TiO_2 nanoparticle-encapsulated polyacrylonitrile nanofibres as transparent air filters for indoor air quality

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Rapid industrialization and urbanization have increased air pollution, which poses a serious threat to human life. In megacities, the number of patients suffering from cardiovascular and respiratory diseases have increased rapidly. Face mask can be used for protection from outdoor air pollution, but indoor air quality can only be maintained through expensive, energy-inefficient air filtration devices. In the present study, nanofibrous polyacrylonitrile (PAN) filters encapsulated with titanium dioxide (TiO_2) nanoparticles were developed using the electrospinning technique. The filters are porous and nearly transparent and therefore do not resist sunlight and airflow. Field testing of the PAN : TiO_2 filters was done against the real aerosol particles and laboratory testing was done against ammonium sulphate particles. The field test results showed efficiency of about ~81% against total suspended aerosol particles (TSPM) and ~75% against laboratory generated particles. The TiO_2 nanoparticles have been reported to kill the influenza virus and may help minimize an individual's exposure to many harmful microbes due to their antimicrobial properties.

Keywords: Air pollution, antimicrobial properties, electrospinning technique, nanofibrous filters, nanoparticles.

IN the past decades, the air quality in urban as well as rural areas deteriorated significantly. The suspended aerosol particles are one of the most important constituents of air pollution that are emitted from various anthropogenic and natural sources. High concentration of aerosol particles affects visibility, climate, human health and the whole ecosystem[1–5]. The particles are classified as total suspended particulate matter (TSPM), particulate matter with an aerodynamic diameter less than 10 μm (PM_{10}), particulate matter with an aerodynamic diameter less than 5 μm (PM_5) and particulate matter with an aerodynamic diameter less than 2.5 μm ($PM_{2.5}$)[6]. The $PM_{2.5}$ particles are smaller in size and can travel deep into our lower respiratory system. Aerosol particles can be generated from various sources like vehicular emission, construction dust, industries, biomass burning and other sources[6–8]. The air quality of India, especially Delhi and its surroundings, has become a matter of concern as the PM concentration always exceeds the National Ambient Air Quality Standards (NAAQS) and the World Health Organization (WHO) standards. A recent United States Environmental Protection Agency (USEPA) report has shown that nearly 2.1 million deaths worldwide occurred due to high $PM_{2.5}$ concentration. Therefore, it is necessary to protect humans from pollution exposure. There is an urgent need to develop the most suitable and low-cost techniques for PM capture. Some efforts have already been made to protect individuals from outdoor pollution using masks, but indoor air quality is still a big challenge for the scientific community.

Indoor air quality is more severe than outdoor air quality as we spend ~90% of our time indoors. In 2019, indoor air pollution caused nearly 0.61 million deaths in India[9]. The indoor air quality is determined by both indoor and outdoor sources. Cooking is a major source of indoor air pollution. The use of solid fuel for cooking emits large amounts of atmospheric pollutants like carbon dioxide, carbon monoxide, sulphur dioxide and PM, which cause cardiovascular and pulmonary diseases, especially in women working in the kitchen. Outdoor air pollution also affects indoor air quality. In India, majority of the population relies on natural ventilation to maintain the temperature of the indoor spaces along with fans and water coolers. Neither these devices nor the windows have any air-filtration system. In modern commercial buildings/hospitals/public places, indoor air pollution is controlled using filtration in the ventilation system and centralized air-conditioning (AC). However, due to the COVID-19 pandemic, a centralized AC system is not recommended[10,11]. Air purifiers are expensive and consume a lot of power. Recent studies have shown that air purifiers generate low-level ozone, which harms human health. USEPA has recommended good ventilation and avoiding

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ACs during the COVID-19 pandemic[10,11]. The best suitable technique to control indoor air pollution is natural filtration which can be achieved by fixing a nanofibrous polymer filter over a steel mesh and mounting it on door and window panels. In the present study, we have developed a transparent air filter with high PM capture efficiency to control indoor air pollution. This technique has the potential to save the human population from degrading indoor air quality.

Nanofibrous filters have high PM removal efficiency, so they can trap most contaminants. Several polymer materials have already been used as filter material, e.g. polyacrylonitrile (PAN), poly(tetrafluoroethylene) (PTFE), polyamide (PA), nylon 6, polyvinylpyrrolidone (PVP), polyimide (PI), polyethylene (PE), polyvinyllidene fluoride (PVDF), polytherimide (PEI), polypropylene (PP) and poly(vinylalcohol) (PVA)[12–14]. Previous studies indicated that the PAN polymer is the most suitable material for fabricating electrospun nanofibres for particle filtration. Moreover, nanoparticles of titanium dioxide (TiO_2), zinc oxide (ZnO), silver oxide (AgO) and silicon dioxide (SiO_2) can be easily encapsulated in the filter. Among them, TiO_2 has gained much attention due to its photocatalytic and antimicrobial properties[14–16]. Therefore, the present study focused on developing transparent and window/door-mountable nanofibrous filters of PAN, which have a stronger affinity to PM pollutants. The existing affinity of PAN was further enhanced by adding TiO_2 nanoparticles.

Experimental section

Materials

TiO_2 nanoparticles (CAS: 18436-67-7) of 98% purity were purchased from RANKEM®, PAN polymer was purchased from Aldrich® (CAS: 24968-79-4) and DMF (CAS: 68-12-2) of 98% purity was purchased from Thomas Baker®. All the chemicals were used as such without further purification.

Sampling site and sampler

The filtration efficiency of the PAN : TiO_2 filter was tested using two Envirotech® APM800 samplers. The TSPM and PM_5 particles were collected using the samplers mentioned above at a flow rate of 2.5 LPM (litres per minute) for TSPM and 1.9 LPM for PM_5 respectively. Testing was carried out in ambient conditions on the terrace of the TEC Building, CSIR-National Physical Laboratory (NPL), New Delhi, India (28.70°N, 77.10°E). Figure 1 *a–c* shows the geographical location of the testing site, the sampler used for collecting PM_5 and TSPM, and the small testing chamber (for control observations). PM_5 particles were collected over 37 mm PTFE filters, while TSPM particles were collected over 25 mm quartz filters.

Preparation of PAN/TiO₂ nanofibres

The TiO_2 particles were weighed and dispersed in the *N*,*N*-dimethylformamide (DMF) and sonicated for 2 h for uniformly dispersion. PAN polymer (8 wt%) was added to the dispersion solution, sonicated for 1.5 h and stirred for 4–5 h to get a uniform solution. The final solution was filled in a disposable syringe and subjected to electrospinning. The schematic of the synthesis process of PAN-TiO_2 nanofibres is given in Supplementary Figure 1. The electrospinning parameters were optimized to get smooth nanofibres, i.e. DC voltage of 15 kV, flow rate of 0.2 ml/h and tip-to-collector distance of 20 cm. The nanofibres were collected on a steel mesh (5″ ∗ 5″) as a stationary collector. For each filter, the run time of the electrospinning instrument was ~10 min. A scanning electron microscope (SEM) image of the TiO_2 nanoparticles is shown in Supplementary Figure 2.

Laboratory testing

The filtration efficiency of the PAN : TiO_2 filters was measured using the set-up given in Supplementary Figure 3. It involved an atomizer which generated aerosol particles of ammonium sulphate of variable sizes ranging from 10 to 700 nm. The generated particles were then dried using a diffusion dryer to produce ammonium sulphate aerosol particles and passed through a differential mobility analyser (DMA). Then the air stream containing dry ammonium sulphate particles was passed through the PAN : TiO_2 filter and concentration of the uncaptured particles was measured using a condensation particles counter (CPC). The filtration efficiency was calculated using eq. (1).

$$\text{Filtration efficiency (\%)} = 100 - \frac{C_{\text{up}}}{C_{\text{down}}} \times 100, \tag{1}$$

where C_{down} is aerosol concentration in the downstream (particles/m^3) and C_{up} is aerosol concentration in the upstream (particles/m^3).

Field testing

The filtration efficiency of the PAN : TiO_2 filters was tested with the help of a rectangular aluminum box (testing chamber) fabricated in the workshop of CSIR-NPL. The filter was mounted on one side of the chamber, while the other five sides were closed. The inlet of one of the APM800 samplers was fixed inside the chamber while the inlet of other unit was kept in ambient condition. Figure 2 *a* shows the set-up used for filter testing. The sampler with an inlet inside the chamber sucks the air filtered through the PAN : TiO_2 filter. Both the samplers were operated at the same flow rate. The PM mass concentration (i.e. TSPM/PM_5) was determined for both controlled and ambient conditions simultaneously. The filters were weighed using a well-calibrated weighing

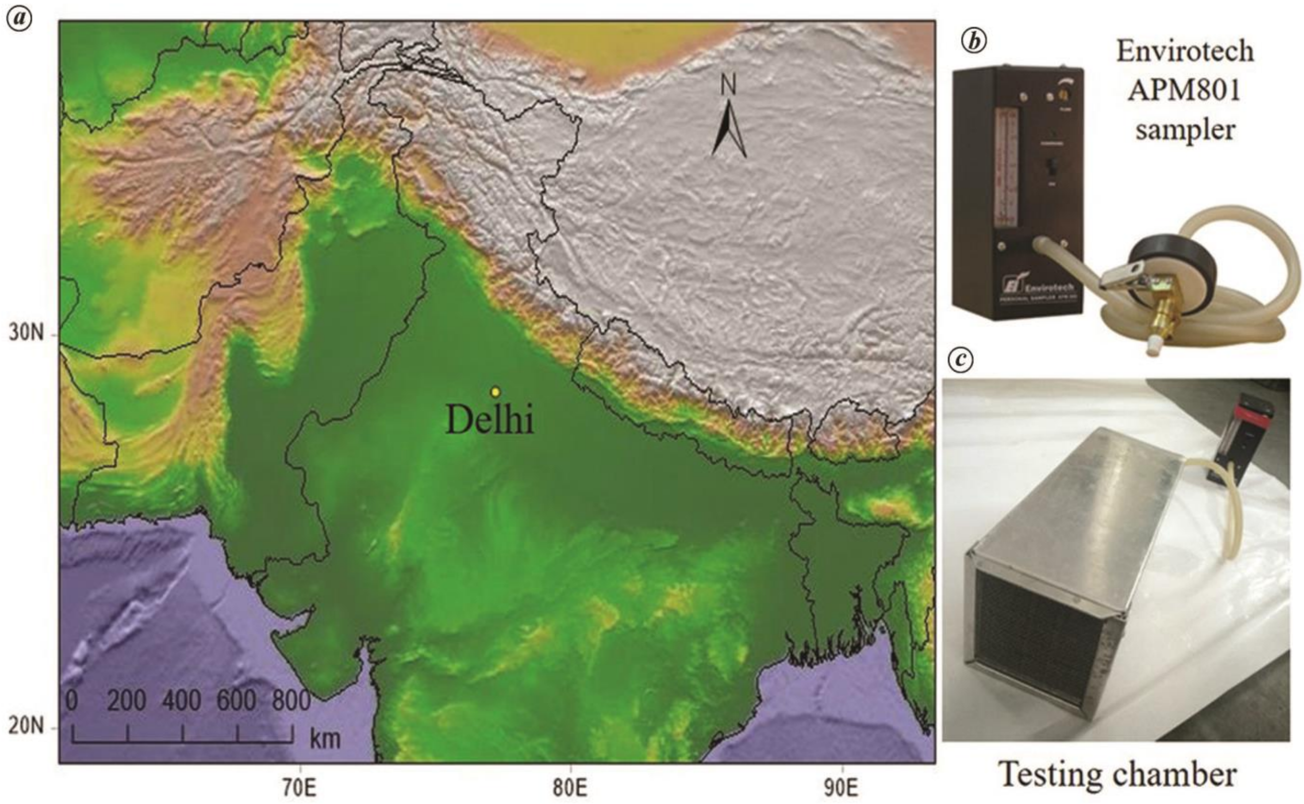

Figure 1. ***a***, Geographical location of testing site; ***b***, sampler used for collection of PM_5 and TSPM; ***c***, testing chamber.

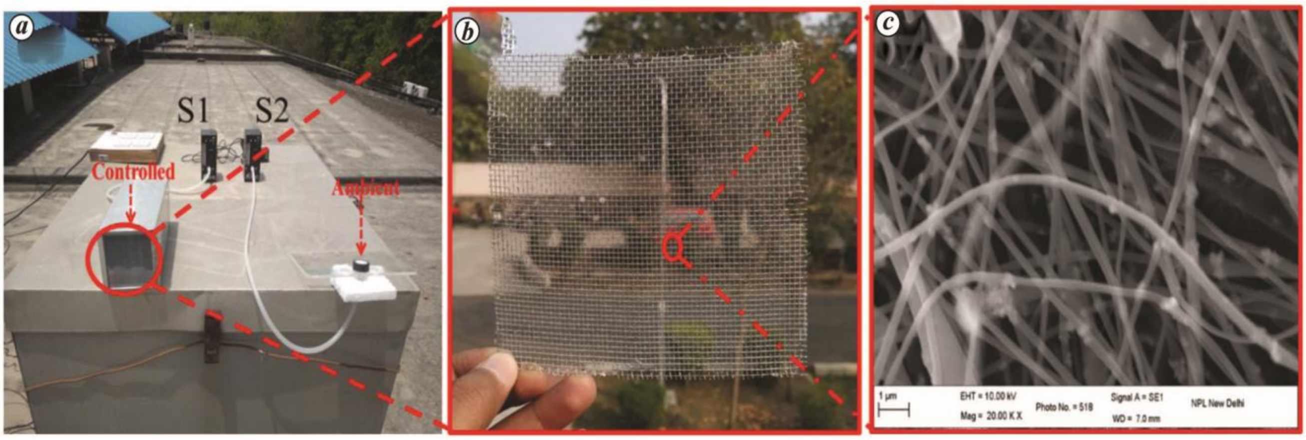

Figure 2. ***a***, Set-up used for the testing of PAN : TiO_2 filter; ***b***, nearly transparent filter; ***c***, SEM photomicrograph of the filter.

balance installed in the Weights Metrology Standards Laboratory of CSIR-NPL. Figure 2 *b* shows that the filter is nearly transparent. Figure 2 *c* shows the SEM micrograph of the filter where the PAN fibres and encapsulated TiO_2 nanoparticles are visible.

Characterization

The PAN : TiO_2 filters were mounted on conducting tin substrates with the help of a carbon tape for scanning electron microscope-energy dispersive X-ray spectrometer (SEM-EDS) analysis. The surface morphological analysis of the filters before and after testing was carried out using SEM (model: ZEISS EVO MA-10) and elemental composition analysis was done using EDS (Oxford Link ISIS 300, England, UK). The SEM images of the filters were recorded at different magnifications at 10 kV. The UV–visible analysis was done using a Perkin Elmer Lambda-35 spectrophotometer and the FTIR analysis was done using a Perkin Elmer Spectrum Two™ IR spectrophotometer.

Results and discussion

Development of transparent air filters

Electrospinning is widely used for the fabrication of nanofibres, as it has the advantage of developing uniform fibrous filters with controllable collection dimension[12,13,17,18]. In electrospinning, a high potential is applied across the tip of the syringe and the collector plate, which generates the electric field. The generated electric field charges the polymer solution and pushes it towards the oppositely charged collector plate in the form of nanofibres. The nanofibres get collected over that grounded collector. In the present study, the collector is a commercially used steel mesh and the polymer is PAN. The generated nanofibres are collected across holes in the steel mesh, making the transparent nanofibrous membrane suitable for air filtration. Using steel mesh as a supporting and adhering substrate increases the mechanical strength of the filter. Nanoparticles of TiO_2 are also added to the PAN polymer solution during fabrication. TiO_2 nanoparticles are non-toxic, inexpensive, chemically stable and generally recognized as safe. These particles are well known for their photocatalytic, antifungal, bactericidal and virucidal properties[19,20]. TiO_2 nanoparticles are also being used to control the viability of coronavirus in the ongoing pandemic. Prague is now treating all of its public transit vehicles with an antiviral coating of TiO_2 nanoparticles[21]. In a typical urban environment like Delhi, there is a high concentration of organic compounds ranging from 30% to 60% of the total $PM_{2.5}$ and PM_1 (refs 22–24). The photocatalytic activity of the TiO_2 nanoparticles can decompose the organic compounds present in the atmosphere during filtration and help in air filtration[25–27]. Earlier studies have also shown the effectiveness of TiO_2 nanoparticles in killing the influenza virus[28]. Apart from mounting these filters on doors/windows, they also have the potential to be used as an additional layer in the masks that protect individuals from various harmful microbes. However, detailed studies in this regard are necessary for the near future.

Structure and composition of nanofibrous filters

The structural and compositional properties of the filters were studied using SEM-EDS, UV–visible spectroscopy and Fourier Transform Infrared Spectroscopy (FTIR). The SEM images were analysed using the ImageJ software to measure the fibre diameter, which ranged from 100 to 450 nm with an average diameter of 221 nm (Figure 3 *a*). Figure 4 shows the SEM-EDS results of the PAN : TiO_2 filter. The fibres of the filter and TiO_2 nanoparticles are clearly visible in the SEM micrograph (Figure 4 *a* and *b*); the fibrous network structure of the filter allows the free flow of air. The EDS analysis confirmed the presence of TiO_2 nanoparticles in the filter (Figure 4 *c*). The cumulative effect of a fibrous structure and less thickness makes the filter nearly transparent (Figures 3 *b* and 2 *b*). The UV–visible analysis showed that the filter could transmit ~66% of the incoming light in the visible spectrum (Figure 3 *b*). The FTIR spectra showed that the TiO_2 nanoparticles were encapsulated in the filter (Figure 3 *c*). SEM micrographs of the filter recorded after the testing are shown in Figure 5, where the captured aerosol particles are clearly visible. The aerosol particles were physically trapped between the fibres of the filter or got adsorbed over the surface of the fibre. The PAN : TiO_2 filters can improve indoor air quality by effectively hindering intruding aerosol particles. The filters clean the air through physical (filtration) and chemical processes. The encapsulated TiO_2 nanoparticles are well known for their photocatalytic and antimicrobial properties[29–32]. The nanoparticles can destroy volatile organic compounds by producing reactive oxidants like hydroxyls and superoxides[27]. The testing for the antimicrobial activity of PAN : TiO_2 filter was not done in this study; nevertheless, several studies have shown the antimicrobial property of TiO_2 nanoparticles[19–21,28,33]. TiO_2 nanoparticles show antimicrobial properties because of their strong oxidizing power that generates free radicals like hydroxyl and superoxide radicals, which results in the inactivation of several microorganisms[19,20]. The PAN : TiO_2 membrane could be a boon to the poor, who cannot afford expensive air filtration devices, especially during the ongoing COVID-19 pandemic. The cost analysis of the PAN : TiO_2 filter is given in Supplementary Table 1.

Filter performance

Field testing: In the present study, the PAN : TiO_2 filters were tested with the help of an indigenously developed chamber, PM_5 and TSPM handheld samplers (Figure 1). The rectangular chamber was closed on five sides and kept open from one side that could be used as a window. The open side was utilized to fix the filter. The inlet of the aerosol sampler was kept inside the chamber and the main unit was kept outside. The inlet of the sampler and the main unit were attached with a sampling line. The sampler (main unit) sucks air through the inlet (using the fixed PTFE/quartz filter paper on which the particles are deposited) from the chamber so that the air enters inside the chamber after getting filtered through the PAN : TiO_2 filter. Efficiency of the filter was tested using two samplers, of which one sampler inlet was kept inside the chamber (S1) while the other sampler inlet was kept outside the chamber (S2). S1 was used for sucking the filtered air, whereas S2 was for the ambient air (Figure 2 *a*). The aforesaid testing was carried out for both PM_5 and TSPM samplings. The weighing of the filter papers was done before and after the collection of aerosol particles (PM_5 and TSPM). The gravimetric analysis gave the weight of the particles deposited on the PTFE (37 mm diameter)/GMF (25 mm diameter) filter papers. The testing was done using two instruments simultaneously, one in controlled condition (C) and the other in ambient condition (A). Table 1 shows the gravimetric analysis results. Here, *W*1 is the weight of the

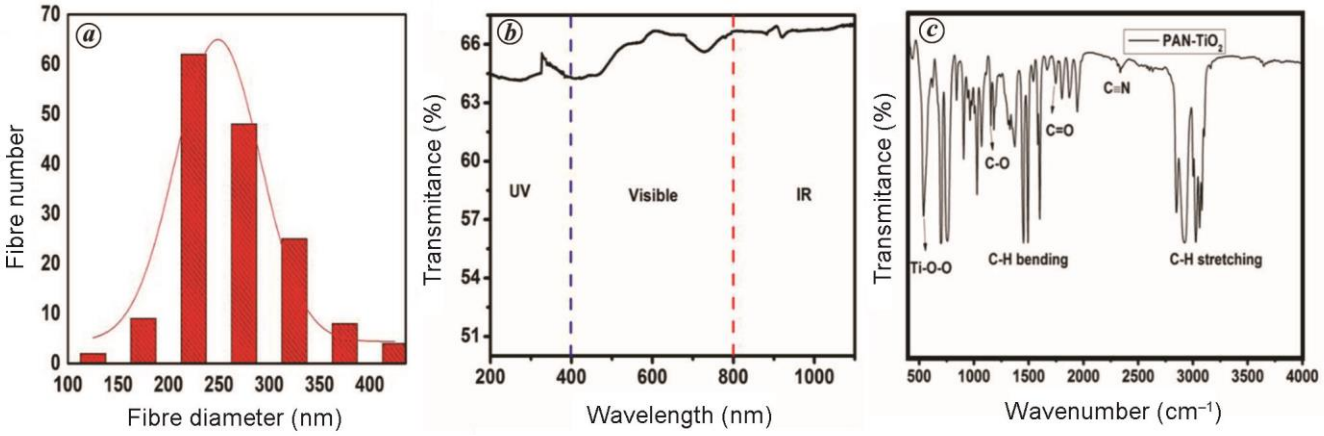

Figure 3. (***a***) Fibre diameter distribution; (***b***) UV–visible spectra and (***c***) FTIR spectra of polyacrylonitrile (PAN) fibres encapsulated with TiO_2 nanoparticles.

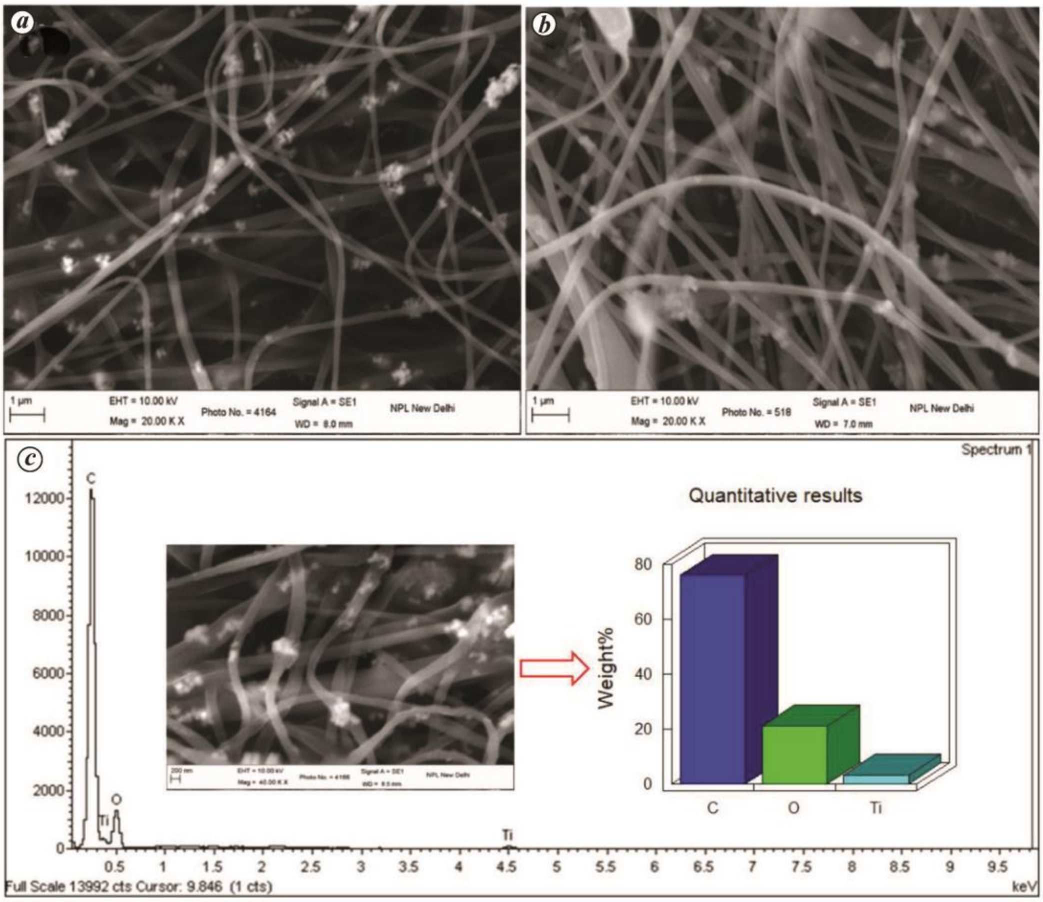

Figure 4. ***a***, ***b***, SEM images of the filter before testing. ***c***, Elemental composition of the filter.

filter before testing, $W2$ the weight of the filter after testing and $W3$ is the weight of the deposited particles, which is calculated as $W2 - W1$. Filtration efficiency of the PAN : TiO_2 filter was calculated according to eq. (2)

$$\text{Filtration efficiency} = \frac{W3\,(\text{A}) - W3\,(\text{C})}{W3\,(\text{A})} * 100. \qquad (2)$$

The gravimetric analysis showed that the filter could purify the air. In the controlled environment, developed filter was used to clean the air, whereas in the ambient environment no filter was used. Results showed that the PAN : TiO_2 filter was able to reduce the PM_5 concentration inside the box up to 47% and TSPM concentration up to 81%. The filtration efficiency can be improved by increasing the thickness of

Table 1. Gravimetric analysis of PM_5 and TSPM samples collected from controlled (C) and ambient (A) environments

Date	Sampling	Membrane composition	Controlled (C)/ ambient (A)	Filter number	*W*1 (μg)	*W*2 (μg)	*W*2 – *W*1 (μg)	Volume of air sucked (m^3)	Mass concentration (μg/m^3)	Filtration efficiency (%)
17 April 2018	PM_5	PAN + TiO_2	C	PTFE_9	71,790	72,010	220 ± 4.4	2.6	85 ± 1.7	45.00
			A	PTFE_10	71,380	71,780	400 ± 8	2.6	154 ± 3.0	
15 May 2018	PM_5	PAN + TiO_2	C	PTFE_11	71,900	72,150	250 ± 5	2.79	90 ± 1.79	44.44
			A	PTFE_20	71,300	71,750	450 ± 9	2.79	161 ± 3.22	
10 May 2018	TSPM	PAN + TiO_2	C	GMF_10	26,210	26,690	480 ± 936	3.3	145 ± 2.90	80.80
			A	GMF_13	26,660	29,160	2500 ± 50	3.3	758 ± 15.15	
3 May 2018	PM_5	PAN + TiO_2	C	PTFE_19	78,150	78,320	170 ± 3.4	2.5	68 ± 1.36	46.88
			A	PTFE_22	72,420	72,740	320 ± 6.4	2.5	128 ± 2.56	
4 May 2018	TSPM	PAN + TiO_2	C	GMF_04	27,330	27,800	470 ± 9.4	3.9	121 ± 2.41	80.17
			A	GMF_07	26,960	29,330	2370 ± 47.4	3.9	608 ± 12.15	

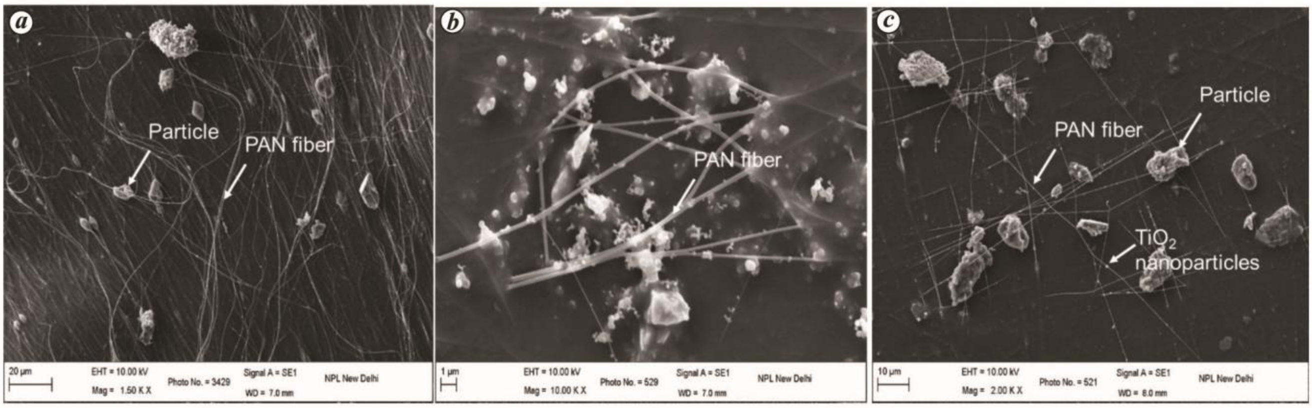

Figure 5. SEM photomicrographs of the filter (at different magnifications) after testing.

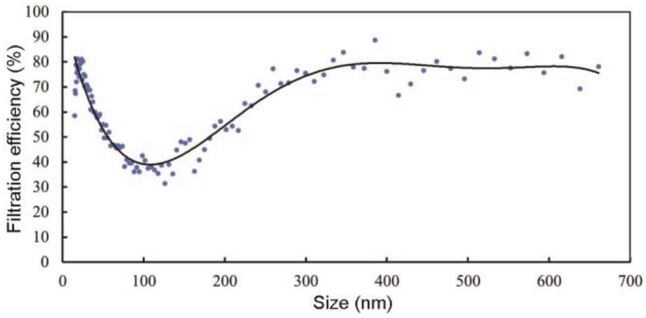

Figure 6. Filtration efficiency of PAN : TiO_2 nanofibrous filter as a function of particle diameter.

the filter, but this will affect the transparency[12]. A balance between PM removal efficiency and transparency can be maintained according to the end-user's requirements. These filters have a variety of applications – they can be used as window/door mountable filters or in masks, ACs, car ventilation, etc. The manufacturer can increase/decrease the thickness of the filter runtime of the electrospinning instrument according to the end-user's requirements.

Laboratory testing: The PAN : TiO_2 filters were also tested in the laboratory against ammonium sulphate particles (Supplementary Figure 3). The filters were tested for particles of variable sizes ranging from 10 to 700 nm. For the particles of 300 nm size, the filtration efficiency was 75% ± 3% (Figure 6). The filtration efficiency was measured for the 300 nm-sized particles according to the globally accepted approach[34,35]. Also, 300 nm is considered the most penetrating particle size, where the particle penetration is maximum and filtration efficiency is minimum. Large or smaller particles are trapped with even higher efficiency (USEPA).

Conclusion

The effectiveness of transport nanofibrous PAN : TiO_2 filters is demonstrated in against real and laboratory generated aerosol particles. The developed filters can inhibit PM from entering the indoor environment, maintain optical transparency, and natural ventilation when mounted on window/door. These filters will be a boon to the poor, who cannot afford expensive and energy-extensive air purifiers. The filters were tested in ambient conditions and found to reduce the PM_5 concentration inside the test chamber up to 47% and TSPM concentration up to 81%. The laboratory testing showed that the filters had a filtration efficiency of 75% ± 3%. The filtration efficiency can be increased by increasing the thickness of the filter according to the requirements. These filters do not need electricity and they work on natural ventilation. During the fabrication of the filters, nanoparticles of TiO_2 were encapsulated in the polymer that showed antimicrobial activity and did not allow microorganisms to enter the homes. These filters can also be used an additional layer of protection in masks to prevent individuals from harmful microbes. We plan to conduct focused research on the antimicrobial activity of the PAN : TiO_2 filters in the near future.

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