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Synthesis of *Aloe Vera*-lignin Based Electrospun Air Filter

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Received: *15 March 2021*
Accepted: *2 June 2021*
Published online: *23 February 2022*

Keywords: *electrospinning, aloe vera-lignin, bio-air filter, filtration efficiency, masks, antimicrobial finish*

The current study aimed to synthesize an *Aloe vera* -lignin-based electrospun air filter with an antimicrobial finish to improve filtration efficiency for particulate matters, gaseous pollutants, and pathogenic microorganisms. The synthesized material was characterized by SEM and FTIR. The electrospun layer was combined with non-woven polypropylene fabrics coated with the tested antimicrobial finish on the interior and exterior fabrics, along with activated carbon cloth to form hybrid masks. The filtration efficiency of the electrospun air filter and the hybrid mask was evaluated using Cigarette secondhand smoke which showed significant performance compared to the tested commercial masks with about 95-99% for micron size particles (1 µm, 2.5 µm, 5 µm, 10 µm), 95-99.9% for particulate matter (PM_1, $PM_{2.5}$, PM_{10}), and 82-98% for various gaseous pollutants (HCHO, TVOC, CO_2). It was evaluated that the bio-based electrospun air filter showed significant performance compared to tested commercial masks and can efficiently be used as an alternative filter media that can alleviate air contamination and simultaneously preserve good breathability. Thus, this study can be regarded as a promising solution for air filtration following eco-friendly, green, and sustainable development strategies.

Introduction

Masks have now become a part of daily life since the outbreak of COVID-19 and to combat the increasing atmospheric pollution load and infectious microorganisms. Though the commercially available multi-layered synthetic masks claim better protection against air pollutants and microorganisms, most of them have shown disadvantages of poor $PM_{2.5}$ rejection, low air permeability, and inefficiency against microorganisms [1]. Further, the synthetic nature of the available masks takes a longer time for degradation, leading to environmental and health implications during usage as well as disposal and economically not feasible for regular usage [2].

The air filter membranes have a great role in the filtration process that reflects on the filtration efficiency and filtration result. Air filter membranes with various functions like antibacterial, high-temperature performance, excellent mechanical and filtration properties, hydrophobic, have increased attention mostly in recent times. Compared with conventionally used mask filter media, Electrospun membranes possess unparalleled advantages such as controllable small diameter, porous structure, high surface area to volume ratio, good internal connectivity, and controllable morphology [3], which implies higher filtration efficiency at low cost without the sacrifice of permeability during the filtration process significantly increasing the diffusion, interception, and inertial impaction efficiencies [4]. Hence, the emerging bio-based electrospun membrane, which is environment-friendly and harmless to the human body can be considered for developing the high-performance air filter media.

With the increase in new antimicrobial fiber technology, various methods have been developed to increase the effectiveness of the respirators. A range of synthetic antimicrobial products such as triclosan, metals and their salts, organometallics, and their quaternary ammonium compounds are used extensively. But those nanoparticles themselves have several side effects on human health. It is worth mentioning that due to the unknown effects of nanoparticles, the use of such material in the masks can lead to skin irritation in people with sensitive skin [5]. Although they claim to be effective, they are of concern due to the associated side effects, action on non-targeted areas, and cause water pollution. As there is a great demand for antimicrobial agents based on natural eco-friendly agents, researchers are seeking new types of safe and cost-effective materials which not only help to improve the antimicrobial effect but fulfill statutory requirements by regulating agencies [2,6]. However, as of our knowledge, no study

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was undertaken to incorporate plant-based antimicrobial agents in the respiratory mask product.

Numerous studies were published on the application of fabrics treated with *Aloe vera* [6-10] imparting antifungal, antitumor, anti-inflammatory, anti UV, antimicrobial and antiviral agents [11,12]. In different trials, the electrospinning method was used to produce nanofiber composites loaded with *Aloe vera* for wound dressings and scaffolds [13].

Lignin, second to cellulose in natural abundance, is readily available, relatively inexpensive [14], biocompatible, viscoelastic with a good film-forming ability and small particle size, great efforts are being made to find new applications and research like the addition of lignin to biodegradable polymers, permitting both the improvement of mechanical properties and acceleration in the rate of biodegradation [15] for their potential use in biomedical applications. Studies have revealed that lignin and lignin extracts have antimicrobial and antifungal properties [16], act as antioxidants [17], absorb UV radiation [18]. The hydrophilic or hydrophobic character of lignin depending on origin, allows a wide range of blends to be produced [19]. Also, the functional groups present in the lignin suggest that it can act as a precursor for activated carbon adsorbents. Together, all this makes lignin an ideal natural polymer for the synthesis of environmentally friendly nanomaterials.

However, limited scientific evidence is available for the utilization of *Aloe vera* leaf rind that is spawned as waste during their processing [20]. In this context, the present study tried to synthesize *Aloe vera*-Lignin-Based Electrospun Air Filter and evaluated its filtration efficiency using cigarette secondhand smoke.

Materials and Methods

In this study, *Aloe vera* is chosen as the raw material for the extraction of lignin and antimicrobial agent for producing the antimicrobial treated bio fabric. *Aloe vera* plants were collected from the natural habitats in Coimbatore, Tamilnadu, India. The gel and the rind of *Aloe vera* were separated, shade-dried, powdered, and stored in a refrigerator for further use.

Lignin extraction

10 g of dried *Aloe vera* rind powder is subjected to alkaline treatment with 20% NaOH for 4 hrs and centrifuged at 2000 rpm for 15 minutes. It is then filtered and washed with distilled water. It is followed by acidified sodium hypochlorite bleaching (0.7 g in 100 ml) for 4 hrs and centrifuged, filtered, washed, air-dried, powdered, and stored [21].

Electrospinning

0.8 g lignin, 0.2 g dried *Aloe vera* gel powder was dissolved in 5 ml Trifluoroacetic acid (TFA) and 0.5 g Polybutylene terephthalate (PBT) was added to it and dissolved completely. About 5 ml of the solution is used for electrospinning with a flow rate of 500 µl at 10 kV and a distance of 12 cm for a period of 2 hrs [22].

Soxhlet extraction

The antimicrobial components from shade-dried, powdered *Aloe vera* gel were extracted using Soxhlet extractor for 24 hrs (four cycles per hour) using (10% w/v) methanol, ethanol, acetone, hexane [6].

Antibacterial assay by agar well diffusion method

Agar well diffusion method was used to evaluate the antimicrobial activity of the plant extracts against standardized inoculums of the test organisms (*Staphylococcus aureus*, *Escherichia coli*). It was uniformly spread on the surface of Mueller-Hinton agar (M173 Hi-Media) plates using a sterile cotton swab. Gentamycin and DMSO were maintained as positive and negative control respectively. It was then incubated under suitable conditions and after incubation, a clear zone of inhibition of the bacterial growth was observed and measured in mm.

Antimicrobial finish application

Aloe vera methanol extract that showed a better zone of inhibition against tested microorganisms was applied on the interior and exterior fabric of the masks by the pad and dry method. The fabrics were immersed in the 5 g/L concentration of methanol extract for five minutes and padded on a padding mangle individually in the presence of citric acid to maintain pH 5.5. It was again immersed in the solution for another five minutes and repeated the same process and then dried at 80ºC for 3 min and cured at 110ºC for 2 min on a lab model curing chamber [9].

Formation of hybrid mask

For comparing the particle filtration efficiency with the commercial masks, a hybrid mask (4 plies) was developed by sandwiching the electrospun air filter layer (1 ply) and activated carbon layer(1 ply) between the non-woven polypropylene fabric with antimicrobial finish (2 plies).

Particle filtration efficiency

This procedure was performed to evaluate the particle filtration of the test samples. The procedure employed the basic filtration method with some exceptions; notably, the procedure incorporated passing of cigarette secondhand smoke consisting of smoke, gas/aerosols through the test sample. The particles that passed through the test sample were enumerated using CAIR+ Monitor, Prana Air, a laser particle counter working on the principle of light scattering. It can measure the parameters like $PM_{2.5}$, PM_{10}, CO_2, HCHO, TVOC, temperature & humidity with an accuracy of 0-150 µg/m^3 ± 10% and 150 µg/m^3 ± 15% onwards for particulate matters. The ranges are within 0-2000 µg/m^3 for particulate matter, 0-2000 PPM with a resolution of 1 PPM for CO_2, 0-2 PPM with a resolution of 0.001 PPM for HCHO, and 0-20 PPM with a resolution of 0.01 PPM for TVOC. Three one-minute counts were performed, with and without the test sample in the experimental setup (figure 1), and the results were averaged. Control counts were performed to determine the average number of particles delivered to the test sample. The filtration efficiency was calculated using the average number of particles penetrating the test sample compared to the average of the control values.

Filtration efficiency of tested filters

Filtration efficiency (FE %) is calculated by the following equation.

FE % = [(Upstream value-test mask value)/Upstream value] x 100

Fourier transform infrared (FTIR) spectrum of lignin

The extracted lignin was subjected to FTIR analysis with the sample

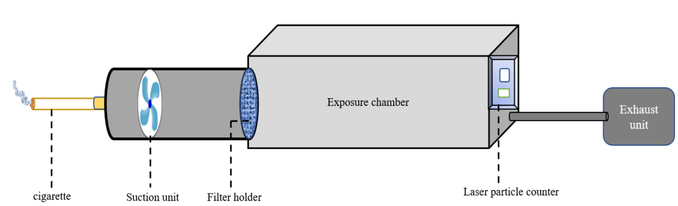

Figure 1: Particle filtration efficiency setup

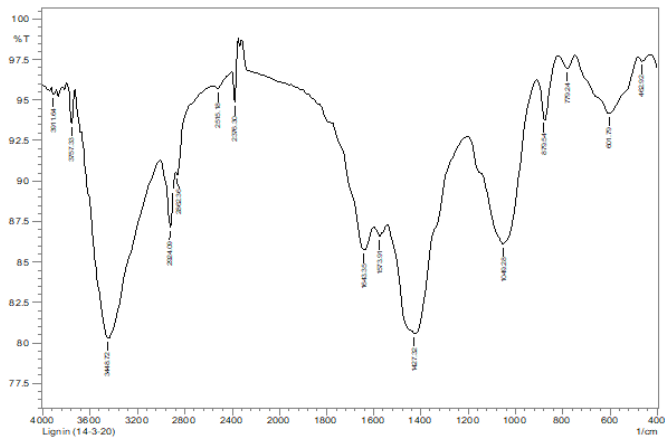

Figure 2: FTIR spectrum of lignin extracted from aloe vera

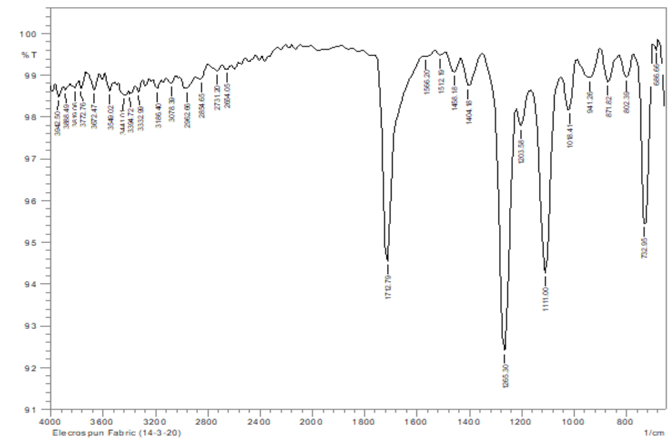

Figure 3: FTIR spectrum of electrospun Aloe vera-Lignin and PBT Nanofibers

prepared with KBr in the pelletized form. The FTIR spectrum of the sample was recorded in the region 4000-400 cm^{-1} employing Shimadzu IR Affinity 1 FTIR Spectrophotometer with a resolution of 16 cm^{-1}.

The FTIR spectrum of lignin (figure 2) has a strong wideband between 3500- 3100 cm^{-1} assigned to O-H stretching. This band is caused by the alcoholic and phenolic hydroxyl groups involved in hydrogen bonds. The bands at 2920 and 2860 cm^{-1} are due to C-H stretching vibrations of the methoxyl group. 1600 and 1500 cm^{-1} are characteristics of aromatic compounds (phenolic hydroxyl groups) and are attributed to aromatic skeleton vibrations and 1049.28 strong, broad, stretching vibrations of carbonyl groups. Bands were observed at 1427 cm^{-1} (deformation combined in CH_3 and CH_2), 2515 cm^{-1}(O-H stretching of the carboxylic group). In the region, 900-700 cm^{-1} absorption bands caused by deformation vibrations of C-H- bonds on the benzene ring are located. The band at 601 cm^{-1} is attributed to strong C-I stretching of halo compound may be caused due to bleaching of lignin [21,23].

Attenuated total reflectance - fourier transform infrared spectrum (ATR-FTIR) of electrospun fibers

Attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) (IRAffinity-1, Shimadzu, Japan) equipped with a single reflection ATR accessory (MIRacle 10) was used to determine the functional groups of the electrospun fibers in the wavenumber range from 4000 to 700 cm^{-1} and a resolution of 16 cm^{-1}.

The FTIR spectrum of electrospun fibers (figure 3) confirmed the presence of functional groups present in lignin (figure 2) along with the appearance of characteristic stretching and bending vibrations of PBT structure as follows: The asymmetric and symmetric stretching vibrations in C–H bonds have been identified at 2954–2960 cm^{-1} due to the vibrations of CH_2 groups of PBT. Meanwhile, bending vibrations of C–H bonds are located between 1360 and 1454 cm^{-1}. The bands corresponding to C–O, C–O–C, C=C stretching vibration are identified at ~1260, ~1100, and ~1578 cm^{-1}, respectively. The sharp peaks at 1710 and 725 cm^{-1} at the PBT spectrum can be attributed to ester group C=O stretching vibration and aromatic ring C–H out-of-plane deformation, respectively. The spectral analysis indicates a good polymeric binding property of lignin with PBT due to the polymeric hydrogen bonding of O-H stretching. Also, the functional groups present in the lignin favor the high adsorption of gases.

Scanning electron microscope (SEM) analysis

The electrospun fibers were sputtered with gold before observation under vacuum. Fiber formation and morphology of the electrospun PBT *Aloe vera* - lignin fibers were determined using the Scanning Electron Microscope (FEI Quanta 200, ICON analytical).

SEM micrographs (figure 4) showed that the fibers were non-woven, homogenous, finely spun with the uniform binding of *Aloe vera* and lignin, that increased porosity for effective filtration of airborne contaminants. Also, PBT played a crucial role in

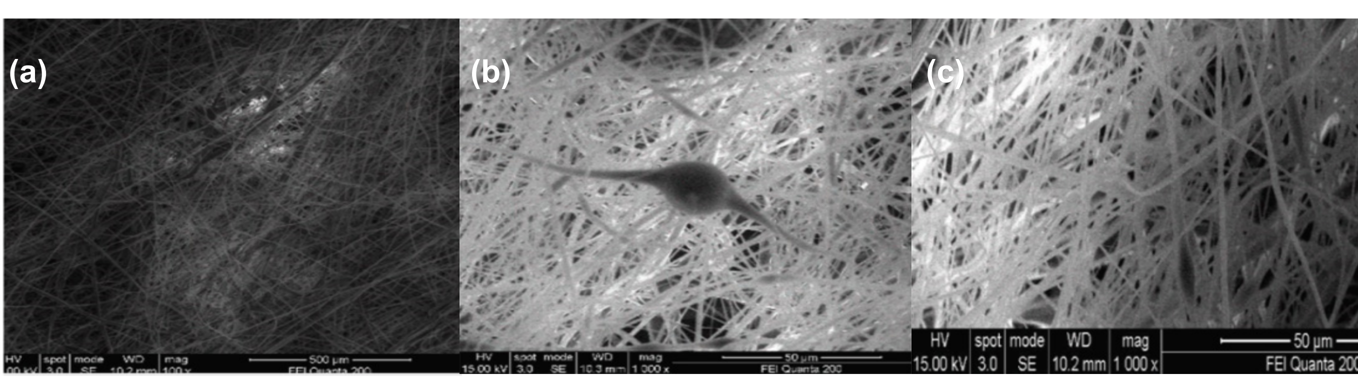

Figure 4: SEM Micrographs of the electrospun *Aloe vera*-lignin and PBT; a) at 500 µm with 100 x magnification , b) and c) at 50 µm with 1000 x magnification

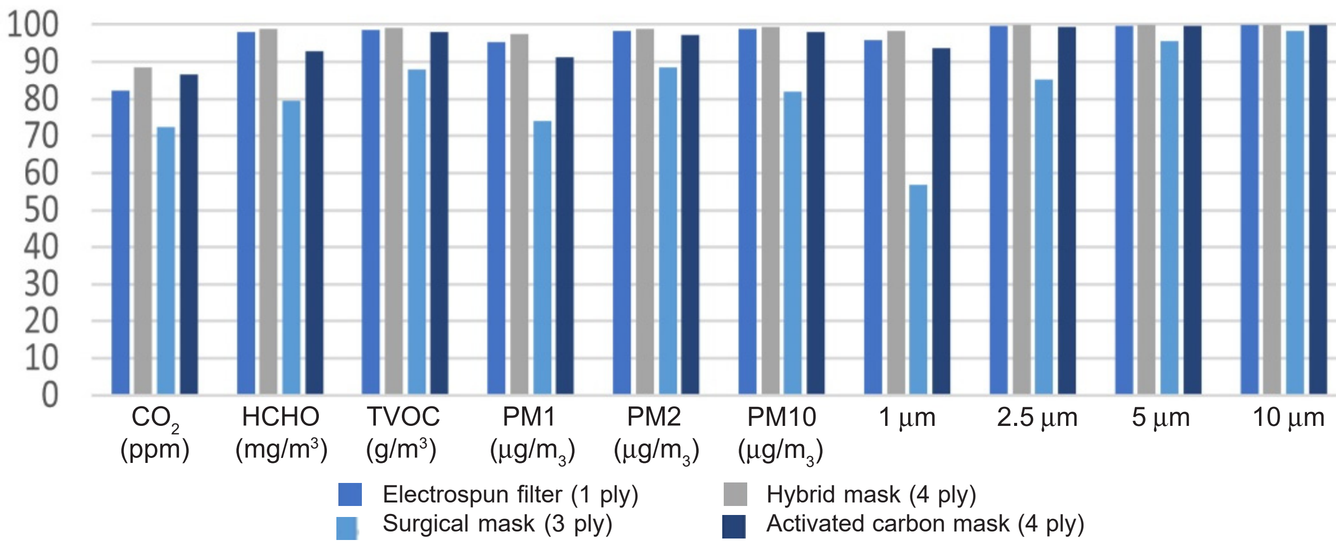

Figure 5: Filtration efficiency of test samples for different parameters

facilitating electrospun lignin fibers due to its effects on the solution's viscosity, electrospinning behavior, and morphology of fiber.

Results and Discussion

Antibacterial assay

The better-extracting solvent to recover antimicrobial agent was identified as methanol with a zone of inhibition of 18 mm and 17 mm using the agar well diffusion method for *Escherichia coli* and *Staphylococcus aureus,* respectively. The results of the antibacterial assay (table 1) indicate that *Aloe vera* exhibits its activity against *S. aureus* and *E. coli* to a reasonable extent. The zone of inhibition for *Aloe vera* is less when compared to the zone of inhibition of other synthetic antibacterial agents. This can be understood from the point that Aloe vera gel consists of almost 200 components or ingredients and its antibacterial agents may be present in less quantity as compared to other components [6].

Particle filtration efficiency of test samples

Particle filtration efficiency of test samples for different parameters is summarised in table 2 and figure 5 illustrates the graph of filtration efficiency of test samples for different parameters. The filtration efficiency for 1 µm size particles was recorded highest with 98.48% in the hybrid mask followed by the electrospun air filter with 95.87% and the activated carbon mask with 93.74%. Also, for 2.5 µm, 5 µm, and 10 µm size particles, the hybrid mask shown the highest filtration efficiency with above 99% followed by the electrospun air filter and the activated carbon mask in a similar range. The surgical mask showed the least filtration efficiency for micron size particles which eventually decreased with the decrease in particle size.

Table 1: Antibacterial assay of methanolic extract of *Aloe vera*

Samples	Concentration	Zone of Inhibition	
		E. coli	*S. aureus*
Standard	10 µg	23 mm	25 mm
	50 µl	16 mm	15 mm
Methanolic Extract of *A. vera*	75 µl	17 mm	16 mm
	100 µl	18 mm	17 mm

In the case of PM_1, $PM_{2.5}$, and PM_{10} the order of filtration efficiency in the increasing order is hybrid mask>electrospun air filter> activated carbon mask> surgical mask. Filtration efficiency was also recorded the highest by the hybrid mask for the tested gaseous pollutants (HCHO, TVOC, and CO_2); followed by the electrospun air filter for HCHO and TVOC and the activated carbon mask for CO_2. It must be noted that the challenge particles used in the previous filtration efficiency studies have been NaCl [24], diesel emissions [25], standard polystyrene latex spheres [26] parafin oil [27], or volcanic ash particles [28] depending on the purpose of the masks. There is evidence that the particle size of the challenge aerosol/dust as well as the mask material and flow rate, impact substantially on the filtration efficiency [28]. Results of previous studies, especially those which used ultrafine, homogenous, or coarse aerosols, can therefore not be directly translated and applied to secondhand cigarette smoke.

Compared with conventionally used mask filter media, the electrospun filter showed better performance due to the addition of *Aloe vera* and lignin resulting in the formation of finer fibers with increased porosity, enhanced flow, high surface area to volume ratio, good internal connectivity, and controllable morphology which guarantee the excellent filtering performance [3]. Also, the performance of the hybrid masks achieved excellent filtration efficiency for particulate matters, gaseous pollutants as well as pathogenic micro-organisms which was comparatively higher than commercially available masks. This may be attributed to the fabrication of masks by incorporating additional layers of the non-woven spun-bond layer with an antimicrobial coating of *Aloe vera* that might have acted as a bulwark for penetration of tested pollutants along with activated carbon layer and electrospun air filter with relatively highest performance. The filtration efficiency exhibited by the electrospun air filter for gaseous pollutants indicates lignin as a precursor to activated carbon in the adsorption of gases. Yet, the role of dried *Aloe vera* gel and the lignin extracted from the rinds of *Aloe vera* in gaseous adsorption has to be studied further.

Table 2: Filtration efficiency of different parameters by test samples (in Percentage)

Parameters	Electrospun filter (1 ply)	Hybrid mask (4 plies)	Surgical mask (3 plies)	Activated Carbon Mask (4 plies)
CO_2	82.25	88.59	72.45	86.69
HCHO	98.02	98.8	79.4	92.8
TVOC	98.7	99.2	87.89	97.98
PM_1	95.35	97.51	74.03	91.35
$PM_{2.5}$	98.35	98.96	88.51	97.21
PM_{10}	98.9	99.38	82.1	98.2
1 µm	95.87	98.48	56.91	93.74
2.5 µm	99.69	99.94	85.17	99.37
5 µm	99.81	99.96	95.53	99.81
10 µm	99.97	99.99	98.45	99.94

Conclusion

The present study successfully synthesized an electrospun *Aloe vera* -lignin air filter with high filtration efficiency for particulate matters, gaseous pollutants as well as pathogenic micro-organisms. It was characterized by SEM, FTIR and evaluated the efficiency in terms of filtration using cigarette second-hand smoke. The particle filtration efficiency test using cigarette secondhand smoke shows that the synthesized air filter and hybrid mask showed significant performance for micron size particles, particulate matter, and gaseous pollutants than the tested commercial masks. Though the air filters used in masks, air filtration, and air purification systems claim higher filtration efficiency for particulate matters and micron size particles, they fail to provide better filtration of gaseous pollutants and are not biodegradable. The blending of the natural polymers (lignin extracted from the rinds of *Aloe vera* and *Aloe vera* gel powder) with the synthetic polymer PBT improves its mechanical properties, accelerates the rate of biodegradation, and imparts its medicinal properties. It also facilitated the reduced dependence of synthetic polymers in the preparation of air filters. The results also validate that lignin can be used as a precursor to activated carbon thereby reducing carbon footprints.

Thus, this study can be efficiently used as an alternative filter media to alleviate air contamination and simultaneously preserve good breathability. As there is an ever-growing demand for novel, efficient, and long-life filtering media and based on the results of this study, future research in this study include: tailoring new functional materials as a membrane or filtration systems (either on their own or in combination with other filtration media) offering a potential solution for a wide range of environmental issues such as air filtration, water purification, process industries following eco-friendly, green, and sustainable development strategies.

Acknowledgement

This research was partly funded by Tamilnadu State Council for Science and Technology, Chennai through the project grant sanctioned for ES-025 under the Student Projects Scheme 2019-2020. The authors acknowledge the assistance in electrospinning by Polymer Lab, The South India Textile Research Association, Coimbatore.

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