

Enhancing wettability and antimicrobial activity of lotus fabric through air plasma treatment and neem extract coating

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This study investigates the functional enhancement of lotus fibres — a sustainable, environmentally friendly and easily accessible raw material — through atmospheric dielectric barrier discharge (DBD) plasma treatment. The fabric is exposed to air plasma for durations ranging from 1-5 min before being coated with five different concentrations of neem leaf extract. The surface wettability of the treated fabric is assessed through wet-out time analysis, hydrophilicity tests, and contact angle measurements. Structural and chemical modifications are characterised using Attenuated Total Reflectance–Fourier transform infrared (ATR-FTIR) spectroscopy, Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM), and X-ray photoelectron spectroscopy (XPS). FTIR and XPS analysis confirms increased polar functional groups (-COOH, -OH, -C=O) on the plasma-treated fabric surface, improving the surface hydrophilicity and functionality. Antimicrobial properties are evaluated using agar plates and modified Hoenste in tests, both qualitatively and quantitatively. Results demonstrate that when coated with neem extract, the plasma-treated lotus fabric exhibits durable antimicrobial activity even after three washing cycles. This suggests that air DBD plasma treatment followed by neem leaf extract application offers a better approach for manufacturing natural antimicrobial textiles, with potential applications in medical and healthcare industries.

Keywords: Natural extract, Natural fibre, Lotus fibre, Plasma treatment, Surface modification, XPS analysis

1 Introduction

The textile sector creates a massive water footprint with reference to water usage for crop farming, water consumption during textile production, and water pollution¹. Cotton is a naturally occurring fibre extensively employed in the textile industry since it is a soft, light, and breathable fabric. However, cotton farming is typically associated with the usage of chemicals, the textile effluent comprises nutrients, salts, and pesticides, and cotton production requires a large land area². Furthermore, despite providing significant socioeconomic advantages, genetically modified Bt cotton struggles with various drawbacks, such as Bt resistance emergence and secondary pest outbreaks³⁻⁵.

The use of synthetic fibres, on the other hand, is associated with the requirement of high quantities of water for processing along with the release of massive amounts of microplastics in the water bodies that threaten to the aquatic ecosystem⁶. Considering the drawbacks linked with the over-exploitation of cotton and synthetic fibres, as well as with emerging public awareness of environmental problems, there appears

to be a huge potential for sustainable textiles procured from agricultural residues to overcome these issues⁷. Recently, the application of sustainable, eco-friendly fabrics has become a rapidly emerging trend among both manufacturers and consumers owing to their appealing properties, such as abundant availability, renewability, increased comfort, and biodegradability. One sustainable cellulosic fabric procured as an agricultural residue is lotus, whose stalks are discarded following the flowering season or the harvesting of lotus root. These wastes might provide a rich supply of natural cellulose in the textile, construction, medicinal, and paper sectors.

Lotus (*Nelumbo nucifera*) belongs to the family Nymphaeaceae and is an aquatic perennial plant native to temperate as well as subtropical regions⁸. Owing to the numerous advantageous features such as easy cultivation practices, inexpensive nature, and remarkable yield, it is abundantly grown throughout Asian nations⁹. According to several investigations, the lotus plant possesses significant beneficial components such as vitamins, polysaccharides, antioxidants, starch, alkaloids, flavonoids, steroids, catechins, and phenols¹⁰⁻¹². Apart from all of the advantages of lotus fabric, it has a few drawbacks that restrict its utilisation. The fabric is hydrophobic due to

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the presence of non-cellulosic components such as lignin, hemicellulose, pectin, and waxes on its surface, and the fibre is also susceptible to microbial damage¹³.

With the ever-increasing global warming situation, implementing changes to traditional processing methods is more vital than ever to implement changes to traditional processing methods¹⁴. Plasma treatment is one such eco-friendly technique that addresses the issue of green chemistry. The dielectric barrier discharge (DBD) plasma treatment is a dry, economically feasible and environmentally friendly process of surface modification that has gained considerable attention in the recent past as it is capable of altering the surface characteristics of materials without modifying their bulk properties^{15–21}. The current study presents a unique method for improving various surface properties of lotus fabric by using the DBD plasma technology and subsequently coating the surface with neem extract to prevent the fabric from microbial attacks. Neem (*Azadirachta indica*) is a medicinal plant from the Meliaceae family. The leaves of the plant are known to possess various therapeutic properties, including antibacterial, antioxidant, and anticancer properties, such as nimbin, nimbidol, gedunin, sodium nimbin, quercetin, salannin, and azadirachtin^{22,23}.

In this investigation, DBD air plasma is employed to functionalise the lotus polymer fabric, which is then coated with a natural neem extract coating to overcome its antibacterial property limitation. The study investigates the changes in surface wettability, absorption, wicking behaviour, contact angle, surface roughness, morphology, and chemical composition of the fabric before and after treatment.

2 Materials and Methods

2.1 Sample Preparation and Plasma Treatment

Lotus fabric was selected as the base material for surface modification. The fabric samples were cut into 5 × 5 cm squares and subjected to atmospheric-pressure dielectric barrier discharge (DBD) air plasma treatment. The treatment was performed for durations ranging from 1 to 5 min. All subsequent analyses were conducted on plasma-treated and untreated control samples.

2.2 Surface Wettability Assessment

The wet-out time of the lotus fabric was evaluated according to AATCC Test 79-2007. This test measured

the time water took to completely wet the fabric surface, allowing comparison between untreated and plasma-treated samples. Changes in hydrophilicity/water absorption were carried out using the British Standard 3449:1990 (Static Immersion Test). The water contact angle was measured to assess changes in surface wettability using the OCA15EC (DataPhysics, Germany), a video-based optical contact angle measuring system. Five different spots were measured on the surface of the lotus fabric samples, and the mean contact angle value was calculated and reported. Measurements were recorded both before and after the plasma treatment.

2.3 Tensile Strength

The tensile strength of lotus fibres was measured by ASTM D3822, both before and after plasma treatment, to determine any changes in mechanical integrity resulting from surface modification.

2.4 Neem Extract Preparation and Antimicrobial Treatment

Fresh neem leaves were collected from the campus of Manipal University Jaipur. An aqueous-methanolic neem leaf extract was prepared and applied to the lotus fabric samples for antimicrobial functionality. Untreated and plasma-treated fabric samples were immersed in the extract at a material-to-liquor ratio of 1:20 for 30 min to impart an antibacterial finish.

The antibacterial activity was examined using the Agar diffusion test (AATCC Test 90-2011) and modified Hohenstein test (JIS L 1902). The tests were performed against *S. aureus* and *E. coli* to determine antibacterial efficacy.

2.5 Wash Fastness

The durability of the antimicrobial finish was assessed through wash fastness testing as per AATCC Test Method 124-2009. Fabric samples were tested after 1, 2, and 3 washing cycles, and antimicrobial retention was recorded.

2.6 Surface Analysis

The chemical structure and functional group changes on lotus fabric surfaces were analysed using Attenuated Total Reflectance–Fourier Transform Infrared Spectroscopy (ATR-FTIR) with an Alpha spectrophotometer (Bruker, Germany). Surface morphology was examined using a scanning electron microscope, the Nova Nano FE-SEM 450 (FEI, USA). The shape and roughness of the lotus fibre surface as a result of the plasma treatment were examined using the NT-MDT NTEGRA AFM system.

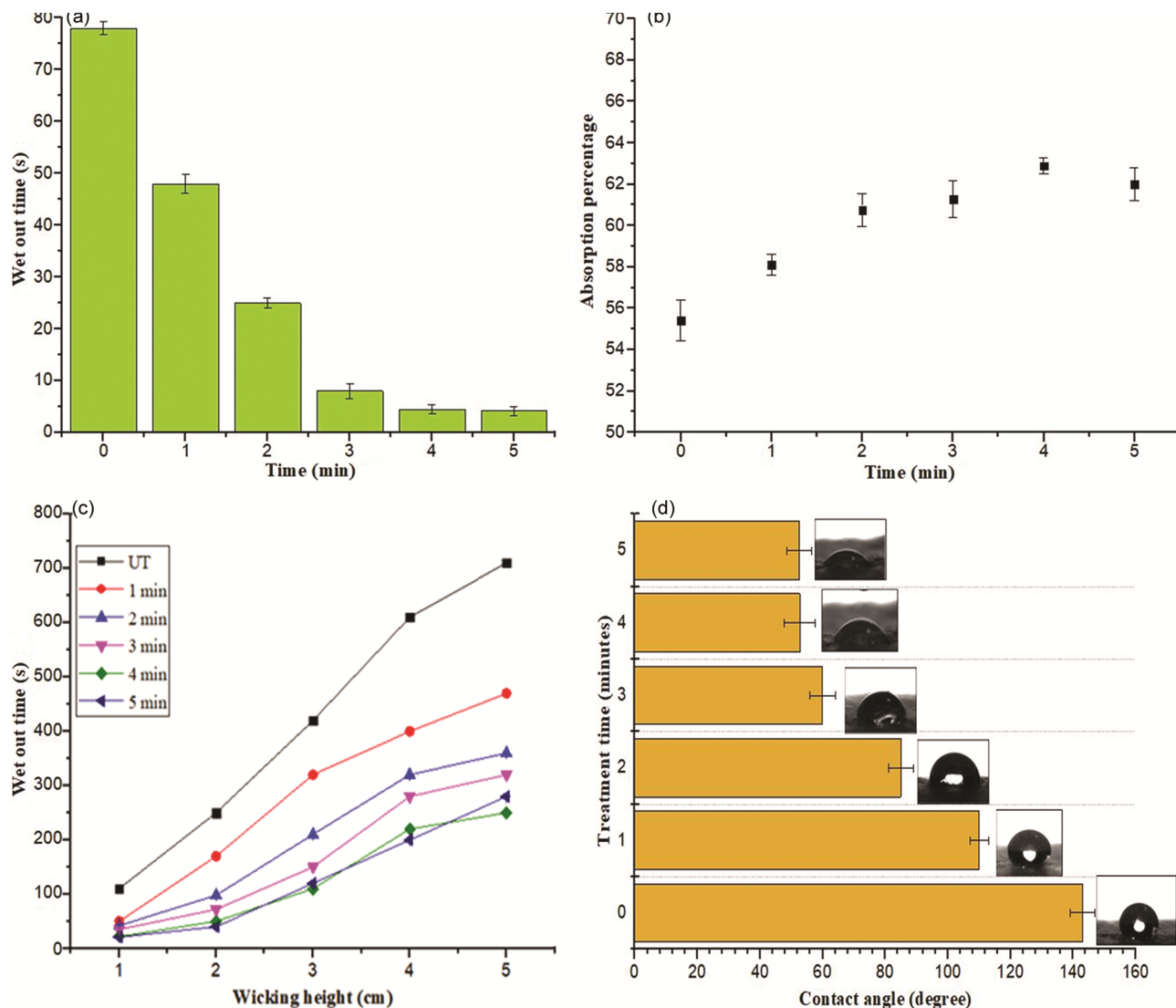


Fig. 1 — (a) Wet out time, (b) absorption percentage, (c) wicking rate, and (d) static contact angle measurement of untreated and plasma-treated lotus fabric

3 Results and Discussion

3.1 Change in Surface Hydrophilicity after Plasma Treatment

The wettability of plasma-processed lotus fabric is investigated using wet-out time, wicking test, and water contact angle analysis before and after plasma treatment. The wet-out time is evaluated at three distinct places on each sample. Figure 1 (a) demonstrates a significant increase in water absorbency as the plasma exposure duration increases. The untreated sample took 78 s to absorb 0.1 mL of water droplet, whereas the sample exposed to 1 min plasma treatment took only 48 s. The wet-out time further decreases to 4.5 s after 4 min of plasma exposure. This reduction is attributed to the removal of hydrophobic impurities and the production of

oxygen-containing hydrophilic groups, which enhance surface wettability. However, increasing the treatment beyond 4 min may cause severe deformation of the fibre strands, as observed in the SEM images.

The hydrophilicity analysis of untreated and plasma-treated fabrics with various treatment durations is shown in Fig. 1 (b). A gradual rise in hydrophilicity is observed from 1 to 5 min of exposure in air plasma, with a maximum at 4 min. As a result of free-radical species, including ions and functional groups (-OH, -COOH) generated in plasma reacting with the cellulose layer of the fabric, the hydroxyl groups of cellulosic fabric are oxidised into aldehydes and further into carboxylic acids, making it highly reactive. The fabric's hydrophilicity increases at 4 min of plasma treatment as carboxylic acids have a stronger polarity than hydroxyl

groups. In a similar fashion, a steady increase in the absorption percentage from 55.42% (untreated) to 62.89% is observed after 4 min of plasma exposure. This indicates that when the treatment time is increased, more number of activated plasma species react with the fabric surface for a longer period, resulting in efficient etching of impurities such as non-cellulosic components²⁴. Excessive etching occurs when the treatment period exceeds 4 min, causing fibrillation, making the surface rough and uneven, preventing water from spreading and reducing the absorption efficiency^{25,26}.

The dynamic wicking test shows that plasma treatment significantly improves the capillary action of the fabric [Fig. 1 (c)]. The wicking rate of the fabrics increases considerably after the plasma treatment. The untreated sample takes 710.30 s to reach a height of 5 cm, while the 4 min plasma-treated sample reaches the same height in 250.6 s. This improvement could be due to two factors: first, chemical changes on the fabric surface caused by the formation of polar hydrophilic groups such as –OH, –COOH, and free radicals, and second, physical changes on the fibres caused by the etching effect of impinging plasma species, which result in the removal of non-cellulosic impurities²⁷. The ablation action was induced by bombarding the plasma species on the surface, as well as the anchoring of oxygen-containing polar groups, enhancing the wettability. Furthermore, plasma treatment improves the mean pore size of the fabric structure, decreasing capillary pressure and improving wickability, according to previous literature. This might be due to a change in the shape and size distribution of the capillary space between fibres and yarns, leading to higher capillary velocity. Higher wettability resulted in increased wicking of the fabric, as previously stated because a fabric that is not moist cannot wick into the capillary region^{28–31}. Furthermore, several studies indicate that plasma treatment increases the mean pore size of independent fibres, lowering the capillary pressure and improving fabric wickability^{15,32}. Fig. 1 (c) further reveals that, compared to untreated fabric, which took 110.5 s to move to a height of 1 cm, the 4 min plasma-treated sample took 22.01 s²⁴.

Figure 1 (d) shows a significant reduction in the contact angle of plasma-exposed lotus fabric. After 4 min of plasma treatment, the water contact angle reduces from 143.67° (untreated) to 52.72°. The generation of polar functional groups such as –OH, –COOH, and –C=O on the lotus fabric surface due to oxidation by air plasma and an increase in

microporosity might explain the reduction in the water contact angle^{27–30}. As seen in XPS spectra, a decrease in the contact angle is associated with a reduction in carbon content and C-C/C-H units and a concomitant increase in the oxygen content. Several investigations have demonstrated that air DBD-treated materials considerably improve hydrophilicity^{31–34}. The dramatic change in wettability of the treated fabrics may be explained by the increased degree of ionisation of plasma species, which would contribute to the cleavage of the bigger and heavier molecules of the waxy layer exposed on the surface.

3.2 Change in Surface Morphology after Plasma Treatment

Figure 2 (a) and (b) show SEM images of untreated and plasma-treated lotus fibres. The untreated fibres display a smooth exterior except for a few contaminants, like lignin, pectin, etc. as seen in figure 2a. Following 4 min of plasma treatment, the fibres' surface becomes cleaner with longitudinal flutes and increased spacing between fibres due to the removal of impurities. Removal of impurities creates new pathways for water penetration, eventually enhancing the fabric's absorption capacity. Etching by active species generated in plasma is the primary cause of erosion of impurities. The interaction of plasma electrons and ions with substrate molecules, as well as certain oxidative processes with activated oxygen atoms may have induced the etching action by atmospheric plasma^{35,36}. All the experiments have established that only the amorphous component erodes since it is loosely linked^{28,37–38}.

AFM results in Figure 2 (c) and (d) further reveal changes in nanoroughness after plasma exposure. For each sample, AFM scans were performed in a 2 µm × 2 µm area with a magnification of 500 nm/division, and the findings are presented in a three-dimensional (3D) plot with section analyses. The untreated sample has an average surface roughness of 9.01 nm, while the treated fabric demonstrates a significant increase in nanoroughness (10.25 nm) due to contact with plasma ions, which was measured using section analysis software. These changes also strongly agree with the SEM results and the etching phenomenon of air DBD plasma that led to the formation of the valley-like structures on the otherwise planar surface³⁹. The surface conditions are of utmost importance with regard to the antimicrobial adhesion property of the polymeric substances as they encourage stronger binding of plant extracts to the fabric⁴⁰. As documented and examined by numerous researchers,

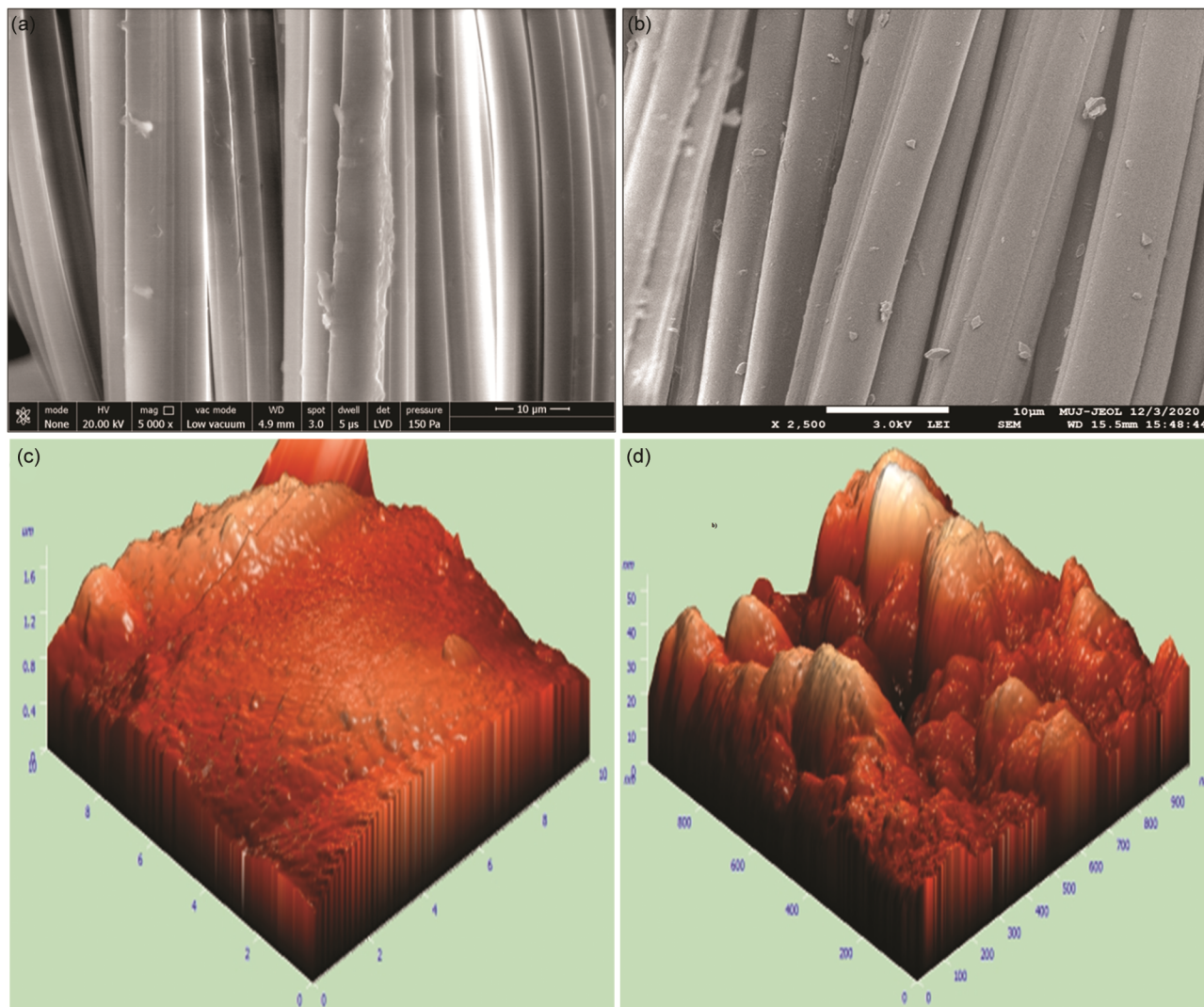


Fig. 2 — SEM images of (a) untreated and (b) plasma-treated lotus fabric at 2500x magnification; AFM images of (c) untreated and (d) plasma-treated lotus fabric

the changes seen on the plasma-treated surface are attributable to the etching action of plasma ions, which eliminates surface impurities and improves the average roughness¹². Thus, it can be concluded that applied plasma ions attack the surface of the cellulosic fabric, causing the non-cellulosic components to be removed and the surface to be etched, thereby exposing each fibre and causing an increase in the surface area and eventually an increased average roughness^{12,15}.

3.3 Change in Surface Chemistry after Plasma Treatment

Fig. 3 (a) displays the FTIR spectra of untreated and plasma-treated samples at a constant voltage of 3 kV. The lotus fabric structural skeleton is composed of cellulose, and the rest of the part is occupied by

hemicellulose, lignin, fats and waxes, pectin, water-soluble minerals and proteins. Cellulose is a highly crystalline material made up of glucose repeat units, whereas hemicellulose, wax, and pectin are amorphous in nature¹². Since both the untreated and plasma-treated samples are cellulosic in origin and have identical chemical compositions, they display almost comparable peaks, with the only variation being their intensities²⁴. The FTIR spectrum of lotus fabric demonstrates a similar pattern of peaks, which is also shown in cellulosic fabric. The peaks in the region 3100 to 3700 cm^{-1} correspond to OH stretching vibration. This provides considerable information on the hydrogen band related to the scission of intra and inter-molecular hydrogen bonds¹². The peak at 3851 cm^{-1} relates to the bending vibration of OH

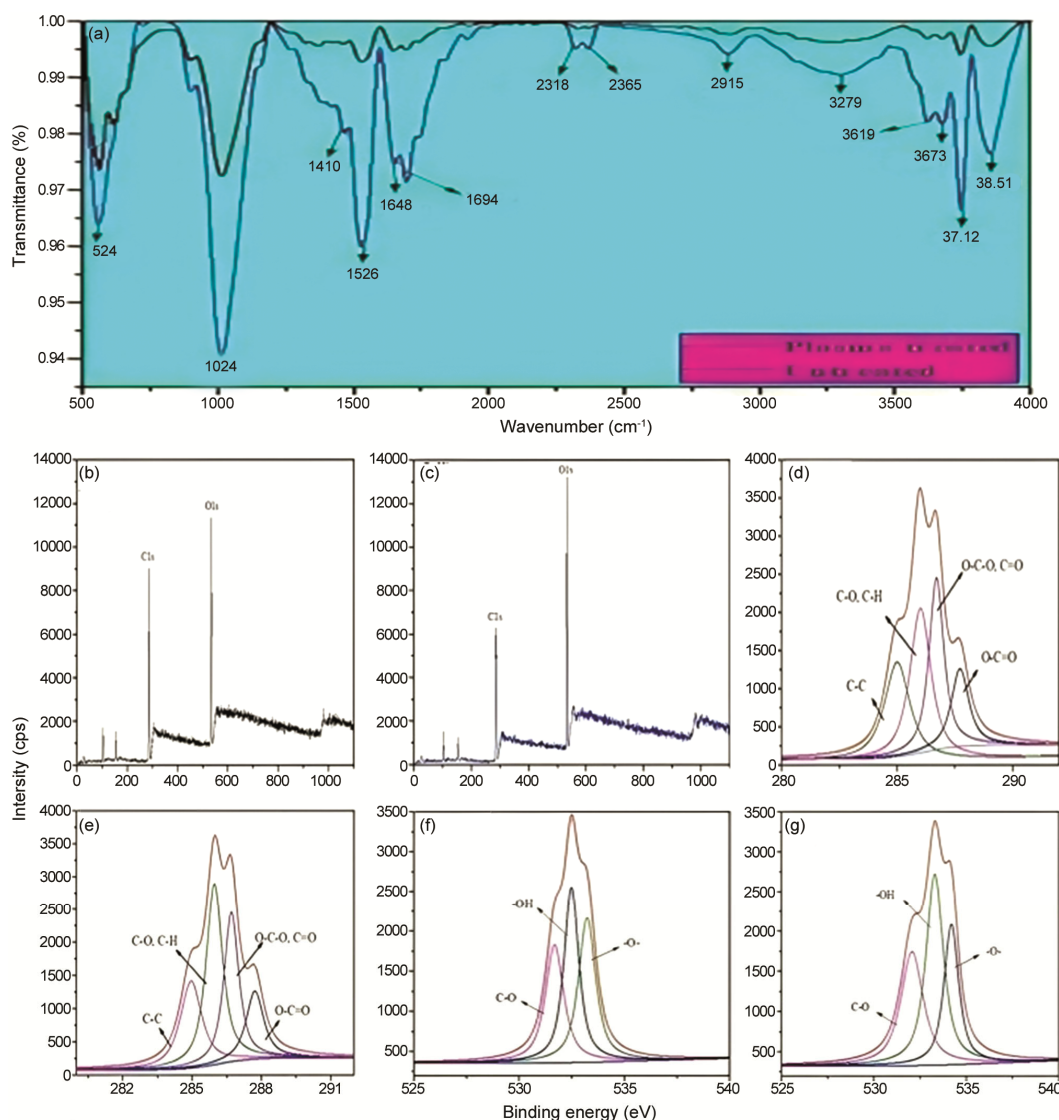


Fig. 3 — (a) FTIR spectra of untreated and plasma-treated lotus fabric; XPS survey spectra of (b) untreated and (c) air plasma-treated lotus fabric; C 1s peaks of (d) untreated and (e) air plasma-treated lotus fabric; O1s peaks of (f) untreated and (g) air plasma-treated lotus fabric

groups, which leads to improved hydrophilicity of fabric. The peak at 664 cm^{-1} in Fig. 3 (b) represents the OH out-of-plane bending mode, which, according to the literature, has a direct association with cellulose content and crystallinity % in lotus fibre. Similarly, the C-H bending vibrations and C-H stretching mode peaks are observed at 1322 cm^{-1} and 2889 cm^{-1} , respectively, which is related to the indirect estimation of the % crystallinity of the cellulose molecule. As the plasma treatment period increased, the strength of all three peaks intensified, indicating an increase in the crystalline percentage and elimination of amorphous molecules¹⁵. C-O stretching mode is involved for the peak area observed at 1027 cm^{-1} . Due

to the combined impact of the C=O and C-O-C groups, we obtain a wide peak with two minor split peaks because the two peaks are so closely located. The strengthening of these peaks post the plasma treatment indicates that the concentration of carbonyl groups has increased, which explains the dramatic increase in hydrophilicity of these fabrics along with improved plant extract absorption. The peak at 1424 cm^{-1} also validates the crystalline cellulosic structure with CH_2 scissoring mode. The -OH stretching of the hydroxyl group of cellulose corresponds to a broad and strong peak at 3278 cm^{-1} , and the increasing intensity shows the attachment of additional polar groups upon plasma exposure¹⁸.

Figs 3 (b) – (g) illustrate the untreated and plasma treated lotus fabric's elemental surface composition. Carbon is the primary component of the cellulose fabric, followed by oxygen, as confirmed by the survey scan spectra in Figs 3 (b & c) and the computed atomic percentages. As seen in Table 1, the O/C ratio of the untreated fabric was found to be 0.79, which increased to 0.84 after plasma exposure. The deconvoluted graph of carbon and oxygen was used to calculate the relative concentration of various concentration groups. The rise in oxygen group suggests that bombardment with plasma-activated species causes the integration of oxygen-containing functional groups on the lotus fabric, increasing activated oxygen species concentration. Increased concentrations of polar oxygen species on the fabric surface can also explain the improved wettability upon the plasma treatment, generating strong bonds with water molecules^{14,41}.

3.4 Effect of Plasma Treatment on Antimicrobial Efficacy

The 4 min plasma-treated lotus fabric is chosen as the testing sample for antimicrobial assessment based on optimised findings from surface and wettability experiments. The magnitude of antimicrobial properties is assessed in terms of the zone of inhibition in a qualitative antimicrobial investigation

utilising the Agar plate technique. Even though both fabrics are treated with the same concentration of plant extract, the plasma treatment increases the antibacterial activity of the fabric relative to the untreated fabric [Fig. 4 (a)]. In addition, the zone of inhibition enlarged as the concentration of plant extracts increased. A similar result has been reported, as neem contains active compounds such as azadirachtin, quercetin, and nimbinin, which contribute significantly to its antibacterial properties. These findings have also been confirmed through phytochemical investigations²².

These antimicrobial agents bind to bacterial lipid bilayer cell membranes, causing damage to the bacterial cell walls and disintegration of cell membranes^{19,40-43}. The quantitative microbial reduction is assessed using the test technique described in the materials section, which involves counting the number of *S. aureus* and *E. coli* colonies. Evidently, the percent bacterial reduction on neem leaf extract-coated fabric increased by 3%-4% against *E. coli* and 5%-8% against *S. Aureus* post plasma treatment (Fig. 4c). The higher concentration of oxygen-containing surface functional groups of plasma-treated lotus fabric can be connected to these observations. Furthermore, removing non-cellulosic impurities enhances the fabric's hydrophilicity, leading to improved adsorption of phytoconstituents on

Table 1 — XPS survey scan and elemental composition of untreated and plasma-treated lotus fabric

	Carbon content %, 284.7 eV				Oxygen content %, 532.4 eV		O/C ratio
Untreated	54				43		0.79
Plasma-treated	53.8				45.4		0.84
	Elemental carbon content				Elemental oxygen content		
	C-C	C-O/C-OH	C=O/O-C-O	O-C=O	=O-	-O-	-OH
Untreated	22	30.1	25	12	25	35	25
Plasma-treated	19.2	35.2	29	18	32	46	30

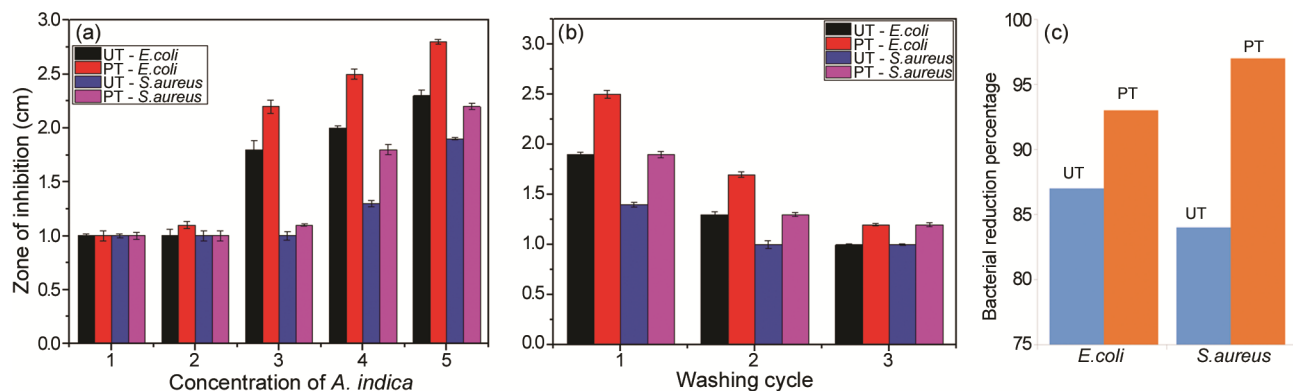


Fig. 4 — (a) Effect of *A. indica* leaf extract concentration on antimicrobial activity of untreated (UT) and 4 min plasma-treated (PT) lotus fabric against *E. coli* and *S. aureus*; and (b) effect of washing cycles on the antimicrobial performance of *A. indica* on untreated (UT) and 4 min plasma-treated (PT) lotus fabric and (c) bacterial reduction percentage in untreated and plasma-treated samples

the fabric surface, resulting in microbe cell lysis. Compared to *S. aureus*, the antibacterial effect of the treated fabric is much better against *E. coli*.

The wash fastness test (AATCC 124-2009) assesses the antimicrobial finish's durability. Fig. 4 (b) shows the antibacterial activity in terms of the zone of inhibition after 1 to 3 washing cycles. The inhibitory zone for untreated fabric became minimal after three washing cycles, while plasma-treated fabric preserved roughly 75 % of its antimicrobial activity. This might be due to the plant extract's active components being lost significantly after washing due to poor covalent binding or cross-linking with the untreated fabric. The plasma-treated fabric, on the other hand, demonstrates an improvement in antimicrobial finish uptake and penetration due to an increase in surface roughness and oxygen and nitrogen content, as observed in XPS, AFM, and FTIR analyses^{31,39}. In aqueous and alcoholic extracts of neem leaves, numerous scientific studies have documented the presence of alkaloids, terpenoids, flavonoids, tannins, and phenolic compounds with proven antibacterial and pharmacological activity⁴¹⁻⁴⁴.

FTIR analysis of neem extract alone and in combination with citric acid is depicted in Fig. 5. A strong stretching band is observed at 3345 cm^{-1} that can be ascribed to the O-H stretching vibrational mode of the hydroxyl group present in alcohols as well as the N-H stretching vibrational mode of amines and amides in the neem extract. Another peak is observed at 1634 cm^{-1} , corresponding to the C=O

stretching of the carboxyl group in the neem extract. When citric acid is combined with neem extract, a small peak at 1715 cm^{-1} occurs, which might be attributable to the C=O stretching vibrations of the acids in the citric acid molecule.

Peaks in the 1200 cm^{-1} - 1000 cm^{-1} region confirm the reaction of citric acid with neem extract. The C-H bending vibrations are represented by a weak peak in all spectra in the region of 1402 cm^{-1} - 1454 cm^{-1} . In addition, aromatic alcoholic groups exhibit C-O stretching vibrations in the region of 1044 cm^{-1} - 1030 cm^{-1} . The FTIR spectra of untreated and plasma-treated fabrics coated with neem extracts are shown in Fig. 5. The carboxylic acids of citric acid molecules interacted with the carboxylic groups generated on the lotus fabric following plasma exposure, resulting in the formation of acid anhydride when the lotus fabric was coated with an antimicrobial finish, as observed in Fig. 6. This phenomenon can be observed by the presence of peaks at 1753 cm^{-1} and 1691 cm^{-1} ascribing to the C=O stretching vibration and peak at 1140 cm^{-1} owing to the C-O stretching of anhydride molecule. Because the acid anhydride generated is not water-soluble, unlike acidic or hydroxyl groups, it offers durability to the treated fabric^{19,33}. The C-O stretching of aryl alkyl ethers in neem extract is responsible for the peak at 1221 cm^{-1} .

The potential interaction is depicted in Fig. 6 between the functional groups of cellulose exposed to the surface after the plasma treatment of lotus fabric and imparting antimicrobial agent molecules. Due to

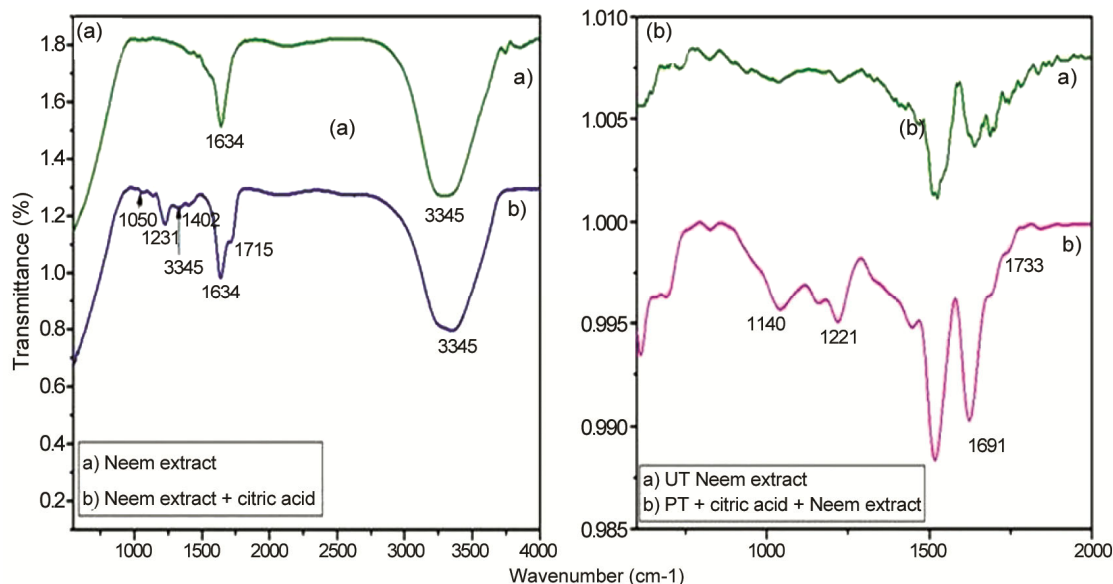


Fig. 5 — FTIR spectra of (a) crude neem leaf extract and neem extract with citric acid and (b) untreated (UT) fabric coated with crude neem extract and plasma-treated fabric coated with neem extract containing citric acid

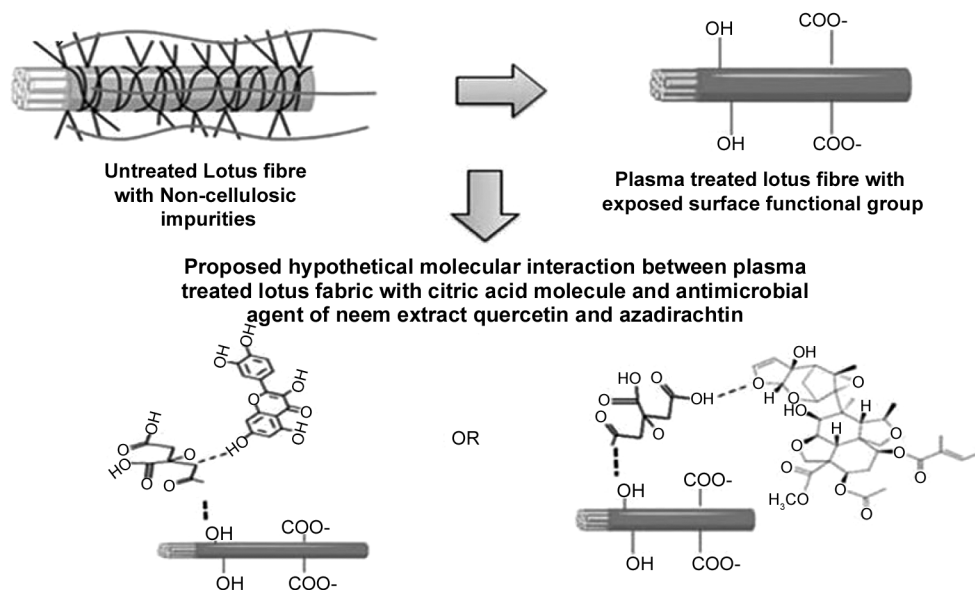


Fig. 6 — Proposed hypothetical molecular interaction between plasma-treated lotus fabric with citric acid molecule and antimicrobial agent of neem extract quercetin and azadirachtin

the plasma treatment of lotus fabric, hydrophobic non-cellulosic impurities attached to amorphous cellulose were removed, and the fibre surface became hydrophilic with exposure to the functional group of cellulose. This change in surface properties results in better adsorption of antimicrobial agents on the lotus fabric due to the hydrogen bonding between the antimicrobial agent, citric acid and various exposed functional groups on the lotus fibre surface, resulting in improved antimicrobial adsorption and attachment on plasma-treated lotus fabric^{19,33}.

4 Conclusion

In the current study, plasma treatment has been demonstrated to have a pronounced impact on the physical and chemical structures of the lotus fabric. The impact of parameters such as treatment time is studied using experiments such as wet-out time, weight loss, as well as hydrophilicity tests. These experiments reveal that DBD plasma treatment for 4 min considerably modifies the surface of the lotus fabric. After the generation of cellulosic radicals, DBD plasma treatment increases the concentration of carbonyl groups, enhancing hydrophilicity. The increase in the strength of polar groups following plasma treatment is confirmed by FTIR analysis. The O/C ratio increased after the treatment. Higher concentrations of herbal compounds (azadirachtin and quercetin) bond to the lotus fabric surface due to these chemical and physical modifications, providing

better antibacterial action to the plasma-treated fabric. The current study has demonstrated that DBD air plasma treatment followed by neem leaf extract surface coating may improve the antimicrobial properties of lotus fabric in a sustainable and environmentally friendly manner.

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