TiO$_2$ Nanoparticle-Encapsulated Polyacrylonitrile Nanofibers as Transparent Air Filters for Indoor Air Quality

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Abstract:

Rapid industrialization and urbanization have increased air pollution that poses serious threats to human life. In megacities, the number of patients suffering from cardiovascular and respiratory diseases had increased rapidly. Protection from outdoor air pollution can be attained through masks but, indoor air quality can only be maintained through expensive and energy inefficient air filtration devices. In the present study, nanofibrous polyacrylonitrile (PAN) filters encapsulated with TiO$_2$ nanoparticles were developed using the electrospinning technique. The filters were fabricated over the steel mesh so that it can be easily mounted over the window. The filters are porous and nearly transparent therefore don’t resist the sunlight and airflow. The field testing of the PAN:TiO$_2$ filters was done against the real aerosol particles and the lab testing was done against the ammonium sulfate particles. The field test results showed that the filters have high filtration efficiency for Total Suspended Particulate Matter (TSPM) upto 81% and the lab testing showed that the filters have efficiency ~75 ± 3%. The TiO$_2$ nanoparticles have already been reported to kill the influenza virus and may help in minimizing the exposure of an individual from many harmful microbes due to its antimicrobial properties.

Keywords: Air Filter; Window Mountable; Transparent; Nanoparticles; Indoor Air Quality, Antimicrobial.
1. Introduction

In the past decades, the air quality in urban as well as rural areas has been 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. The particles are classified as TSPM (Total Suspended Particulate Matter), PM$_{10}$ (particulate matter with aerodynamic diameter less than 10µm), PM$_5$ (particulate matter with aerodynamic diameter less than 5µm) and PM$_{2.5}$ (particulate matter with aerodynamic diameter less than 2.5µm). 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. The air quality of India and specially Delhi and its surrounding has become the matter of concern as the PM concentration always exceeds the National Ambient Air Quality Standards (NAAQS) and world health organization (WHO) standards. Recent report by United States Environmental Protection Agency (USEPA) has shown that nearly 2.1 million deaths in world occurred due to high PM$_{2.5}$ concentration. Therefore, it is necessary to protect the human from pollution exposure. There is an urgent need to develop most suitable and low-cost techniques for PM capture. Some of the efforts have already been made to protect the individual from outdoor pollution using mask. But the use of mask by individual is often bulky and resists air flow.

Indoor air quality is more sever than the outdoor air quality as we spend ~90% of our time indoor. In 2019, indoor air pollution causes nearly 0.61 million deaths in India. The indoor air quality is determined by both indoor and outdoor source. Cooking is a major source of indoor air pollution. Use of solid fuel for cooking purpose emits large amount of atmospheric pollutants like
carbon dioxide, carbon monoxide, sulfur dioxide and Particulate Matter (PM) which causes severe cardiovascular and pulmonary diseases in the inhabitants especially in the women working in the kitchen. Outdoor air pollution also affects the indoor air quality. In India, majority of the population rely on the natural ventilation to maintain the temperature of their indoor spaces along with the use of fans and water coolers. Neither these devices nor the windows have any kind of air filtration system. In the modern commercial buildings/hospitals/public places, the indoor air pollution is controlled using filtration in ventilation system and centralized air conditioning but in the current COVID-19 pandemic, the centralized AC system is not recommended. The air purifiers are very expensive and consumed lot of electricity. Recent studies have shown that the air purifiers generate low level ozone which is harmful to human health. USEPA has recommended to ensure good ventilation and recommended not to use AC during the ongoing COVID-19 pandemic. The best suitable technique to control indoor air pollution is natural filtration that can be achieved by fixing the nanofibrous polymer filter over steel mesh of door and window panels. In the present study, efforts have been made to develop a transparent air filter with high PM capture efficiency to control the indoor air pollution. This technique has strong potential to save the population of the whole world from degrading indoor air quality.

Recently nanofibrous filters are considered as a most effective against the air pollution. Nanofibrous filters have high PM removal efficiency so they can trap most of the contaminants. Several polymer materials have already been tasted as a filter material e.g. polyacrylonitrile (PAN), poly(tetrafluoroethylene) (PTFE), polyamide (PA), nylon 6, polyvinylpyrrolidone (PVP), polyimide (PI), polyethylene (PE), polyvinylidene fluoride (PVDF), polyetherimide (PEI), polypropylene (PP), poly(vinylalcohol) (PVA), etc. Earlier reported studies indicated that the PAN polymer is the most suitable material for the fabrication of electrospun nanofibers for particle
filtration. Moreover, the nanoparticles of TiO$_2$, ZnO, AgO, SiO$_2$ can be easily encapsulated in the filter. Among the aforesaid nanoparticles, TiO$_2$ has gained much attention due to its photocatalytic and antimicrobial property $^{14-16}$. Therefore, the present study is focused on the development of transparent and window/door mountable nanofibrous filters of polyacrylonitrile (PAN) which have stronger affinity to PM pollutants. The existing affinity of PAN was further enhanced by adding nanoparticles of TiO$_2$.

2. Experimental section:

2.1. Material:

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.

2.2. Sampling Site & Sampler:

The filtration efficiency of the PAN:TiO$_2$ filter was tested using two Envirotech® APM800 samplers. The TSPM and PM$_5$ particles collection was carried out using the said samplers at flow rate of 2.5 LPM (Liter Per Minute) for TSPM and 1.9 LPM for PM$_5$, respectively. Testing was carried out in the ambient conditions over the terrace of TEC building of CSIR-National Physical Laboratory (NPL), New Delhi, India (28.70° N; 77.10° E). Figure 1 shows the geographical location of testing site, sampler used for collection of PM$_5$ and TSPM and small testing chamber (for control observation). PM$_5$ particles were collected over 37 mm PTFE filters while TSPM particles were collected over 25 mm quartz filters.

2.3. Preparation of PAN/TiO$_2$ nanofibers:
The TiO$_2$ particles were weighed and dispersed in the N,N-dimethylformamide (DMF) and sonicated for 2h so that the particles get uniformly dispersed. Polyacrylonitrile (PAN) polymer (8 wt %) was added in the above prepared dispersion solution and sonicated for 1.5h. The solution was further stirred for 4-5 h to get a uniform solution. The final solution was filled in the disposable syringe and subjected to electrospinning. The schematic of the synthesis process of PAN-TiO$_2$ nanofibers is shown in supplementary information figure S1. The electrospinning parameters were optimized to get smooth nanofibers i.e. DC voltage 15kV, flow rate 0.2ml/h and tip to collector distance 20 cm. The nanofibers were collected on the steel mesh (5˝ * 5˝) as a stationary collector. For each filter the run time of the electrospinning instrument was ~10 mins. The SEM image of the TiO$_2$ nanoparticles is given in supplementary file (Figure S2).

2.4. Lab testing:

The filtration efficiency of the PAM:TiO$_2$ filters was measured using the setup shown in figure S3 of supplementary information. The setup involved an atomizer which generated aerosol particles of ammonium sulfate of variable size ranging from 10-700 nm. The generated particles then dried using a diffusion dryer to produce ammonium sulfate aerosol particles and passed through a Differential Mobility Analyzer (DMA). Then the air stream containing dry ammonium sulfate particles was passed through the PAN:TiO$_2$ filter and concentration of the uncaptured particles was measured using Condensation Particles Counter (CPC). The filtration efficiency was calculated using the eq 1.

\[
\text{Filtration efficiency (\%)} = 1 - \frac{C_{\text{down}}}{C_{\text{up}}} \\
\]

Here, $C_{\text{down}}$ is aerosol concentration in downstream and $C_{\text{up}}$ is aerosol concentration in upstream.
2.5. Field testing:

The filtration efficiency of the PAN:TiO$_2$ filters was tested with the help of a rectangular aluminum box fabricated in the workshop of CSIR-NPL named as testing chamber. The filter was mounted on one side of the chamber while 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. The setup used for the filter testing is shown in Figure 2A. The sampler with 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 concentrations (i.e., TSPM/PM$_{5}$) was calculated for both controlled and ambient conditions, simultaneously. Weighing of the filters was carried out using well calibrated weighing balance installed in weights metrology standards laboratory of CSIR-NPL. Figure 2B shows that the filter is nearly transparent and Figure 2C shows the SEM micrograph of the filter where the PAN fibers and encapsulated TiO$_2$ nanoparticles are visible.

2.6. Characterization:

The PAN:TiO$_2$ filters were mounted on the conducting tin substrates with the help of the carbon tape for 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 the elemental composition analysis was done using EDS (Oxford Link ISIS 300, England). The SEM images of the filters were recorded at different magnification at 10 KV. The UV-Visible analysis was done using Perkin Elmer Lambda-35 spectrophotometer and FTIR analysis was done using Perkin Elmer Spectrum Two™ IR spectrophotometer.

3. Result and Discussions:
3.1. Development of transparent air filters:

Electrospinning an ideal technique for development of transparent air filters it has an advantage of making uniform fibrous filters with controllable collection dimension\textsuperscript{12,13,17,18}. In Electrospinning, high potential is applied across the tip of the syringe and the collector plate, which generated the electric field. The generated electric field charges polymer solution and pushes it toward the collector plate in the form of nanofiber where it gets collected over the grounded collector. In present study, the collector is a commercially used steel mesh and the polymer is PAN. The generated nanofibers are collected across the holes in the steel mesh which make a transparent nanofibrous membrane suitable for air filtration. The use of steel mesh as a supporting and adhering substrate increases the mechanical strength of the filter. Nanoparticles of titanium oxide (TiO\textsubscript{2}) were also added to the PAN polymer solution during fabrication. TiO\textsubscript{2} nanoparticles are non-toxic, inexpensive, chemically stable, and generally recognized as safe substances. These particles are well known for their photocatalytic, anti-fungal, bactericidal and virucidal properties\textsuperscript{19,20}. TiO\textsubscript{2} nanoparticles are also being used to control the viability of corona virus in the ongoing pandemic. Prague is now treating all of its public transit vehicles with an antiviral coating of TiO\textsubscript{2} nanoparticles\textsuperscript{21}. In a typical urban environment like Delhi, there is a high concentration of organic compounds ranging from 30-60\% of the total PM\textsubscript{2.5} and PM\textsubscript{1}.\textsuperscript{22-24} The photocatalytic activity of the TiO\textsubscript{2} nanoparticles can decompose the organic compounds present in the atmosphere during filtration and helps in air filtration\textsuperscript{25-27}. Earlier studies have also shown the effectiveness of the TiO\textsubscript{2} nanoparticles in destroying the influenza virus\textsuperscript{28}. Apart from mounting these filters on doors/windows, these filters also have the potential to be used as an additional layer in the masks that can protect the individual from various harmful microbes. However, the detail studies in this regard are necessary in near future.
3.2. Structure and composition of nanofibrous filters:

To find out the structural and compositional properties filter were analyzed using Scanning Electron Microscope coupled with Energy Dispersive X-ray Spectrometer (SEM-EDS), UV-visible spectroscopy and Fourier Transform Infrared Spectroscopy (FTIR). The SEM images were analyzed using the ImageJ software to measure the fiber diameter. The diameter of the fiber was found to be in the range of 100-450 nm with average dimeter of 221 nm (Figure 3A). SEM-EDS results of the PAN:TiO$_2$ filters are shown in Figure 4. The fibers of the filter and TiO$_2$ nanoparticles are clearly visible in 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 4C). The cumulative effect of fibrous structure and less thickness makes the filter nearly transparent (Figure 3B and 2B). The UV-visible analysis showed that the filter is able to transmit ~ 66% of the incoming light in visible spectrum (Figure 3B). The FTIR spectra shows that the TiO$_2$ nanoparticles are encapsulated in the filter (Figure 3C). SEM micrographs of the filters recorded after the testing are shown in Figure 5, where the captures aerosol particles are clearly visible. The aerosol particles are physically trapped in between the fibers of the filter or get adsorbed over the surface of the fiber. The PAN:TiO$_2$ filters can improve the indoor air quality by effectively hindering the intruding aerosol particles. The filters clean the air by physical (filtration) and chemical processes. The encapsulated TiO$_2$ nanoparticles are well known for their photocatalytic and antimicrobial properties. The nanoparticles can destroy the volatile organic compounds by producing the reactive oxidants like hydroxyls and superoxides. The testing for antimicrobial activity of PAN:TiO$_2$ filter is not done in this study but there are many studies which shows the antimicrobial property of TiO$_2$ nanoparticles. TiO$_2$ nanoparticles show antimicrobial properties because of its strong oxidizing power that generates free radicals like
hydroxyl and superoxide radicals, which results in inactivation of several microorganisms\textsuperscript{19,20}. The PAN:TiO\textsubscript{2} membrane could be a boon to the poor people of the society who cannot afford the expensive air filtration devices especially during the ongoing pandemic. The cost analysis of the PAN:TiO\textsubscript{2} filter is given in supplementary file (Table. S1).

3.3. Filter Performance:

3.3.1. Field Testing:

In the present study, the PAN:TiO\textsubscript{2} filters were tested with the help of indigenously developed chamber, PMs, and TSPM hand held samplers (Figure 1). The rectangular chamber was closed from five sides and kept open from one side that can be used as a window. The open side was used 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 main unit were attached to each other using a rubber tube. The sampler (main unit) sucks the air through inlet (through the PTFE/quartz filter paper fixed in inlet on which particles are deposited) from the chamber so that the air enters inside the chamber after getting filtered through PAN:TiO\textsubscript{2} filter. Efficiency of the PAN:TiO\textsubscript{2} filters was tested using two samplers out of which one sampler inlet was kept inside the chamber (S1) while another sampler inlet was kept outside the chamber (S2). The S1 was sucking the filtered air whereas S2 was sucking the ambient air (Figure 2A). The aforesaid testing was carried out for both PMs and TSPM samplings. The weighing of the filter papers was done before and after the collection of aerosol particles (PMs and TSPM). The gravimetric analysis gives 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 (C) condition and other in the ambient conditions (A). The gravimetric analysis results are given in Table 1. Here, W\textsubscript{1} is the weight of the filter before testing, W\textsubscript{2} is the weight of the filter after testing, and W\textsubscript{3} is
the weight of the deposited particles which is calculated as $W_2 - W_1$. Filtration efficiency of the PAN:TiO$_2$ filters was calculated according to Eq. 2:

\[
\text{Filtration efficiency} = \frac{W_3 (A) - W_3 (C)}{W_3 (A)} \times 100 \tag{2}
\]

The gravimetric analysis results showed that the filter is capable of purifying the air. In the controlled environment, PAN:TiO$_2$ filter was used to filter the air whereas in ambient environment no filter was used. Results showed that the PAN:TiO$_2$ filter is able to reduce the PM$_{5}$ concentration inside the box up to 47% and TSPM concentration up to 81%. However, the filtration efficiency of the filter can be improved by increasing the thickness of the filter but that will affect the transparency$^{12}$. The balance between PM removal efficiency and transparency can be maintained as per the requirement of the end user. These filters have variety of applications, they can be used as a window/door mountable filters, or they can be used in masks, ACs, car ventilation etc. The manufacturer can increase/decrease the thickness of the filter by increasing/decreasing the run time of the electrospun as per the end user requirement.

3.3.2. Lab Testing:

The PAN:TiO$_2$ filters were also tested in the lab against the ammonium sulfate particles with the help of the setup shown in Figure S3 of supplementary information. The filters were tested for the particles of variable size ranging from 10 - 700 nm. For the particles of 300 nm size the filtration efficiency of the PAN:TiO$_2$ filter was 75 ± 3% (Figure 6). The filtration efficiency was measured for the 300 nm sized particles as per the globally accepted approach. 300 nm is considered as Most Penetrating Particle Size (MPPS) where the particle penetration is maximum
and filtration efficiency is minimum. Particles that are larger or smaller are trapped with even higher efficiency (USEPA).

4. Conclusion:

The PAN:TiO$_2$ filters will be very useful to curb intruding air pollution that will increase the indoor air quality. The filter can be easily fitted over window/door and it is porous and transparent that allows the air and sunlight inside the house. The filter will be a boon for the poor section of the society which cannot afford expensive and energy extensive air purifiers. The filters were tested in the ambient conditions and found to reduce the PM$_{5}$ concentration inside the test chamber up to 47% and TSPM concentration up to 81%. The lab testing showed that the filters have filtration efficiency of 75 ± 3%. The filtration efficiency of these filters can be increased by increasing the thickness of the filter. One has to determine the filter thickness as per their requirement/purpose. These filters do not require any electricity and they work on natural ventilation. During fabrication of filter, nanoparticles of TiO$_2$ were encapsulated in the polymer that can show antimicrobial activity and does not allow microorganisms to enter in the home. These filters can also be used an addition layer of protection in the masks to prevent an individual from harmful microbes. However, we plan to execute focused research on the antimicrobial activity of the PAN:TiO$_2$ filter in near future.

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References:


21. DPP. DPP to disinfect its entire fleet of public transport vehicles using nanotechnology-based polymers. This disinfecting coating is projected to last for up to two years. (2020).


Table: Gravimetric analysis of PM$_5$ and TSPM samples collected from controlled (C) and ambient (A) environment.

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<th>Membrane Composition</th>
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<th>Filter Number</th>
<th>W1 (µg)</th>
<th>W2 (µg)</th>
<th>W2-W1 (µg)</th>
<th>Volume of air sucked (m$^3$)</th>
<th>Mass concentration (µg/m$^3$)</th>
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<td>Filtration efficiency of PAN:TiO\textsubscript{2} nanofibrous filter as a function of particle diameter.</td>
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**Figures:**

**Figure 1:**

![Figure 1](image1.png)

(A) Map of India with location marked. (B) Envirotech APM800 Sampler. (C) Indigenously Developed Chamber.

**Figure 2:**

![Figure 2](image2.png)

(A) Setup with labeled areas S1 and S2. (B) Close-up of mesh material. (C) Microscopic view showing details.
Figure 5:

(A) Particle PAN fiber

(B) PAN fiber

(C) PAN fiber TiO₂ nanoparticles