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PVA-graphene-hydroxyapatite electrospun fibres as air-filters

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Abstract

In this study, the feasibility of specially designed multifunctional electrospun fibres to absorb particulate matter 2.5 (PM2.5) is studied. Here, Poly(vinyl)alcohol (PVA) is used as the polymer for electrospinning, along with colloidal graphene (G) containing in situ synthesized hydroxyapatite (HA) nanoparticles. This fibre is in the nanometre range, has good mechanical resistance, is not toxic and can be washed and reused and is biodegradable. A comparison of PVA, PVA-G and PVA-G-HA before and after heat treatment with two commercial masks, by scanning electron microscopy (SEM), shows differences in the fibre diameter. The smaller the fibre diameter and the more compact the woven fibres, the better is the PM capture, studied using a PM monitor. Post PM capture, the SEM shows a web-like film formation on the fibres and a distinct colour change. Anti-bacterial studies using two different bacteria, *Escherichia coli* and *Serratia marcescens* shows the zone of inhibition comparable to the control antibiotic. The UV absorption of all the fibres is in the 300—400 nm range with PVA-G-HA showing a maximum at 300 nm, an important property for face masks. The conclusion that can be drawn from detailed XPS studies is that the PVA-G-HA is capable of adsorbing toxic and pollutant gases from the mosquito coil which serves as a source of PM 2.5, considerably more than the other fibres studied. Mechanical studies also show PVA-G-HA to be the most stable fibre studied and hence the potential to be used as face masks/air filters.

Introduction

Electrospinning is a unique way of producing polymer nanofibres, it is possible to produce nanofibers with diameters ranging between a few nanometers to a few hundred nanometers. PVA is commonly used for continuous nanofibre fabrication, the average diameter from SEM is in nanometers which helps better PM capture. The fibres are washable and reusable, it is light in weight and is biocompatible. These are the many advantages compared to many commercial fibres. Electrospun nonwoven mats have small pore size, high porosity, high surface area and high fiber interconnectivity. The application that we are targeting is face mask and/or air filter with these fibres. Vehicular emissions, industrial exhausts, natural or agricultural emanations and incinerations, contain what is called Particulate Matter (PM); the major components of PM include organic compounds, nitrates, ammonia, sulphates, organic aerosols, sodium chloride and also gaseous elements. Fine PM usually consists of particles having a diameter between 0.1 and 2.5 μm and is designated as PM2.5. Owing to their ability to penetrate into the alveolar gas exchange region of the lungs, PM2.5 is also referred to as respirable particles. The coarse particles consist of particles having diameters greater than 2.5 μm, they may vary in size up to 100 μm [1, 2]. The most health-damaging particles are those with a diameter of 10 μm or less, which can penetrate and lodge deep inside the lungs [3]. From WHO studies of 2016 on PM2.5 and PM10 from measurements from about 3000 cities and towns worldwide, it was claimed that only about one in ten people breathe clean air [4]. Hence, it is no wonder that PM is known to cause respiratory, cardiovascular, cerebrovascular diseases, nervous and reproductive dysfunctions and carcinogenic disorders [4]. It is now a well-established fact that indoor pollution is more dominant than outdoor pollution because the exposure to PM mostly occurs indoors where people spend the majority of their time [5]. Many air filters have been developed over time, ranging from fibrous filters to porous filters. The commercially available air filters such as
precipitators, scrubbers, cyclones etc consume a lot of energy, power and space and have high operation and maintenance costs. Moreover, they are not very efficient in capturing <10 μm particles. Few of these filters are noisy and most of them are quite expensive. High Efficiency Particulate Air Filters (HEPA) is emerging and proving to be instrumental in improving air quality. But the drawback is that they are heavy and bulky, even though HEPA filters are used by a large number of industries, it is not very suitable for air filtration at homes as people at large are reluctant to make such exorbitant investments.

The pore size of the fibre developed in this paper unlike the ones mentioned earlier, ranges in nanometres and therefore its increased surface area enables improved capture efficiency. It is light and does not need to be replaced at recurrent intervals. Besides a good surface area, the fibre shows high chemical stability and mechanical strength. It is water resistant and thermally stable up to 160 °C. Also, the major highlight of this fibre is that it is antibacterial and UV-protective. Previous work on air filtration include fibres of chitosan/PEO blend solutions onto a spun bonded non-woven polypropylene substrate, chitosan, however, has low mechanical resistance and is difficult to electrospin [6]. Fibres of bimodal sized silica nanoparticles (SiO₂ NPs) incorporated into electrospun polyacrylonitrile (PAN), PAN nanofibres with ZIF modifications and transparent PAN fibres have been used as air filters. But PAN is not biodegradable, is toxic, not anti-bacterial and carcinogenic [7–9]. Nanofibrous membranes of Polyvinylidene fluoride (PVDF) as the matrix polymer and polytetrafluoroethylene nanoparticles (PTFE NPs) have been used for PM 2.5 capture, but again they are not biodegradable and not anti-bacterial [10].

Materials and methods

Materials

High purity natural graphite (GR) powder 200 mesh 75 mm was obtained from Alfa Aesar and Collagen Type I was purchased from Bovine Achilles tendon, Sigma-Aldrich. Calcium nitrate, diammonium hydrogen phosphate and liquor ammonia (30% v/v) solution were bought from Merck. The poly (vinyl alcohol) 96% hydrolyzed (MW: 95000) was provided by Acros Organics. For the antibacterial tests, Soyabean Casein Digest Medium and Nutrient Agar were both supplied by HIMEDIA. Distilled water was prepared by all quality double distiller (Bhanu) available in our laboratory. All the chemicals were of analytical grade and were used as received.

Synthesis of G, G-HA nanoparticles

Graphite was exfoliated using collagen to give a colloidal graphene collagen composite (designated as G). This is a patented process of the laboratory and the colloidal G synthesized has a graphene concentration of 0.2 mg ml⁻¹. Our previous study has shown that collagen is a largely hydrophobic protein where every third amino acid is hydrophobic glycine. Collagen exfoliates natural graphite into an average of 1–3 layer G-functionalized composite [11–14]. This is further used as a template to synthesize hydroxyapatite (HA) nanoparticles [15]. For the first step, 0.4 M calcium nitrate was mixed in 20 ml G-solution and liquid NH₃ was added to maintain the pH at 9.5. The mixture was stirred overnight. In the second step, 0.156 M diammonium hydrogen phosphate was dissolved in 20 ml G-solution and the pH was again maintained around 9.5 with the addition of liquor ammonia. Both the solutions were mixed and stirred overnight and left for seven days to age. Greyish-white precipitation was observed, the settled HA was washed till pH reaches 7 and the volume was made up to 50 ml by adding distilled water.

Fabrication of the PVA; PVA-G and PVA-G-HA fibre

All the nanofibres are electrospun using the Nanofibre Electrospinning Unit from Kato Tech Comp Ltd, Japan. Electrospinning comprises of the two very basic components- one is an electrically conducting spinneret and other is a collector separated at a specific pre-determined distance. The sample is loaded in the syringe and is placed at its specific position. The tip of the needle is connected to a positive electrode of high voltage and the other end attached to a grounded collector. The cylinder is covered with the Aluminium foil (30 × 30 cm²) as a collector for the electrospun fibres. The target speed is set around 2 m min⁻¹, the traverse speed for the electrospinning is set around 2 cm min⁻¹. The syringe pump speed is set around 0.1 mm min⁻¹ and the applied voltage is around 20 kV. The distance between the needle and the collector is maintained at 17 cm. These parameters maintained for all fibres.

For the PVA fibre, 30 ml of 8% PVA solution is taken in a syringe, for the PVA-G fibre, 24 ml of 10% PVA solution is mixed with 5 ml of G. G here acts as an added functionalization agent and is expected to bring variations in properties of the fibre. Further, for the third fibre i.e. PVA-G-HA, 1 mg of hydroxyapatite nanoparticles is added to the previous mixture of 24 ml 10% PVA solution and 5 ml of G colloidal composite. The fibre deposited on the collector is carefully peeled out and stored in a desiccator until further use.
Preparation of membranes for masks
The synthesized fibres, then heat treated at five different temperatures in a silica crucible inside the horizontal tubular furnace (Electroheat, Naskar & Co., West Bengal, India). The temperatures set are 120 °C, 140 °C, 160 °C, 180 °C and 200 °C for 30 min. PVA being water soluble, the fibres have a tendency to get dissolved. Therefore by heat treating the fibres, physical crosslinking takes place which makes the fibres water-proof.

Fibre characterization
Brunauer-Emmett-Teller (BET) was determined by N₂ adsorption-desorption method at a liquid nitrogen temperature using Quantasorb (Quantachrome, USA). Prior to adsorption-desorption measurements, the samples were degassed at 393 K at 10⁻⁹ Torr for 5 h. Chemical compositions of the nanofibrous membranes was determined before and after PM capture by XPS, using an Al monochromatic electron gun (energy = 1486.6 eV of SPECS, Germany), operating at a vacuum greater than 1 × 10⁻⁹ Torr. De-convolution of XPS spectra was done using CASA XPS software for elemental analysis. Surface morphology of nanofibre was studied by using a scanning electron microscope (Nova NanoSEM 450, FEI, US). ImageJ software was used to calculate the average pore size and fibre diameter. To measure the air filtration performance, the heat treated nanofibre with 20 cm² of exposed area was tested by using a particulate matter meter (TS 03, PM2.5 Air Quality Monitor, Oxy-Air, India).

Mechanical testing of nanofibres carried out using the table top Universal Testing Machine (UTM) (Instron Electropus E1000K6301, UK) equipped with a dynamic capacity of 1000 N. According to ASTM D638-10 Type V, the specimens were cut out from the synthesized fibres in the shape of a dumbbell which is the material test template for tensile strength measurements. 25 mm length, 10 mm width and thickness ranging between 0.02 and 0.05 mm was maintained for all the samples. The thickness of the fibre was measured using the Vernier Caliper. The ramp rate maintained at 0.5 mm min⁻¹ for all samples. During tests, the samples stretched up to the breaking point. From the original load-position data, the ultimate tensile strength was calculated.

Antibacterial tests
In vitro antibacterial tests for all the three different nanofibres of PVA, PVA-G and PVA-G-HA was done using two pathogenic bacteria (two gram-negative bacteria Escherichia coli and Serratia marcescens) with amoxicillin as the control. On the first day of the experiment, frozen bacteria (at −40 °C) is revived in the nutrient broth. To prepare the nutrient broth, 0.148 g of Soya-bean casein digest medium is dissolved in 50 ml of distilled water. The broth is divided into two different test tubes for the two separate bacteria which are then autoclaved at 120 °C for 15 min. Once the nutrient broth cools down to room temperature, one inoculum of the specific frozen bacteria is added to the respective test tube and left overnight in the incubator. The turbidity of the nutrient broth the following day visually confirms the growth of bacteria.

To prepare the nutrient agar medium, which is used as a culture medium for the tests, 2.8 g of Nutrient Agar Medium is added to 100 ml of distilled water. The solution and all the glassware needed for the experiment are autoclaved at 120 °C for 15 min. Once it cools down, it is poured into two different petri dishes and kept aside till they solidify. All these experiments are performed inside the Laminar Air Flow Blower (Gon Engineering Works, India). After agar petri dishes and the samples are UV-cured, 20 μl of the E.coli and S.marcescens is pipetted out on the agar surface using a 20–200 μl micropipette and spread evenly on the two agar plates respectively. Holes are drilled at specific locations in the agar plates in order to load the samples. The antibiotic amoxicillin is used as a control and is placed in the centre of both the bacteria containing solidified agar plates. The plates incubated at 37 °C for 18–24 h, the zone of inhibition measured after the incubation period.

Ultraviolet protective tests
The UV absorption was evaluated by the UV-Vis Spectroscopy (Shimadzu UV-2550 spectrophotometer, Japan). The sample placed in one cuboid and the reference solvent is placed in the other cuboid. The spectra of the sample is obtained after interaction with the UV-Vis light at the room temperature. The wavelength range maintained at 250–800 nm for all the experiments.

Results and discussion
The morphological studies of the synthesized fibres were done using FESEM. The three electrospun fibres were subjected to heat treatment at five different temperatures 120 °C, 140 °C, 160 °C, 180 °C and 200 °C for 0.5 h each. Out of the several polymers that are commercially available, PVA was chosen because it is non-toxic, inexpensive and biocompatible. It is also known to have high thermal and mechanical properties. Besides this, as the synthesis protocol for G and G-HA is aqueous it helps to have a water-soluble polymer [16–20]. PVA can be stabilized by physically or chemically crosslinking. While chemical crosslinking has the tendency to introduce
toxicity into the fibres, physical crosslinking with heat treatment is known to improve the stability and physical properties of the nanofibre minus the toxicity [21–23].

The SEM images before and after heat treatment were compared. The fibres observed to shrink and intertwine with increasing temperature. After 180 °C, the fibres curled, turned light brownish and became stiff. At 200 °C, it became hard and glassy and turned deep brown. The initial shrinkage can be attributed to the removal of moisture which makes the fibre appear stiffer. Heating is also known to affect the crystallinity of the fibre. When the temperature is around 180 °C and 200 °C, close to the melting point of PVA at 240 °C, the fibres tend to lose their integrity.

The diameter distribution shows that the PVA nanofibre which when heat-treated at 140 °C has the least average diameter of 369 nm. A smaller diameter makes the fibres flexible and enhances its mechanical strength and facilitates increased mobility of ions or air (in case of the air filter) across the pores. For PVA-G nanofibre membrane, a minimum diameter of 154 nm observed after the fibre was heat-treated at 140 °C. The decrease in diameter can be attributed to the addition of graphene to the PVA matrix. Change in solution properties changes the fibre diameter, a more conductive solution will allow greater stretching of the electrospinning jet due to the presence of more charge carriers. Since graphene is highly conductive, the addition of the G solution to PVA spikes the solution and increases its conductivity. And therefore there is a drastic change (almost 60% reduction) in the fibre diameter. In case of PVA-G-HA, a minimum fibre diameter of 104 nm observed in figure 1c, it may be assumed that HA might also have additionally spiked the solution and played a role in further reducing the surface tension. Lesser surface tension leads to greater fibre stretching as the force required to overcome the surface tension is less [24–27]. The diameter distribution for the PVA-G-HA fibres is shown in figures 1(a)–(d). Figure 1(a) shows the PVA-G-HA fibre at room temperature. After heat treatment, the diameter seems to alter and the change in the diameter with variation in temperature is shown in figures 1(b)–(d). The diameters of the commercial fibres are around 20–25 microns, much higher than the synthesized nanofibres. The average pore size of heat treated PVA, PVA-G and PVA-G-HA are 434, 402 and 395 nm, hardly much of a difference but this value is enough for efficient PM capture. The BET surface area of four different HA nanoparticle concentrations are 35.54, 166.66 and 176.35 m² g⁻¹ respectively and the highest surface area obtained was considered for PM capture.

The ability of the electrospun fibres to trap bacteria was determined by measuring the size of inhibitory zones on the agar surface around the well inside which the fibre was placed. It was observed that for control amoxicillin the zone of inhibition was 2.3 cm and for the fibres, PVA-G-HA and PVA-G, the zone of inhibition was restricted to 1.5 cm and 1 cm respectively when S.marcescens was used. In the case of E.coli, the zone of inhibition for control was 1.5 cm and for the fibres, PVA-G-HA and PVA-G, the zone of inhibition was restricted to 1.4 cm and 1 cm respectively. However, the PVA fibre and the commercial fibres showed no antibacterial activity as there was no zone of inhibition around the sample. The presence of the colloidal G composite makes the samples anti-bacterial. The results of the antibacterial activities are shown in table 1.

Literature reports show that graphene shows good anti-bacterial ability due to the transfer of charges from bacterial membrane onto the fibre till the bacterial membrane loses its viability. Since exfoliated graphene has very sharp needle-like edges, it also helps rip the membrane. PVA-G-HA and PVA-G fibres prove to be antibacterial and its activity can be compared to the antibacterial activity of an antibiotic. Quite naturally, it is clearly evident that the commercial fibres are prone to bacterial attack. With the rising tendency of airborne diseases, a mask which also promises to be antibacterial may be a better bet than the rest for face masks and indoor air-filters [28–30].

The ultra-violet range can be classified as UVA (320–380 nm), UVB (290–320 nm) and UVC (210–290 nm) of which the ozone layer blocks the UVC radiation and only UVA and UVB reach the earth surface [31, 32]. (figure 2, UV absorption spectra shown from 300 nm). Polymer composite materials containing nanomaterials, carbon black, carbon nanotubes and graphene have been used in masks to absorb UV. In the fibres synthesized in this study, we have used PVA + HA + colloidal graphene containing collagen. UV-absorbing materials should either contain pi-bonds or lone pairs. In the graphene structure, carbon is covalently bonded to three other carbon atoms in a hexagonal arrangement leaving one free electron for each carbon atom. Experimental colloidal graphene is multilayer graphene, for 1–3 layer graphene, the layers stacked on top of each other result in an array of delocalised carbon electrons. Each hexagon has 2 pi-electrons which are delocalized and this tendency of the overlapped pi-electrons extends all over its surface, therefore the movement of electrons is quite flexible. PVA has a high degree of conjugation and shows good absorption capacity for UV-radiation. Graphene has no band gap which further facilitates the free flow of electrons across the surface. HA, being a piezoelectric material, when exposed to UV rays, there is a possibility of charge transfer on the surface. Hence, in the figure, it is evident that PVA-G-HA and PVA-G exhibit higher absorption as compared to the blank PVA fibre. The commercial white mask shows very strong UV absorption at 300 nm which means it could be coated with TiO₂ nanoparticles. Commercial blue mask, however, is not as good as the experimental fibres.
PVA has substantial tensile strength, flexibility and hardness. The physical structure of PVA is largely dependent on its molecular weight and degree of hydrolysis. Since we have used a high molecular weight PVA to prepare the fibre (MW: 95000), we obtained a fibre with impressive strength. It has been reported that higher molecular weight and a greater solution concentration may give considerably higher tensile strength but the process of electrospinning becomes difficult [33]. The addition of exfoliated graphite is expected to improve the mechanical properties because of innumerable interfacial interactions. And it is quite evident from the results, that addition of G does increase the tensile strength of the nanofibres. From the Stress-Strain curve in figure 3, 

Figure 1. The SEM images and the diameter distribution of the PVA-G-HA fibre at different temperatures (a) room temperature (b) 120 °C (c) 140 °C and (d) 160 °C.
Table 1. Zone of inhibitions of the fibres in the case of E. Coli and S. Marcescens.

| Sl. No | Sample       | Well size (cm) | Sample volume (μl) | Zone of inhibition E. coli (cm) | Zone of inhibition S. Marcescens (cm) |
|--------|--------------|----------------|--------------------|---------------------------------|---------------------------------------|
| 1      | Amoxicillin  | 1              | 20                 | 1.5                             | 2                                     |
| 2      | PVA-G-HA     | 1              | 20                 | 1.4                             | 1.6                                   |
| 3      | PVA-G        | 1              | 20                 | 1.0                             | 1.0                                   |
| 4      | PVA          | 1              | 20                 | None                            | None                                  |
| 5      | Commercial-1 | 1              | 20                 | None                            | None                                  |
| 6      | Commercial-2 | 1              | 20                 | None                            | None                                  |

Figure 2. The UV absorption spectrum of the synthesized nanofibres compared to the commercial masks.

Figure 3. Mechanical tests denoting Stress Strain curves for the three different nanofibres.
the heat treated PVA fibre has a tensile strength of 3 MPa and the PVA-G fibre has a tensile strength of 4.2 MPa. There is a significant increase of about 40% after the addition of graphene. In PVA-G-HA, HA nanoparticles offer further interaction possibilities per unit area and hence helps in enhancing the mechanical properties. The tensile strength of PVA-G-HA is 5.4 MPa. The weight of the fibres of 1 cm x 1 cm area range from 0.002–0.004 g, the fibres are extremely flexible and can tolerate a bending of 180 deg with absolutely no weight loss [34, 35].

Previous reports state that electrospun fibres exhibit higher mechanical strength as compared to non-spun fibres. These electrospun fibres do not necessarily have a necking phenomenon which gives it an edge over the non-spun fibres. Also, it has been stated that a decrease in fibre diameter gives away to better interlocking between the molecules in the fibre matrix which leads to increased tensile strength [36, 37]. It was difficult to get fibres of similar thickness and weight from the commercial masks and hence their mechanical studies not done.

To observe the capture of PM2.5 particles on the synthesized fibres, mosquito coil was used as a potential source of particulate matter. The smoke comprises oxides of sulphur, nitrogen, carbon and volatile organic compounds. Pollutants in the smoke of mosquito coil, indoor pollutants, can be compared to smog, outdoor pollution as well. We studied the PM capture process by using the setup as shown in figure 4(a). It the setup, the chambers are separate and nanofibre is placed in the middle. The percentage efficiencies recorded by the PM2.5 detector are mentioned in figure 4(b). It is clear that the PVA-G-HA fibre trapped PM2.5 the best as compared to the others.

For all the three fibres, SEM was done before and after the exposure to PM2.5. It is evident from visual observation of the SEM micrographs that the PM particles were captured on the surface. As the synthesized fibres have an average diameter in the range of 100–300 nm, it easily traps the PM particles which are much bigger and have a size of about 2.5 microns. The SEM images of the different fibre surfaces before and after PM capture is shown in figures 5(a)–(c). The particles seem to form a thin layer over the surface resembling a web-like structure. PM capture is based on various factors but the most dominating factors here is fibre diameter and high surface area. Electrospun nanofibres with different properties result because of varying functional groups on the polymer side chains, and other additives, for e.g., PVA has only –OH group. Here, in addition, G is exfoliated using collagen protein, in which HA nanoparticles are in situ synthesized. HA is known to be a very strong adsorbent, hence the addition of HA nanoparticles enhances the PM capture [38, 39]. The values of capture efficiencies for PVA, PVA-G, and PVA-G-HA are 90, 93 and 96% compared to the commercial ones where one had capture efficiency of 92% and the other had 60%. The 6% difference between PVA and PVA-G-HA is significant owing to its added properties like increased mechanical strength, its anti-UV property and antibacterial effect. Collagen, present inherently in the G accounts for a large number of polar and non-polar functional groups. It is a well-established fact that characteristics of the chemical functional moieties of the fibre determine their capability for interaction with other foreign or external polar molecules enabling PM capture. The interactions with PM include H₂ bonding, hydrophobic bonding, Vander der Waals interactions or electrostatic interactions. In a few of these active sites, there may also be a possibility of chemical reaction with the surface functional groups. For the three fibres- PVA, PVA-G and PVA-G-HA, the size effects backed by
strong charge-charge interactions ensure maximum particle trapping. The notable change in colour as observed from the photographic images in the inset of SEM (shown in figures 5(a)–(c)) also denotes trapping of particles. It is evident from the SEM images that the particles attach to the fibres and are tightly bound and wrapped around the fibres, forming a thin layer.

To understand the gas adsorbing ability of fibres we carried out the XPS studies of the smoke (coil) exposed fibres. The XPS data for all the three fibres yielded more or less similar peaks and hence only the representative PVA-G-HA is shown. The survey scan shows the prominent C1s and O1s peaks at the ratio of 85.57:14.43 but the N1s peak is not observed. However, the only slow scan of the spectrum around 400 eV shows the presence of N1s peaks as the content of N content is low. In order to identify the captured particulates, we have studied the C1s, O1s and N1s peaks in detail. The C1s peak (figure 6(a)) shows a broad asymmetric peak which is de-convoluted

![Figure 5. SEM and digital images of (a) PVA (b) PVA-G and (c) PVA-G-HA nanofibres before and after exposure to PM particles.](image-url)
using CASA XPS software. The peak fitting data indicates that the spectrum fitted well into two peaks. The lower binding energy peak (284.6 eV) corresponds with the energy values of C–C/C=O peak, while the higher binding energy peak of C1s at 287.3 eV, suggests that some of the carbon atoms are attached with the electron withdrawing functional groups. The possibility of such functional groups, considering smoke chemical composition, could be CO, CO2, NO3, NO2 etc. These adsorbed functional groups tend to decrease the electron density of the C atom due to its electron withdrawing nature. As a result, the binding energy of C–C/C=O shifts towards higher binding energy. In addition, it is noted that the peak intensity corresponding to 287.3 eV is higher than that of the 284.6 eV peak, suggesting that significant amount of these functional groups are adsorbed on the surface of the fibre. To precisely evaluate the electron withdrawing functional groups, we have analyzed the slow scanned N1s and O1s peaks. Both these peaks also appear to be broad and asymmetric. Furthermore, the previous reports show that the full width half maxima (FWHM) of the oxy-nitrogen species appears in the range of 1.3 to 1.7 eV [40]. However, the contribution of FWHM in this case is larger, > 3 eV, indicating adsorbed gas contains more than one functional group. The de-convoluted data of N1s spectrum (figure 6(c)) indicates that the spectrum consists of two peaks at the peak positions, 406.03 and 402.59 respectively. Based on the earlier reported data, these two peaks can be assigned to the NO and physio-absorbed NO2 entities [40]. In addition, it is evident from the O1s spectrum (figure 6(b)) that CO and CO2 groups are adsorbed on the surface of the fibre. The analysis of the fitting data shows that the O1s peak is composed of two components; 532.6 and 329.7 eV corresponding to CO2 and O2− groups, respectively. The conclusion that can be drawn from the XPS data is that the fibre is capable of adsorbing toxins and pollutant gases from the smoke.

Figure 6. XPS peaks of the nanofibre PVA-G-HA after PM capture denoting (a) C1s (b) O1s and (c) N1s spectra.
Conclusion

This work optimized the electrospinning parameters for a multi-functional PVA-G-HA fibre with the required pore size and surface area for PM capture. The heat treatment criteria for the fibres were also optimized for maximum absorption of toxic gases and mechanical stability. The detailed analysis of the C1s, O 1s and N 1s peaks prove the ability of the PVA-G-HA fibre to absorb the toxic gases. The PM capture was repeatedly checked using a PM meter. Its anti-bacterial and UV absorption properties make PVA-G-HA fibre a potential candidate as an air-filter/mask.

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