofibrous membranes. The concentration of PM2.5 captured using 0.5% PU/SiO_2 nanofibrous membranes is 0.79 mg/m^3/s, which is comparatively higher in comparison to PU and 1.0% PU/SiO_2 nanofibrous membranes. These nanofibrous membranes are also found to be effective against *E. coli* bacterial strain with 99.3% BFE. The three-layered fabric developed using cotton, 0.5% PU/SiO_2 nanofibrous membranes and polyester fabrics, is found to have waterproofness and breathability property, suggesting it to be developed in the form of face masks for protection against aerosols.

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