

Original Research Article

<http://dx.doi.org/10.20546/ijcmas.2016.508.048>

**Effective Optimization and Evaluation of Air Plasma Treatment on the Surface of 100% Bleached Cotton Fabric for Better Dabbing of Antibacterial Finishes**

**Punam Sen\* and R. Rajendran**

Department of Microbiology, PSG College of Arts and Science, Coimbatore-641014, India

\*Corresponding author

**A B S T R A C T**

**Keywords**

Hydrophilic, antimicrobial, pathogen, Plasma, herbal plant extract.

**Article Info**

**Accepted:**

21 July 2016

**Available Online:**

10 August 2016

The vital biological systems are sensitive and may react to the environmental factors with adverse effects. And so are the microorganisms, which invade in textile fabrics with conducive contaminations and have challenged the healthy life style of mankind. Therefore, technological boon has brought about an aspect of applying air plasma on fabrics for the development of hydrophilic property, which in turn can render effective antimicrobial finishes to the textile materials. An attempt for optimization of different parameters of air plasma was carried out in this study, in accordance with its absorption capacity for achieving a significant antibacterial coating on the fabric. Thus, antibacterial finishes would reduce the detrimental effect of pathogenic bacteria and provide a measure of safety to our day to day life activities. The methods employed here are AATCC methods and standard microbiological test methods. The herbal plant extract explored here for antibacterial coating are a combination of *Calendula officinalis* and *Azadirachta indica*.

**Introduction**

Microorganisms play a vital role in causing various infections in our body. The normal bacteria found on skins can invade our system and produce characteristic foul odour due to the degradation of sweat components, especially on the foot and armpits etc. So this microbes can easily create havoc any time anywhere, if they are not controlled. Clothing being an important need for human life, protects us from various infections and other external stimulus factors. Hence, functional textiles with an ideal antimicrobial finish can bring about an

effective, durable and compatible multi-dimensional biotextiles. Several studies showed that cotton- the king of fibres are more susceptible to microbial attack than synthetic materials (Ranganathan, 2005). This is due to the porous hydrophilic structure of the cotton fabric which has the ability to retain water, oxygen and nutrients (Gupta, 2001). Microorganisms can utilise such substrates and decompose the *cellulosic* fibre of the cotton fabric. Therefore, to avoid deleterious effects of microorganisms, efficient antimicrobial

dabbing is required for health and hygiene purposes.

Nature has bestowed with abundant medicinal herbs which could be a source of relief from infections (Mahesh *et al.*, 2008). A rich source of bioactive compounds in herbs can act as antimicrobial agent. A wide range of medicinal plants could be exploited for better antimicrobial finishes. Herbs like *Calendula officinalis*, *Azadirachta indica* are some of the plants having potent bioactive compounds which can render therapeutic mechanisms of controlling infectious pathogens (Punam *et al.*, 2014). *C.officinalis* is known to have wound healing and haemostatic activity (Mathur *et al.*, 2011) and *A. indica* is an excellent antiseptic.

Technology being a boon to mankind, adapted a cost effective and ecofriendly method of plasma application for the textile materials. Plasma treatment of material are designed to alter the surface character of the textile fabric without affecting their bulk properties (Rajpreet *et al.*, 2004). Plasma, the fourth state of matter was identified by Sir William Crookes, an English physicist in 1879 and named plasma by Irving Langmuir in 1928 (Samanta *et al.*, 2006). It was defined as “a partially or wholly ionized gas with a roughly equal numbers of positive and negative charged particles”. The treatment of such ionized gas will lead to surface erosion of cotton fabric, with a negligible weight loss accompanied by an increase in fibre carbonyl group (Malek *et al.*, 2003) As a result, fabrics hydrophilic property will be increased which in turn can help in absorbing the herbal extract for efficient antimicrobial dabbing. Literature revealed, not all plasma gas can bring about hydrophilic property, for example fluorocarbons can modify cotton fabric for hydrophobicity whereas air and oxygen can impart hydrophilicity. Hence, as per the

requirement of our work plasma gas could be applied for the surface modification of textile fabrics.

## **Materials and Methods**

The fabric used for this current study was procured from Aravind Mill, Kolkata, West Bengal, India. The specification of the fabric are given in table 1.

The Potent herbs selected for imparting antimicrobial activity are obtained from Kunjaban, Agartala, Tripura (w), India. The two important herbs exploited here for the study are *Calendula officinalis* and *Azadirachta indica*. The common names of the plant are marigold and neem. The parts of the plants used here are leaves for the extraction process.

## **Solvent Extraction of the Herbs**

Each 20gm of the powdered plant material was reflexed in a soxhlet apparatus in 100ml of the solvent (Methanol). After overnight incubation, the supernatant was filtered through Whatman no.1 filter paper and the filtrate was dried to evaporate the organic solvent at room temperature (Natarajan *et al.*, 2002). The filtrate solution was used as an extraction solution after the preliminary assessment of antimicrobial activity. Later, the combination of herbs were adopted for further investigations. Here different ratios like 1:1, 1:2, 1:3, 2:1, 2:2, 2:3 of the herbal extracts were used (w/w) for evaluating the highest antimicrobial property by using disc diffusion assay.

## **Method of plasma treatment**

The plasma treatment was imparted to the cotton fabric in the plasma unit which consist of two electrodes (the lower and the upper electrode).The air plasma treatment was given using Magnetron sputtering unit

(The system was evacuated to a pressure below 0.03 mbar before the introduction of air pressure. Then the system was connected to a power supply of a frequency of 13.56 MHZ (fixed parameter).The air was introduced into the unit through the inlet of the air pressure.

The air pressure was fixed as 0.05 mbar. The process parameters varied in the unit were electrode gap, power and time of exposure. The samples were determined subsequently by subjecting to static immersion test and the results are recorded in percentage (Vaideki *et al.*, 2007).

### **Fabric weight Loss**

Bone dry untreated cotton fabric samples were weighed on an electronic balance, subjected to air and oxygen plasma treatment, reweighed, and then washed at 60°C for 60 minutes in distilled water at a liquor ratio of 40:1. It was bone dried and reweighed. The weight loss was calculated as a percentage of the bone dry cotton fabric weight (Reza *et al.*, 2003).

$$\text{Percentage of weight loss} = (W_1 - W_2 / W_1) \times 100$$

Where  $W_1$  represents weight of the sample before plasma treatment and  $W_2$  is the weight of the sample after plasma treatment.

### **FTIR analysis**

The surface chemical composition of the cotton specimens (treated and untreated plasma samples) were studied by Fourier Transform Infrared spectrophotometer (Perkin Elmer Spectrum 100), with the scanning range between 4000  $\text{cm}^{-1}$  and 700  $\text{cm}^{-1}$  and the resolution of 4 $\text{nm}^{-1}$ , using the attenuated total reflection with 256 scans.

### **Static immersion test (BS 3449)**

The Static immersion test is a method for measuring the total amount of water, that a fabric can absorb. In this test weighed samples of the fabric was immersed in water for 20minutes at a depth of 10cms in a beaker filled with water. A sample of 10X10 cm size was used. After 20 minutes the sample was taken out and surface water was removed immediately by shaking it 10 times in a mechanical shaker. It was then transferred directly to preweighed airtight container and then reweighed.

The percentage of absorption by the fabric was calculated using the formula:

$$\text{Absorption} = \frac{\text{Mass of water absorbed}}{\text{Original Mass}} \times 100$$

Static immersion test was carried out with respect to all the varying parameters of the plasma treated samples and the maximum absorption value was recorded. Later the fabrics were subjected to antimicrobial finishes and the maximum percentage absorption fabric obtained from static immersion test was compared with maximum level of zone inhibition of inhibition for antimicrobial activity assessment.

### **Method of fabric finish**

The methanolic extract of combinatorial herbs (*Azadirachta indica* and *Calendula officinalis*) were prepared as an antimicrobial finishing solution at a concentration of 50gpl with M:L of 1:20. The air plasma treated samples were immersed in the solution for 30 minutes at 50°C. It was then padded then between the rollers to attain a wet pick up of 75%. Further, it was dried and cured at 100°C to 120°C and then given a post treatment with citric acid (8% owf at M:L of 1:20), at 50°C

for 50 minutes. The samples were dried at 80°C and cured at 140°C (Thilagavathi *et al.*, 2007). The application of coating was done using pad dry cure method.

**Antimicrobial assessment of the treated fabric**

The method of assessing the antimicrobial activity of the finished cotton fabric was carried out by Agar disc diffusion method and Parallel streak method as per AATCC method. The activity was recorded as zone of inhibition in mm.

**Results and Discussions**

The methanolic extraction of the herbal extract showed potent antibacterial activity. The ratio at which the zone of inhibition was high revealed the standardized composition of the herbs (2:1 i.e. marigold : neem). The zone of inhibition against *E.coli* and *S.aureus* was 20mm and 23mm whereas for *Bacillus sp.* & *Pseudomonas sp.* it was 22mm & 20mm respectively. The values are the average results of three replicates. The antibacterial activity of marigold and neem would be due to the phytoconstituents present in them like alkaloids, flavonoids,

saponins etc. in common. The antibacterial activity reported a wide range of spectra fighting against both Gram positive & Gram negative bacteria. Thus, extract of this combination was selected for finishing fabrics of healthy lifestyle.

The optimization of the parameters of air plasma in 100% bleached cotton fabric was carried out and the results evaluated are shown in the tabulation.

The following table 3, 4 & 5 revealed the optimized parameters at which the absorption capacity of the cotton fabric was highest. The static immersion test in percentage showed the hydrophilic property development in the fabric. The optimized parameters are found to be 3cm electrode gap with -5.11 negligible % loss of the fabric, 50w power with -6.14 % loss and 10 minutes with a loss % of -7.79. The statistical analysis showed that there are significant differences among the different parameters at 1% level. Reports of earlier investigations also revealed similar type of results where the surface modifications of cotton by air plasma was done by optimizing plasma parameters (Vaideki *et al.*, 2007).

**Table.1** Specifications of the fabric

Fabric	Fabric Count	Weight (gms)	Thickness	Count	EPI / PPI
Plain	100% bleached	110	0.25mm	40s	68 / 54

**Table.2** Preliminary assessment of antibacterial activity of medicinal herbs

SI No.	Herbal Extract Utilized	Zone Of Inhibition (in mm)**			
		<i>E.coli</i>	<i>S.aureus</i>	<i>Bacillus sp.</i>	<i>Pseudomonas sp.</i>
1	<i>Calendula officinalis</i>	18	22	21	19
2	<i>Azadirachta indica</i>	18	21	20	18
3	Combinatorial Herb	20	23	22	20

\*\* values are average of three replicates

**Table.3** Results of optimized Electrode Gap

Sample No	Constant Parameters	Parameter that is varied	Static immersion test result (in %)	% Loss / Gain	F values
Control	-	-	54 ± 0.15	-	35.4828** (p<0.01)
1	5min, 20w	2cm	55.65 ± 0.40	-2.96	
2	5min, 20w	3cm	56.91 ± 0.19	-5.11	
3	5min, 20w	4cm	55.35 ± 0.32	-2.44	
4	5min, 20w	5cm	55.81 ± 0.21	-3.24	
5	5min, 20w	6cm	55.59 ± 0.28	-2.86	

Values are mean ± SD

\*\* - Significant at 1% level

**Table.4** Results of optimized Power

Sample No	Constant Parameters	Parameter that is varied	Static immersion test result (in %)	% Loss / Gain	F values
Control	-	-	54 ± 0.23	-	46.7272 (p<0.01)
6	5min,3cm	20w	56.49 ± 0.36	-4.41	
7	5min,3cm	30w	56.91 ± 0.24	-5.11	
8	5min,3cm	40w	56.91 ± 0.39	-5.11	
9	5min,3cm	50w	57.53 ± 0.31	-6.14	
10	5min,3cm	60w	55.79 ± 0.34	-3.21	

Values are mean ± SD

\*\* - Significant at 1% level

**Table.5** Results of optimized Time

Sample No	Constant Parameters	Parameter that is varied	Static immersion test result (in %)	% Loss / Gain	F values
Control	-	-	54 ± 0.19	-	67.4996** (p<0.01)
11	3cm,50w	2min	57.09 ± 0.12	-5.41	
12	3cm,50w	8min	57.29 ± 0.26	-5.74	
13	3cm,50w	10min	58.56 ± 0.31	-7.79	
14	3cm,50w	15min	58.47 ± 0.39	-7.64	
15	3cm,50w	20min	56.88 ± 0.28	-5.06	
16	3cm,50w	25min	58.05 ± 0.23	-6.98	
17	3cm,50w	30min	57.48 ± 0.43	-6.05	
18	3cm,50w	45min	57.03 ± 0.16	-5.31	
19	3cm,50w	60min	56.07 ± 0.27	-3.69	

Values are mean ± SD

\*\* - Significant at 1% level

**Table.6** Antibacterial assessment of air plasma treated and untreated cotton fabric (Disc diffusion assay)

Specimen	Zone of inhibition (in mm)*			
	<i>S.aureus</i>	<i>Bacillus sp.</i>	<i>E.coli</i>	<i>Pseudomonas sp.</i>
Control (plain cotton fabric with herbal finish)	8.2±0.5	7.7 ±0.5	7.4±0.5	7.8±0.5
Sample (cotton fabric finished with air plasma and herbal extract)	22.0±0.5	21.3±1	19.5±0.5	19.1±1

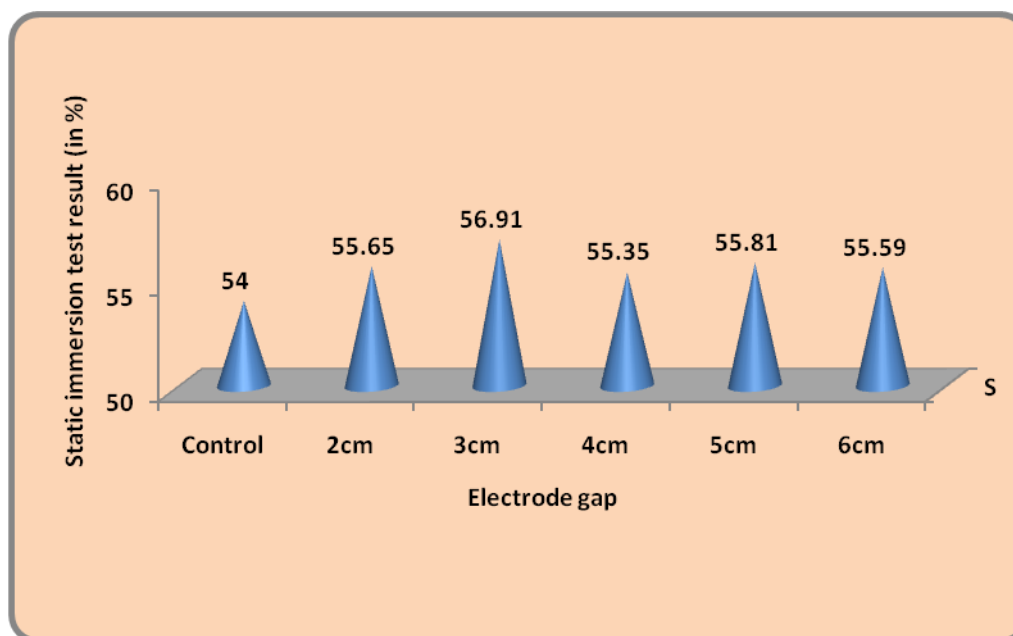
\*values of inhibitions are mean of ± SD

**Table.7** Antibacterial assessment of air plasma treated and untreated cotton fabric (Parallel streak method)

Specimen	Zone of inhibition (in mm)*			
	<i>S.aureus</i>	<i>Bacillus sp.</i>	<i>E.coli</i>	<i>Pseudomonas sp.</i>
Control (plain cotton fabric with herbal extract)	7.2±0.5	6.6 ±0.5	6.2±0.5	6.4±0.5
Sample (cotton fabric finished with air plasma and herbal extract)	22.1±0.5	21.1±1	19.0±0.5	18.9±1

\*values of inhibitions are mean of ± SD

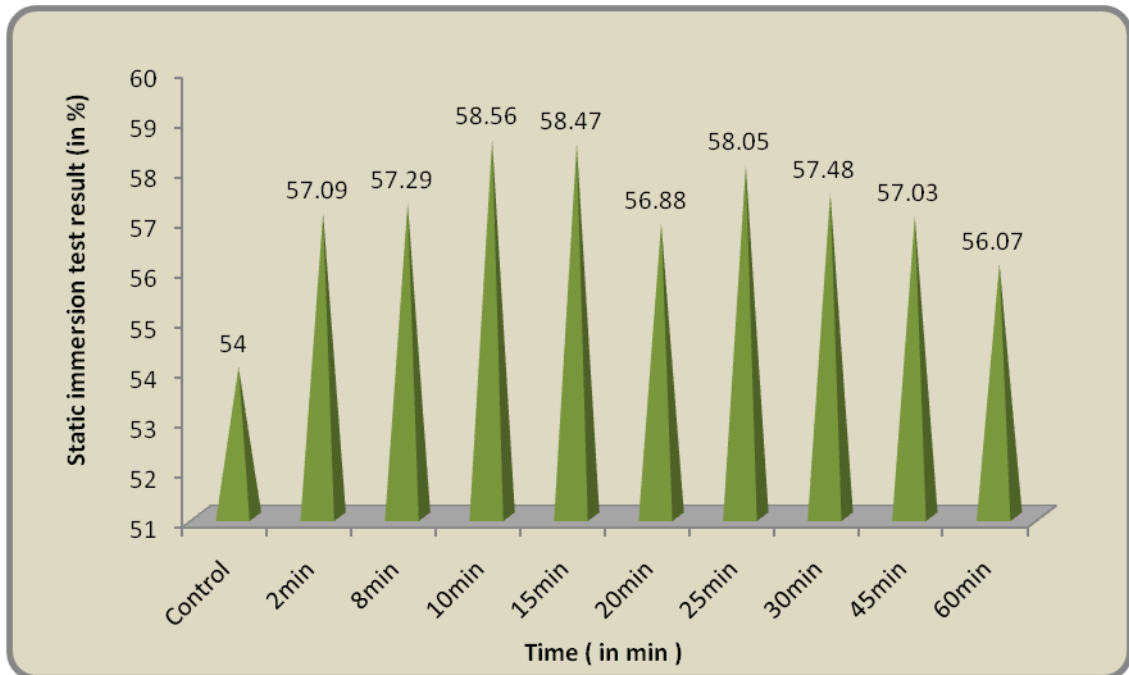
**Fig.1** Graph showing the level of percentage absorption at different electrode gap



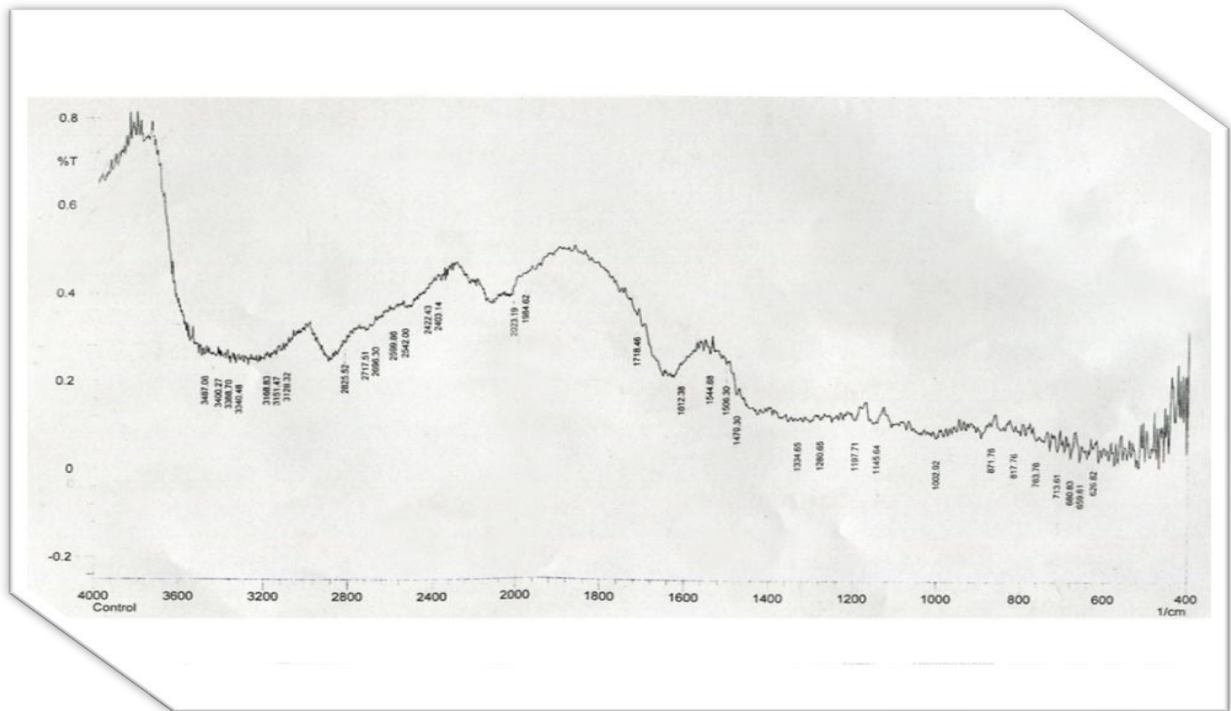
**Fig.2** Graph showing the level of percentage absorption at different watts



**Fig.3** Graph showing the level of percentage absorption at different Time



**Fig.4** Control cotton fabric (untreated air plasma treated fabric)



**Fig.5** Air plasma treated cotton fabric

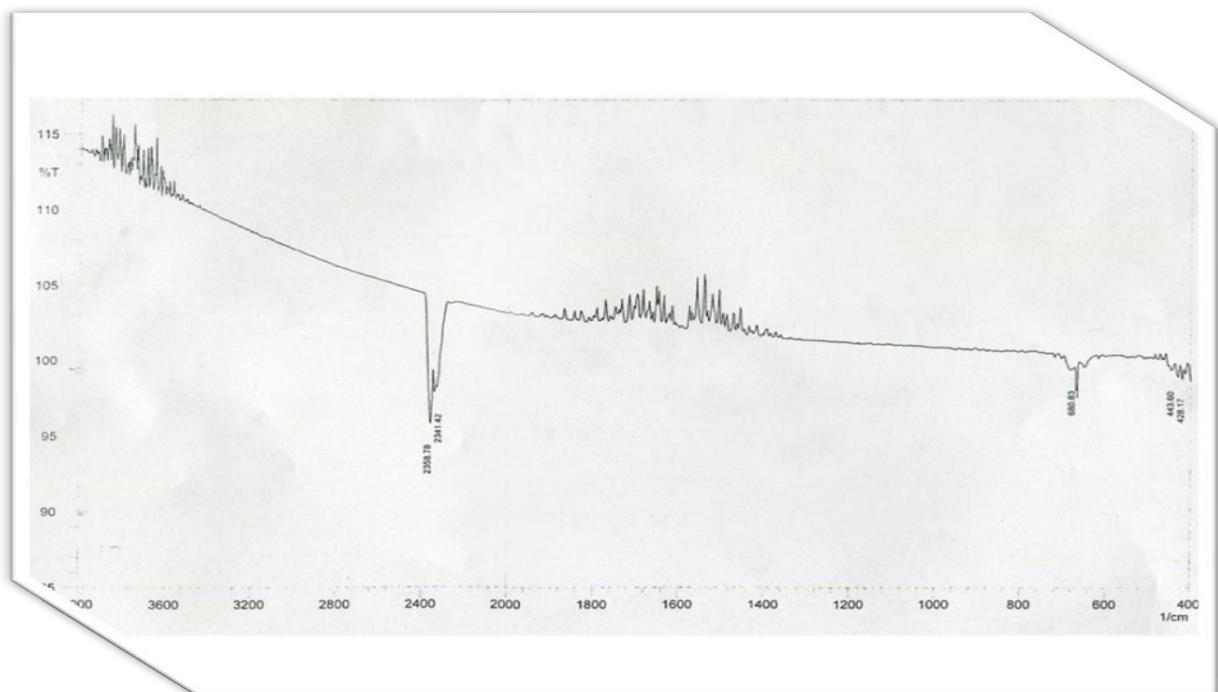




Plate.1 Antibacterial activity against *Staphylococcus aureus*.

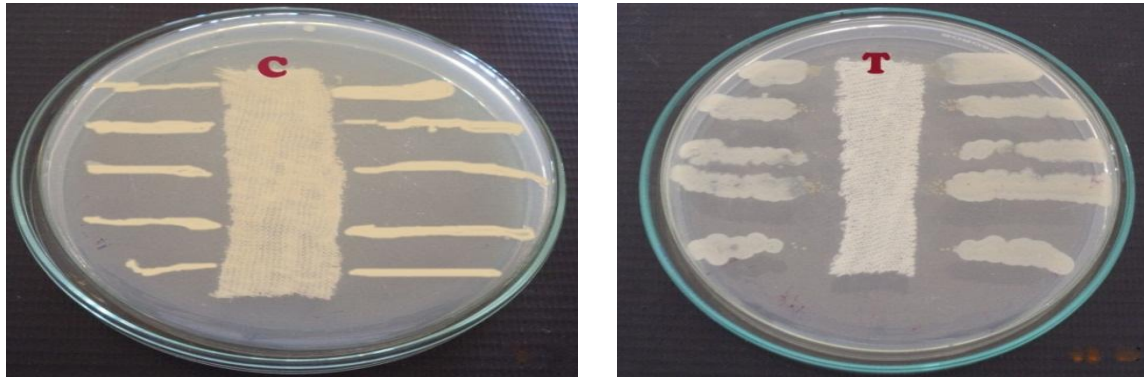


Plate.2 Antibacterial activity against *E.coli*.

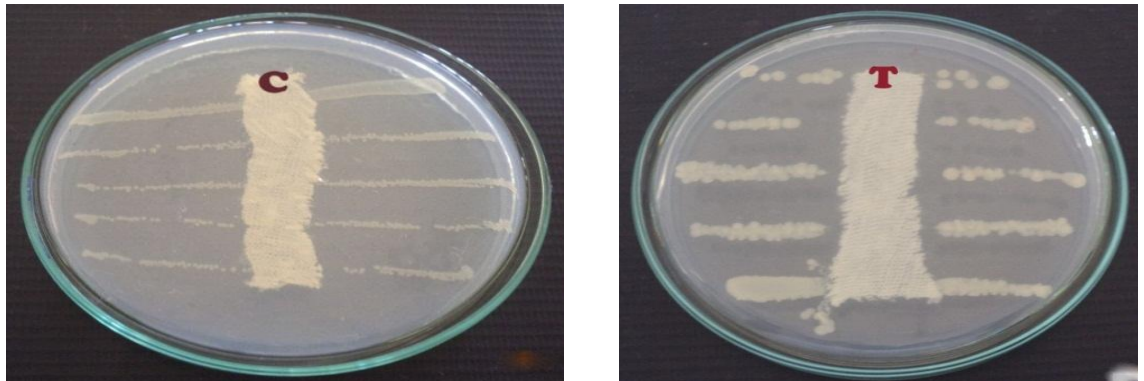


Plate.3 Antibacterial activity against *Bacillus sp.*

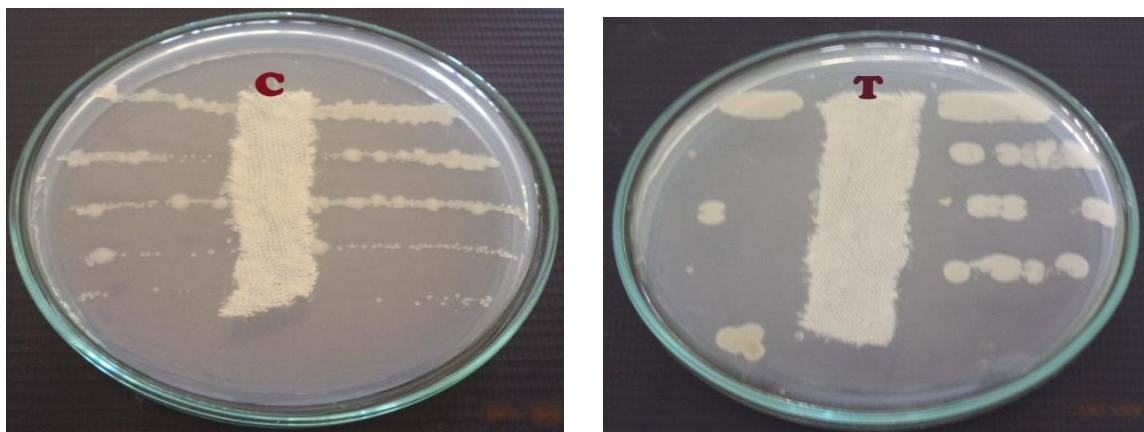
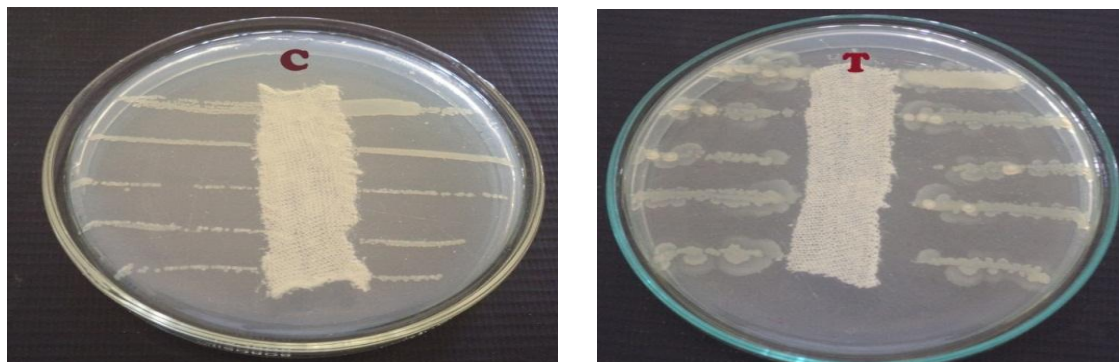
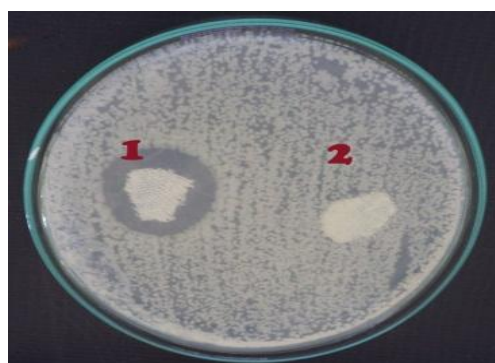


Plate.4 Antibacterial activity against *Pseudomonas sp.*

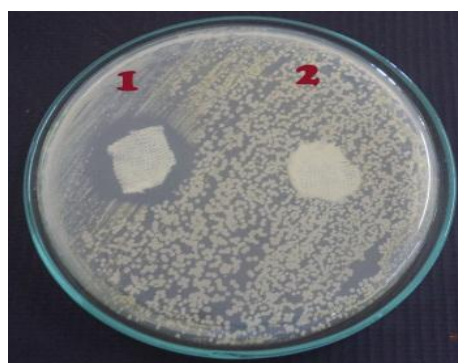


Note : C- control fabric (plain cotton), T- treated fabric (plasma + herbal extract).

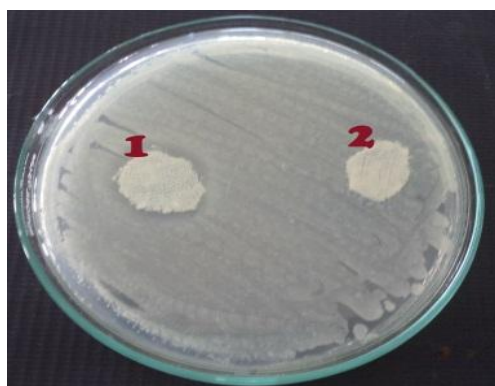
Plate.5 Antibacterial activity (Disc diffusion method)



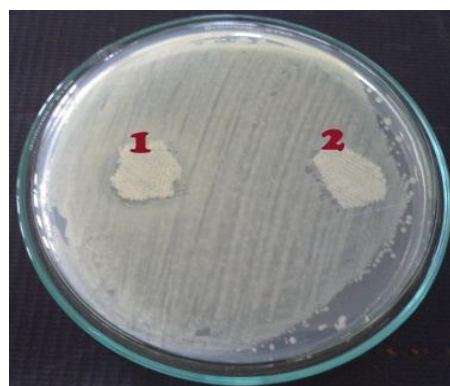
*Bacillus sp.*



*S.aureus*



*E.coli*



*Pseudomonas sp.*

Note: 1- Treated fabric (plasma + herbal extract), 2- Control fabric (plain cotton).

The plasma treatment leads to surface erosion of the cotton fabric which generates a weight loss accompanied by an increase in the fibre carboxyl group and carbonyl group contents. The increase in fibre carboxyl

group content leads to a more wettable fibre and the rate of fibre vertical wicking is increased. The FT-IR spectrum of the control and the optimized air plasma treated sample are shown in fig.4 respectively.

The strong absorption in the region  $2300\text{cm}^{-1}$  to  $2800\text{cm}^{-1}$  in fig.5 confirms the presence of carbonyl  $-\text{C}=\text{O}$  group in the plasma treated sample. This illustrates the  $-\text{OH}$  groups in the cellulose structure has been oxidized to form either aldehyde group or carboxylic acid group which is responsible for increasing the hydrophilicity of the sample. The increase in carbonyl group (polar) resulted in such improvement. In the control which is the untreated plasma fabric, there is no carbonyl group (polar), hence not much hydrophilic property was reported (Reza *et al.*, 2003).

The above table 4 and 5 clearly illustrates the importance of the air plasma treated cotton fabric. The plasma treated fabric was able to absorb the herbal extract in a better way and therefore, zone of inhibition against both Gram positive & Gram negative showed excellent antibacterial activity compared to the control fabric. Such results were also observed in the literature of several articles (Vaideki *et al.*, 2007). The highest antibacterial activity was obtained against *S. aureus* both in disc diffusion and parallel streak method.

In conclusion, technology is indeed a magical tool to bring forth surface modification of cotton fabric. Plasma, the fourth state of matter could be efficiently applied to textile fabrics for better hydrophilic property and this property could be exploited in antimicrobial finishes. The detrimental effects of pathogenic bacteria could be controlled in functional textiles.

### **Acknowledgement**

I extend my heartfelt thanks to the management of PSG College of Arts & Science, Coimbatore for providing the facilities to carry out this work.

### **References**

- AATCC Technical Manual, Vol.76, 2001.
- Anita Gupta. 2001. "Biotechnology applications in textiles", *Indian J. Fibre Sci. Technol.*, Vol.26, 206-213.
- Bhat, N.V., R.N. Bharti, A.V. Gore and A.J. Patil. "Effect of atmospheric pressure air plasma treatment on desizing and wettability of cotton fabrics", *Indian J. Fibre and Textile Res.*, Vol.36, 42-46.
- Hartwig Hocker. 2002. "Plasma treatment of textile fibres", *Pure Appl. Chem.*, Vol.74 (3), 423-427.
- Kartick Samanta, Manjeet Jassal and Ashwini k. Agarwal. 2006. "Atmospheric pressure glow discharge plasma and its applications in textiles", *Indian J. Fibre and Textile Res.*, Vol.31, 83-98.
- Mahesh, B., Satish, S. 2008. "Antimicrobial activity of some important medicinal plant against plant and human pathogen", *World J. Agri. Sci.*, 4(S): 839-843.
- Mathur, Rashmi and Mamta Goyal. 2011. "Antimicrobial and phytochemical estimation of *Calendula officinalis* against human pathogenic microorganisms", *Int. J. Innovations in Biosci.*, Vol.1, 1-10.
- Mucha, H., D. Hafe, H., Swerer. 2002. "Antimicrobial finishes and modifications", *Melli and Int.*, Vol.8, 148.
- Natarajan, v. 2002. "Azadirachta indica in the treatment of dermatophytosis", *J. Ecobiol.*, 14: 201-204.
- Punam Sen and Rajendran, R. 2004. "Evaluation of antibacterial efficacy of medicinal plants obtained from the hilly areas of northeastern state of Tripura", *Int. J. Curr. Res.*, Vol.6 (3), 5902-5905.

- Rad Ford, P.J. 1973. "Application and evaluation of antimicrobial finishes", *American Dyestuff Reporter*, Vol. 62, 48-59.
- Rajpreet, k.,Virk and Gita, N. Ramasamy. 2004. "Plasma and antimicrobial treatment of nonwoven fabrics for surgical gowns", *Textile Res. J.*, 74 (12), 1073-1079.
- Ranganathan, k. 2005. "Weaving versatile woven fabrics of India", Vol.46, 27, 2005.
- Reza, M.A., Malek and Ian Holme. 2003. "The effect of plasma treatment on some properties of cotton", *Iranian Polymer J.*, 12(4): 271-280.
- Thilagavathi, G., Krishna Bala, S., and Kannaian, T. 2007. Micro-encapsulation of herbal extracts for microbial resistance in healthcare textiles", *Indian J. Fibre and Textile Res.*, Vol.32, 351-354.
- Vaideki, k., Jayakumar, S., Thilagavathi, G. 2007. "Investigation on the antimicrobial activity of RF plasma and azadirachtin treated cotton fabric", *J. Instrum. Soc. India*, 37(4): 258-266.

**How to cite this article:**

Punam Sen and R. Rajendran. 2016. Effective Optimization and Evaluation of Air Plasma Treatment on the Surface of 100% Bleached Cotton Fabric for Better Dabbing of Antibacterial Finishes. *Int.J.Curr.Microbiol.App.Sci*. 5(8): 446-457.  
doi: <http://dx.doi.org/10.20546/ijcmas.2016.508.048>