

Blood Repellent And Antimicrobial Finishes For Cotton Hospital Fabrics

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Abstract : Blood repellant and antimicrobial finish has been applied to 100% cotton fabrics used for surgical gowns, bed linens and drapes to reduce the surgical site infections. The extract of cinnamon and clove oil was applied to the fabric for imparting antimicrobial activity by pad-dry-cure method. The cinnamon and clove oil treated fabric was then imparted blood repellency through two different techniques, namely by treatment with fluoro polymer (3%, 4% and 5% owf) using pad-dry-cure method and by 'sputter deposition of Teflon' technique using argon plasma. The antimicrobial activity is found to be higher for Teflon deposited fabric than for the fluoropolymer finished fabric. Blood repellency increases with the higher concentration of fluoropolymer and the highest repellency for the Teflon deposited fabric is observed at 80W power and 20 min exposure in the plasma chamber.

Keywords: Antimicrobial textiles, Blood repellency, Cotton, Fluoropolymer, cinnamon and clove oil, Sputter deposition, Teflon

1 Introduction

Many harmful, infectious and blood-borne bacteria and viruses, such as Pseudomonas, Caudida, S.aureus and E.coli, are present in hospital locations which are conducive for growth of the microorganisms. The textile materials used in hospitals, like operating theatre gowns, drapes, masks, sheets and pillow covers, which are frequently subjected to the human blood areknown to be major sources of cross-infection. Blood and body fluids are considered as the carriers of the several microorganisms and can be transferred through the barrier materials, like surgical fabrics, drapes and bed linens, by wicking of fluids or pressure or leaning on a flooded area of the product.In order to protect patients, hospital personnel andsurgical team from such cross-infections through textile materials with good resistant to microbial attack and cross-infection by giving antimicrobial and blood repellent finish. This dual finishing treatment apart from protecting the wearer safeguards the fabric from staining, discolorations and deterioration of

strength.

Various classes of chemicals have been introduced to impart water repellency to the fabrics. Out of them fluoro chemical polymers are prominently used as repellent finishing agent in industry as well as byresearchers. The surface tension of the fabrics has to be much lower than that of blood and body fluids whose surface tension ranges between 42 dynes/cm and 60 dynes/cm to produce blood repellency. The surface tension of fluorocarbon water repellant agent is 10 dynes/cm, which is lower than any other commonly used water repellents. Hence, it is used for imparting the blood repellency to the fabric.

Teflon also has surface tension lower than that of the blood. The week interaction of the C-F bonds and cotton decreases the surface tension and imparts bloodrepellency to the fabric.

Plasma treatment is one of the environment-friendly processes, which is used to alter the surface character of the textile materials without affecting their bulk properties. Plasma, as a very reactive material, can be used to modify the surface of a certain substrate, depositing chemical materials to impart some desired properties and removing substances which were previously deposited on the substrate.

In the present study, an attempt has been made to impart antimicrobial and blood repellency to 100% cotton fabric using methonalic extract of cinnamon and clove oil followed by treatment with fluoropolymer and Teflon coating using plasma sputtering equipment. Comparison has been made between the two methods in terms of their antimicrobial efficacy and blood repellency. Efforts have also been made to optimize the concentration of fluoropolymer to achieve better antimicrobial and blood repellent character on cotton fabric.

2 Materials and Methods

Fabric Preparation

100% cotton plain woven fabric, made up of 60s combed yarn with 130 ends/inch and 90 picks/inch, was desized (0.5% H₂SO₄), scoured (3% NaOH) and bleached (3% H₂O₂) prior to the application of the antimicrobial finish and blood repellent finish.

Cinnamon and Clove oil Extract Preparation

The freshly collected Cinnamon and Clove oil were taken. Methonalic extract of the cinnamon and clove treating with 100 ml of methanol at room temperature in an air tight flask to dissolve the active substance and kept for 12 h. After that the solution was filtered and the filtrate was used for the study.

Application of Antimicrobial Finish onto Fabrics

Methonalic extract of the active substances of cinnamon and clove oil wasapplied to the fabric by pad-dry-cure method. Thefabric was padded with the extract to attain a wetpick-up of 75%, dried and then cured at 100-120°C for 2 min. In order to fix the active cinnamon and clove oil substance on the fabric, a post treatment with 10 % citric acid was given, keeping material- to-liquor ratio of 1:20 at 50°C for 5 min. The treated fabric samples were then dried at 80°C and cured at 140° C for 2 min.

Application of Blood Repellent Finish

In this work, dual finishes of antimicrobial and blood repellency were imparted to the surgical gown and bed linen fabrics. The antimicrobial finished materials were post treated with fluoropolymer and Teflon separately by the following methods.

Fluoropolymer Treatment

The fluoropolymer Anthydrin FS, supplied by Zschimmer & Schwarz, was applied on the antimicrobial finished fabrics using the following recipe:

Fluoropolymer: 30g/L, 40g/L, 50g/LAcetic acid: 1g/LDMDHEU resin:10g/LMagnesium chloride :1g/L

The fabrics were treated separately with 3%, 4% and 5% fluoropolymer using the above recipe bypad-dry-cure method to attain a wet pick-up of 75%, dried at 90°C and cured at 120-130°C for 2 min.

Sputter Deposition of Teflon

Cinnamon and clove oil pretreated cotton fabric was cut into 10cm × 10cm size and deposited with Teflon using RF magnetron sputtering with argon as the sputtering gas. The sample was placed on the lower electrode which was grounded. The frequency of the RF system was13.56 MHz. The pressure of Ar and the distance between the electrodes were fixed as 0.008mbar and 5cm respectively. Teflon was deposited on the fabric for various periods of time and RF power to optimize the sputtering parameters.

Test Methods for Assessing the Antimicrobial Finish

In this work, the qualitative agar diffusion test and the quantitative bacteria reduction through Hohensteinmodified test were used to assess the antimicrobial activity of the fabrics.

Agar Diffusion Method (SN 195920)

Treated and untreated control fabric samples were placed in intimate contact with AATCC

bacteriostasisagar, which was previously inoculated with a day culture (slant cultures) of the test organisms, i.e. Staphylococcus aureus and Escherichia coli. After incubation, it was assessed by visual examination as well as under a microscope (× 40 magnification). The evaluation was made on the basis of absence or presence of an effect of bacteria in the contact zone under the specimen and the possible formation of a zone of inhibition around the test specimen. The area of inhibition zone is a measure of antimicrobial effectiveness.

Hohenstein Modified Challenge Test (JIS L 1902)

Specimens of the test material were shaken in a known concentration of bacterial suspension and the reduction in bacterial activity in standard time was measured. The efficiency of the antimicrobial treatment was determined by comparing the reduction in bacterial concentration of the treated sample with that of the control sample expressed as a percentage reduction in standard time. Staphylococcus aureus (ATCC 6538) was used as a representative Gram positive organism and Escherichia coli (ATCC 11230) was used as a representative Gram negative organism. The bacterial counts were reported as the number of bacteria per sample (swatches in jar) not as the number of bacteria per ml of neutralizing solution. '0' counts at 10^o dilution was reported as "less than 100". The % reduction of bacteria by the specimen treatments was calculated using the following formula:

100 (B-A) / B = R

where, R is the % reduction; A, the number of bacteriarecovered from the inoculated treated test specimen swatches in the jar incubated over the desired contact period; and B, the number of bacteria recovered from the inoculated treated test specimen swatches in the jar immediately after inoculation (at '0' contact time). The % reduction of bacteria by the specimen treatment against each test organism was reported.

Test Methods for Assessing Blood Repellent Finish

The blood repellency of the sample was assessed using impact penetration and spray test. The synthetic blood was prepared using distilled water, a surfactant (Acrysol G110, Rohm and Hass Co) and red dye (Direct Red 081) according to ASTM F23.40.01(draft) for testing the resistance of protective clothing material to synthetic blood.

Impact Penetration Test (AATCC 42 -2000)

A volume of water/synthetic blood was allowed to spray against a taut surface of the test specimen backed by a weighed blotter. The blotter was then reweighed to determine water penetration and the specimen is classified accordingly. The specimen 178

 \times 330mm and the blotting paper were conditioned in an atmosphere of 65± 2 % RH and 2 1 °C temperature for at least 4 h before testing. The increase in mass of the blotter in grams was

calculated and the average result of the three test specimens was reported.

Spray Test (AATCC 22 -1996)

Water sprayed against the taut surface of a test specimen under control conditions produces wetted pattern whose size depends on the relative repellency of the fabric. Specimen of 18×18 cm size was conditioned at 65 ± 2 RH and $21^{\circ} \pm 1^{\circ}$ C. Evaluation is accomplished by comparing the wetted pattern with the observations as mentioned in the followingstandard rating chart:

Standard Observationrating

100 (ISO-5) - Sticking or wetting of upper surface
90 (ISO-4) - Slight random sticking or wetting of upper surface 80(ISO-3) -Wetting of upper surface of spray points
70(ISO-2) - Dential surface of surface a first set of the upper surface 50(ISO-1). Complete surface of spray points

70(ISO-2)- Partial wetting of whole of the upper surface50(ISO-1)- Complete wetting of whole of upper surface

0 - Complete wetting of whole upper and lower surfaces

Wettability of Fabrics (BS 4554)

In this test, the time taken for the absorption of water drop completely in the fabric was used to analyze the hydrophobic nature of fabric. The specimen was clamped circularly and held taught. A burette with a standard tip of 6mm was used to drop the water. The fabric was illuminated at an angle of 45°C and then viewed from the opposite direction so that any water on the surface reflects the light to the viewer. At the start of the test, a drop of water was allowed to fall from the burette and the timer was started. When the diffuse reflection from the liquid vanishes and the liquid is no longer visible, the timing is stopped and recorded. If the time exceeds 200 s, the sample is considered to be unwettable.

Assessing the Morphology of Fabrics

Morphology of the samples treated with fluoropolymer and Teflon coated under plasma environment was studied to find out the effectiveness and uniformity of the finishing treatment on the surface of the fabric using scanning electron microscope (SEM).

3 Results and Discussion

Interaction of Cinnamon and clove oil Constituent with Cellulose

Azadirachtin, a tetranortriterpenoid with molecular formula $C_{35}H_{44}O_{16}$ has been identified as an active substance of Cinnamon and clove oil. It is an insect antifeedent and ecdysis inhibitor. It contains large number of functional groups and is sensitive to acids, bases and UV light. The

cotton fabric treated with the Cinnamon and clove oil shows very good resistance to both Gram positive and Gram negative bacteria but the durability is found to be very less. Preliminary efforts have beenmade to study the reaction between cellulose and Cinnamon and clove oil through the FTIR spectra of untreated and Cinnamon and clove oil treated cotton fabrics. From the results, we could not establish any type of possible chemical reaction between Cinnamon and clove oil extract and cotton. Hence, it is postulated that week cross linking might have beentaken place between them which is confirmed by the very low durability of the finish. However, furtherexperiments have to be done with large number of samples to find out the exact chemical or physical interaction of neem extract with cotton. The durability of the finish was improved by further treating thematerial with citric acid, a known cross linking agent for cellulose. This treatment was given, assuming that the applied neem chemical could be trapped between cellulose and citric acid to some extent and slowly be released during actual usage.

Comparison of Antimicrobial Efficacy of Cinnamon and clove oil Treated, Fluoropolymer Finished and Teflon Finished Fabrics

Figure1 shows the result of agar diffusion test for antimicrobial effectiveness against Staphylococcus aureus and E.coli cultures. Specimen 1 represents Cinnamon and clove oil treated fluoropolymer finished sample and specimen 2 represents the Cinnamon and clove oil treated Teflon finished sample. The zone of inhibition for Teflon finished fabric is higher compared to fluoropolymer treated sample. The antibacterial activity of the Cinnamon and clove oil treated fluoropolymer finished samples at 3 different concentrations of fluoropolymer and Teflon deposited samples based on agar diffusion and Hohenstein modified method is given in Table 1. It can be inferred that the antimicrobial efficacy reduces apparently with the increase in fluoropolymer concentration.

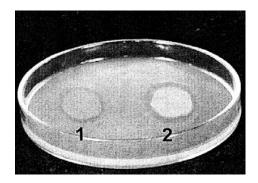


Fig. 1 — Antimicrobial efficacy of Cinnamon and clove oil treated (1) fluoropolymer finished and (2) Teflon finished fabrics (Zone of inhibition by agar diffusion test)

This may be due to the increase in release of Cinnamon and clove oil active substance as well as the restriction formed by the hydrophobic fluoropolymer finish for the growth of microorganisms

on the fabric surface. Teflon deposited fabric shows higher antimicrobial efficacy as compared to fluoropolymer treated fabric. This might be due to the uniform deposition of Teflon on cotton fabric in the plasma environment, forming an effective hydrophobic and blood repellent surface which is partly permeable to active neem substrate. Earlier work in this subject by Jeong-Sook and Cilsoo Choo also confirms the formation of blood repellent surface without hindering the release of active neem substances. add-on of fluoropolymer, especially the higher concentration makes the fabric surface highly hydrophobic and considerably restricts the release of herbal antimicrobials. In general, the bacterial reduction percentage of treated fabrics is higher in comparison to the control fabrics mainly due to the

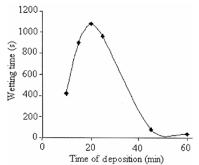
Effect of Fluoropolymer Treatment on Blood Repellency

It is observed that the amount of synthetic blood penetrating the sample is reduced with the increase in fluoropolymer concentration (Table 2). The spraying rate also improves with the increase in fluorocarbon concentration. It may be concluded that the optimum concentration of fluoropolymer is 4% add-on of fluoropolymer; especially the higher concentration makes the fabric surface highly hydrophobic and considerably restricts the release of herbal antimicrobials. In general, the bacterial reduction percentage of treated fabrics is higher incomparison to the control fabrics mainly due to the get better antimicrobial as well as blood repellent characteristics.

Effect of Sputter Deposition of Teflon on Blood Repellency

In case of the sputter deposition of Teflon, the deposition time and RF power are optimized in terms





Sample treated with	S. aureus	E.coli	S. aureus	E.coli
with	96.5	94	43	41
Cinnamon and clove oil (control)				
Cinnamon and	85	85.2	34	33
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Fabrics				

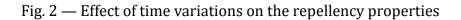
clove oil and 3%				
fluoropolymer				
Cinnamon and	88.9	80.1	39	34
clove oil and 4%				
fluoropolymer				
Cinnamon and	87.2	86.8	39	31
clove oil and 5%				
fluoropolymer				
Cinnamon and	90	90.5	48	40
clove oil and Teflon				
deposition				

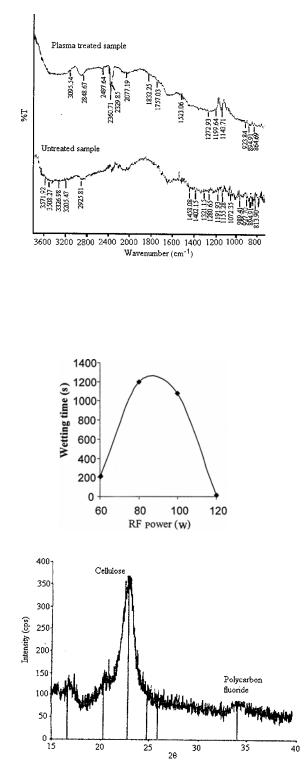
Table 2 — Effect of fluoropolymer treatment on bloodrepellency

Sample	Blood penetrationg	Spray rating
Untreated	25.98	0
Treated with		
fluoropolymer		
3%	12	52
4%	7	77
5%	8	88

of wetting time which indicates the blood repellency. It is found that the maximum blood repellency is imparted for a deposition time of 20 min (Fig. 2). The effect of power variation on the repellency characteristics of the fabrics seems to attain amaximum level at 80W (Fig. 3).

Table 3 depicts the results of spray test. It confirms that the sputter deposition of teflon on cotton fabrics imparts blood repellency. This is due to the argonplasma which is used during the sputtering to perform two functions. Carbonyl groups as well as free radicals are formed on the surface of the cotton fabric due to the irradiation of the fabric by Ar plasma. Ar plasma sputters fragments of CF1, CF2, and CF3 from the Teflon target which deposits C-F fragments on the fabrics. The C-F fragments, which form a cross linking with the carbonyl group or free radical or the hydroxyl group in the sample, exhibit non-polarity and thus impart blood repellency to the sample. The fact that the argon plasma treated cotton fabric contains more carbonyl ($\mathbb{Z}C=O$) groups due to the formation of aldehyde or carboxylic acid functional group has been established by the FT-IR





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Fig. 3 — Effect of power variations on the repellency properties

analysis carried out on the untreated and treated samples (Fig.4). The absorptions of the untreated sample are in the regions of 3200-3600 cm^{\square 1} and 1000-1500 cm^{\square 1}, confirming the presence of the –OH functional group. The absence of absorption in the range of 1600-1900 cm^{\square 1} confirms the absence of carbonyl (\square C=O) groups. The FT-IR spectrum of the plasma treated sample shows the absorptions in the region of 1600-1900 cm^{\square 1} and 2300-2900 cm^{\square 1}, confirming the presence of carbonyl (\square C=O) groups⁹ in addition to –OH related peaks in the regions of 3200 - 3600 cm^{\square 1} and 1000-1500 cm^{\square 1}. X-ray diffraction analysis was also made on the treated and untreated samples to confirm the presence of C-F bond. It was carried out using X-ray diffractometer (Shimadzu-600X) where the Ni monochromator filters the K_β rays from the X-ray tube (CuK α , λ =1.542Å). Data were collected in the range of 2 θ = 20^o-80^o. The X-ray diffractogram of the untreated and PTFE sputter deposited samples are

PowerW	Timemin	Electrode distance cm	Spray rating
60	20	5	50
80	20	5	70
100	20	5	70
100	15	5	50

Table 3 — Effect of sputter deposition of Teflon on blood repellency

Fig. 4 — FT-IR spectrum of plasma treated and untreated samples

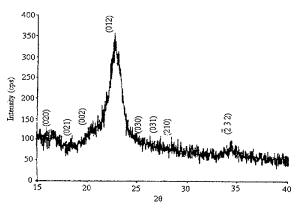


Fig. 5 — X-ray diffractogram of untreated cotton sampl

shown in Figs 5 and 6. The X-ray diffractogram of the untreated sample reveals the strongest peak for a plane of (012) and the corresponding d value is 3.90Å. This matches with the JCPDS (Joint

committee on power diffraction standards) of cellulose (Card No: 03-0226), thus confirming the presence of cellulose in the untreated sample. TheXRD spectra of teflon sputter deposited sample show the first three strongest peaks with d values corresponding to 3.90Å, 4.31 Å and 5.91Å of which the first two matches with the JCPDS of cellulose (Card No: 03-0226) and the third with polycarbon fluoride (card No: 27-1873). This confirms the presence of C-F bonds in the PTFE deposited sample.

Comparison of Blood Repellency Effect of Teflon Deposited & Fluoropolymer Finished Fabrics

The blood repellency effect of the finished fabrics was compared using the spray test because impactpenetration test could not be carried out due to smaller

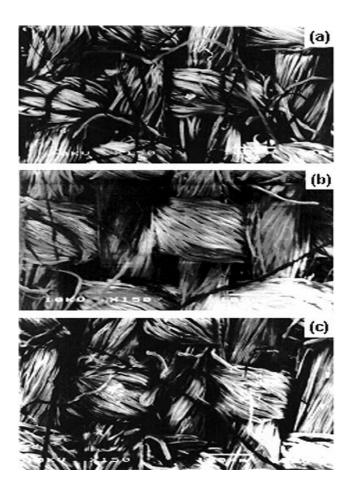


Fig. 7 — Scanning electron micrographs of (a) neem treated control, (b) fluoropolymer treated, and (c) teflon treated samplesdimensions of teflon treated fabrics. The highest rating of the fluoropolymer treated cotton fabrics is found to be 80 and that of the teflon deposited samples is 70. It is inferred from Tables 2 and 3 that the spray rating of the 4% fluoropolymer treated sample

is same as that of the teflon finished sample. The absorbency time for the fluropolymer treated fabrics at 5% (owf) concentrations is 2400s and that for the teflon finished samples is 1200s at 80W for

20 min with Ar pressure of 0.008mbar and 5 cm electrode gap.

Morphology of Samples

The scanning electron micrographs of the neem pretreated fluoropolymer and Teflon coated in plasma environment fabrics are analyzed to study the surface level modifications of the samples due to finishing treatments. Figure 7a shows the surface of the control unfinished sample having open weave structure with more number of loose protruding fibres. Figure 7b shows the reduced pore size in the weave structuredue to the deposition of fluoropolymer but there is no appreciable reduction in number of protruding fibres. In the case of Teflon treated fabric (Fig. 7c), the pore size in fabric surface still gets reduced due to theuniform deposition of Teflon under plasma environment. The Teflon coated surface is also smooth without any loose protruding fibres. This may be due to the formation of better cross linking between the active carbonyl group or free radical or hydroxyl group in the sample with C-F fragment of Teflon under plasma environment.

4 Conclusions

The antimicrobial efficacy of the fluoropolymer treated fabric reduces with the increase in fluoropolymer concentration but the blood repellency increases.

The antimicrobial efficacy and blood repellency of the sputter deposited Teflon fabrics is found to be better than the fluoropolymer treated fabrics.

The highest repellency is observed with the fabrics subjected to 80W power and 20 min exposure.

The increased blood repellency of the Teflon coated sample may be due better cross linking between carbonyl groups (-C=O) created by Ar plasma treatment or hydroxyl groups of the cotton and C-F fragments of the Teflon. FT-IR and XRD analyses carried out on the untreated, plasma treated and Teflon coated samples confirm the above theory.

SEM analysis shows that the deposition of teflon under plasma is very uniform and effective compared to the fluoropolymer treatment. The surfacepore size of the teflon treated sample is more closed and also with less numbers of loose protruding fibres compared to the fluo

ropolymer treated sample.

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