Antibacterial Finishing of Polyester/Cotton Blend Fabrics Using Neem (*Azadirachta indica*): A Natural Bioactive Agent

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ABSTRACT: The study focused on the development of biofunctional polyester/cotton blend fabric using a natural product. An antimicrobial agent extracted from the seeds of Neem tree (*Azadirachta indica*) was used for imparting antibacterial property to the blend fabric. Resin and catalyst concentrations were optimized to get the maximum crosslinking in the fabric blends using glyoxal/glycol as a crosslinking agent. The optimized concentrations were used to treat the fabric with the antimicrobial agent along with the crosslinking agent. Quantitative analysis was carried out to measure the antimicrobial activity against Gram-positive and Gram-negative bacteria. The results showed that the treated fabrics inhibited the growth of Gram-positive bacteria (*Bacillus subtilis*) by more than 90% as compared to the control sample. Antimicrobial activity against Gram- positive bacteria was retained up to five machine washes and decreased thereafter. The antibacterial activity was higher against Gram-positive bacteria as compared to Gram- negative bacteria (*Proteus vulgaris*). The treated fabrics also showed improved crease recovery property although the tensile property showed a marginal decrease. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 106: 793–800, 2007

Key words: natural antimicrobial agent; neem extract; glyoxal/glycol crosslinking; antimicrobial textiles

INTRODUCTION

In the last century, improved standards of human lifestyles have brought a greater sense of appreciation towards comfort, health and hygiene as far as clothing and upholstery is concerned. Although the need to preserve fabrics against rotting and mildew stain, particularly in industrial usage has long been recognized, the use of biostats to inhibit odor development resulting from biological growth on textiles exposed to perspiration had not been considered a real need until recently. The greater use of synthetic fibers and blends in items such as shirts, hosiery, blouses and underwear has accelerated the need for bacteriostatic finishes on clothing. The moisture transport characteristics of such blends and synthetics tend to cause a greater degree of ''perspiration wetness'' than that occurs with fabrics of wholly natural fibers. Additionally, there is a growing volume of literature demonstrating the survival and growth of microorganisms in textiles and their dissemination as a health risk.[1,2] The major classes of antimicrobial agents for textiles include triclosan, metal and their salts, organometallics, phenols, quaternary ammonium compounds and organosilicons etc.[3] The prime consideration related to the end use and function of an antimicrobial and insect repellent finish on textiles is that it should meet environmental and low toxicity criteria along with its functional properties. Therefore, it is vital to research and develop eco-friendly antimicrobial agents for textile applications. The antimicrobial effect of natural products such as chitosan[4–6] and natural dyes[7,8] on textiles has been extensively discussed in literature. Antimicrobial fibers obtained from chitosan are readily available in the market for potential use.[9,10] The effect of various plant extracts on the bacteria has been studied by a number of researchers in the past.[11–13] Although, there are many natural antibacterial agents but the work on the exploration of their antibacterial activity on textile material is very limited and not well documented, and Neem extract is one of them.

Neem tree (*Azadirachta indica*), one of the richest sources of biological active compounds, belongs to the Meliaceae (Mahogony) family and is abundantly found in the Indian subcontinent. The biological activity of Neem has been known from centuries, and preparations have been used in traditional Indian medicine since ancient times. A large number of medicines that include herbal medicine, toothpaste, cosmetics, toiletries and pharmaceuticals are now

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based on Neem derivatives because of its unique properties. The most important quality of Neem compounds is that they are less toxic to warm-blooded animals like human.[14] It is reported that more than 300 active compounds have been isolated from different parts such as leaves, bark and seeds of Neem tree, while some of them have already been identified for their potential antimicrobial effect, the activity of remaining active compounds are yet to be identified. The more common and therefore the most analyzed compounds are azadirachtin, nimbin, nimbidin, salannin, nimbidol, gedunin, sodium nimbinate, queceretin, etc.[15,16] It has an excellent potential as antimicrobial agent as it has been firmly established that the Neem extracts and its main compounds azadirachtin, salannin and meliantriol are insect growth regulator and antifeedent.[17] The antibacterial activity of Neem oil,[18] bark extract[19,20] and seed extract[21] is discussed elaborately in literature. Neem leaf extract has also been reported to show antiviral and virucidal properties.[22,23] Joshi et al.[22] have demonstrated that purified extract of Neem shows activity against HIV and sexually transmitted disease pathogens and also has contraceptive properties. The antibacterial properties of Neem oil in combination with other herbal oils such as Clove, Tulsi and Karanga have been used to impart antimicrobial finish on cotton textiles.[24,25]

Work on antimicrobial finishing of cotton fabric using Neem seed extract along with glycol/glyoxal crosslinking agent has been recently reported by our group.[26,27] The next most useful fabric after cotton is polyester/cotton blend fabric. The present study aims at the development of antibacterial polyester/cotton blend fabric by making use of Neem seed extract as the ecofriendly antimicrobial agent.

EXPERIMENTAL

Materials

A desized, scoured and bleached plain weave (125 ends × 85 picks) PET/cotton (48/52) blend fabric weighing 105 g/m^2 and plain weave (142 ends × 90 picks) PET/Cotton (67/33) blend fabric weighing 130 g/m^2 were used throughout the study. Glyoxal ($C_2H_2O_2$, Thomas Baker, 40% aqueous solution) was used as crosslinking agent. Aluminum sulfate ($Al_2(SO_4)_3.18\ H_2O$, Merck) as catalyst, Tartaric acid ($C_4H_6O_6$, Merck) as catalyst activator and ethylene glycol ($C_2H_6O_2$, Merck) as coreactant additive were used for glyoxal crosslinking. Neemazal Technical^R (seed extract from Neem tree, supplied by EID (Parry) India Ltd, Bangalore, India) was used as natural antimicrobial agent. The Neem seed extract was standardized for its three major active ingredients by high-pressure liquid chromatography (HPLC) and it was found to contain 31.6% azadirachtin, 1.24% nimbin, and 0.18% salannin by mass.[27]

Methanol (CH_3OH) was used as a solvent for Neemazal. Lissapol N was used for washing the treated fabrics. Agar-Agar (Merck), Peptone (Merck) and Beef extract (Merck) were used for the antimicrobial testing of the treated fabrics. All the chemicals used were of reagent grade.

Methods

Application of crosslinking agent

To get optimum crosslink density, glyoxal/glycol resin and catalyst concentrations were optimized for the blend fabrics and are discussed in results and discussions part later. The samples were impregnated for 5 min in the pad bath containing glyoxal, ethylene glycol, aluminum sulfate and tartaric acid. Samples were then padded through a two-bowl vertical laboratory padder with two dips and nips to give a wet pick up of (85 ± 5)% owf. Immediately after padding the samples were dried at 85°C for 5 min and cured at 120°C for 2 min. After curing, the fabric samples were rinsed with warm water at 45–55°C for 15 min and dried at room temperature.

Application of Neem seed extract

Neem seed extract (5 and 10% w/v) (Neemazal Technical) was applied on the blend fabric along with glyoxal/glycol, aluminum sulfate and tartaric acid using conventional curing system. Samples were padded through a two bowl vertical laboratory padder with two dips and two nips to give a wet pick up of (85 ± 5)% owf. After padding the samples were dried at 85°C for 5 min and cured simultaneously in the curing chamber at 120°C for 2 min.

Performance evaluation and testing

Weight add-on

To estimate the amount of Neem extract attached to the fabric, the total weight add-on of the treated fabric was evaluated before and after washing. Since the weight add-on due to the crosslinking agent alone is very low, the total weight add-on of the treated fabrics mostly reflects the amount of Neem extract attached.

Crease recovery angle

Standard AATCC-66-1975 test method was used to determine the dry crease recovery angle (CRA). Samples were conditioned (20°C, 65% RH. for 24 h) prior to testing. For each sample, 12 specimens were tested and the average value is reported.

Tensile properties

Samples were conditioned in the standard atmosphere prior to testing. The breaking strength measurements of fabric strip (20 cm × 5 cm) were made on Instron 4202 (based on CRE principle) (ASTM-D5035-90) with a gauge length and test speed of 75 mm and 100 mm/min, respectively.

Evaluation of antimicrobial activity

The untreated as well as the treated samples were subjected to the modified colony counting method (quantitative) (AATCC Test Method 147-1998) for evaluation of the antimicrobial activity of the finished fabric. Agar plates were prepared by solid culture media using Petri plate. Solid culture media contains peptone (5 g/L), beef extract (3 g/L) and agar (20 g/L) and adjusted to pH of 6.8 + 0.1 at 25°C. The media was then sterilized at 120°C and 15 psi for 30 min before preparing plates.

Fabric swatch (1″ × 1″) was placed in sterilized flask containing liquid culture solution. Flask was shaken for 24 h at 150 rpm on laboratory shaker to release the bacteria from the swatch. After 24 h incubation, serial dilutions of the culture were made in sterilized water. Dilution of 10^{-3}, 10^{-4}, 10^{-5}, and 10^{-6} were used for colony counting method. A total of 100 μL of culture was spread on to the agar plate and plates were incubated at 30°C for 24 h. After incubation bacterial colonies were counted. The percent reduction in number of colonies in treated sample as compared to the untreated sample gives the antibacterial activity of the fabric.

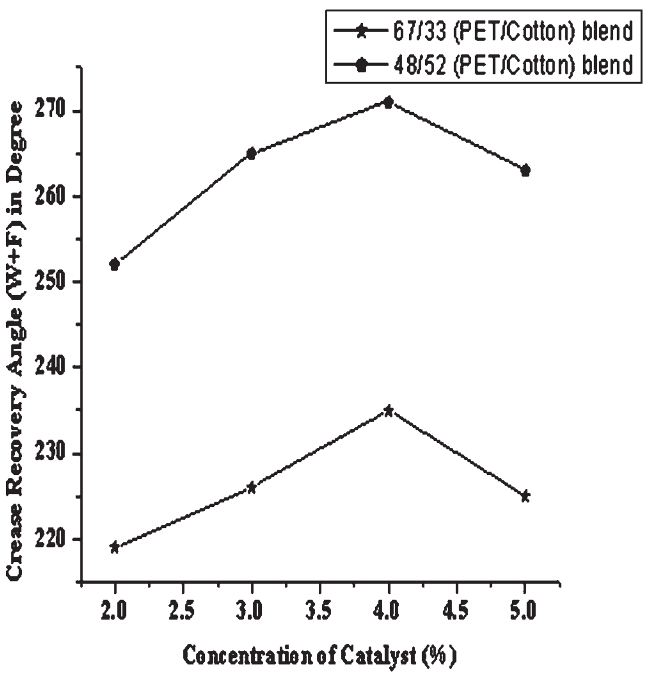

Figure 1 Effect of catalyst concentration on crease recovery angle of PET/cotton blend fabrics.

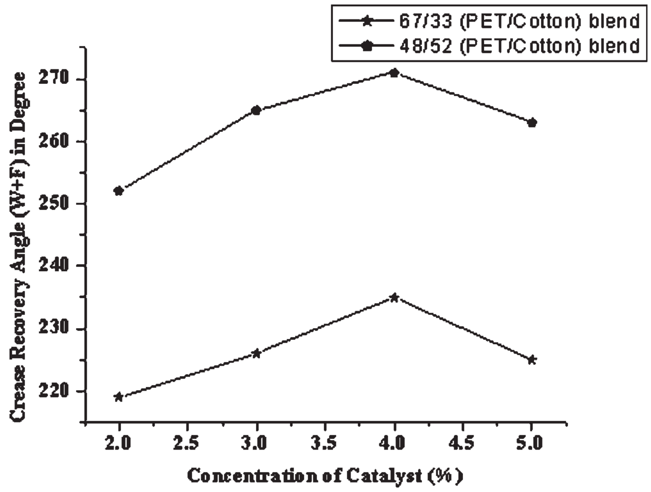

Figure 2 Effect of resin concentration on crease recovery angle of PET/cotton blend fabrics.

$$\text{Antimicrobial Activity or \% Reduction} = [(A - B)/A] \times 100$$

where, A is the bacteria colonies of untreated fabric and B is the bacteria colonies of the treated fabric.

Washing fastness

The finished fabrics were washed in Launder-*o*-meter according to AATCC test method 124-1975 (I I A), using a nonionic detergent, Lissapol N.

Bending length

Bending length measurement was carried out in both warp and weft direction of the treated and untreated samples using Paramount Bending Length tester.

Whiteness index

Whiteness Index of the treated samples was measured by Gretag Macbeth, Color Eye-7000-A using Color-eye Control software.

RESULTS AND DISCUSSION

Antimicrobial finishing of PET/Cotton blended fabrics

Glyoxal/glycol concentration and catalyst concentration were first optimized for PET/Cotton blend fabrics using conventional curing system. Then the optimum concentrations of glyoxal/glycol and catalyst

TABLE I
Comparison of Performance Properties of PET/Cotton Blend Fabrics

Sl no.	Samples	Avg. dry crease recovery angle (W + F) (°)	CV% of dry crease recovery angle	Avg. tensile strength (kg)	CV% of tensile strength	Tensile strength retention (%)
1	Untreated PET/cotton (67/33)	213	0.40	84.02	2.5	100
2	Cured PET/cotton (67/33), washed	235	0.32	80.95	0.56	96.9
3	Untreated PET/cotton (48/52)	240	0.57	78	3.8	100
4	Cured PET/cotton (48/52), washed	271	0.84	73.78	1.2	95.78

Glyoxal = 4.8%, Glycol = 5.2%, Aluminum sulfate = 4% owr, Tartaric acid = 4% owr.

were used for the integration of neem extract on the blend fabrics.

Effect of catalyst concentration

It has been reported in literature that the combination of aluminum sulfate and tartaric acid permits the use of much lower curing temperatures particularly if glycol is present in equimolar amounts with the glyoxal.[28] The concentration of aluminum sulfate and the tartaric acid were taken on the basis of glyoxal content. Figure 1 shows the CRA of conditioned blend fabrics treated with glyoxal/glycol and varying concentration of catalyst system. As the concentration of catalyst increases, the CRA increases and reaches a maximum at 4% (on the weight of resin) catalyst system and then decreases for both the blend fabrics. The decrease in crease recovery with higher concentration of catalyst may be due to the excessive degradation of cotton at lower pH. It will be noted that cotton is susceptible to degradation in acidic condition. It is also likely that the crosslinking is affected in acidic environment. A combination of these factors influences the decrease in crease recovery. It is obvious from the Figure 1 that 4% (o.w.r.) catalyst concentration can be optimized for further studies.

Effect of crosslinking agent

Welch[29] has reported that the durable press appearance rating and fabric weight gain is increased as the glycol/glyoxal mole ratio is increased up to a ratio of 1 : 1. Therefore, in this study, glyoxal and glycol were used in the molar ratio of 1 : 1. Figure 2 shows the CRA of blend fabrics treated with varying concentration of glyoxal/glycol. It is observed that as the concentration of resin increases, CRA increases up to 4.8% (w/v) glyoxal concentration beyond which there is no further improvement. This indicates that the effective crosslink density does not improve further on increasing the concentration of crosslinking agent. Hence, 4.8%/5.2% glyoxal/glycol

TABLE II
Weight Add-on % of PET/Cotton Blend Fabrics

Sample	Wt. add on (%)
Control (without S.E) (unwashed) (48/52 blend)	5.2
5% S.E (unwashed) (48/52 blend)	10.7
10% S.E (unwashed) (48/52 blend)	23.0
Control (without S.E)(washed) (48/52 blend)	0.5
5% S.E (washed) (48/52 blend)	1.3
10% S.E (washed) (48/52 blend)	1.4
Control (without S.E) (unwashed) (67/33 blend)	3.3
5% S.E (unwashed) (67/33 blend)	7.7
10% S.E (unwashed) (67/33 blend)	15.1
Control (without S.E)(washed) (67/33 blend)	0.2
5% S.E (washed) (67/33 blend)	1.2
10% S.E (washed) (67/33 blend)	1.1

S.E is indicating Neem seed extract.

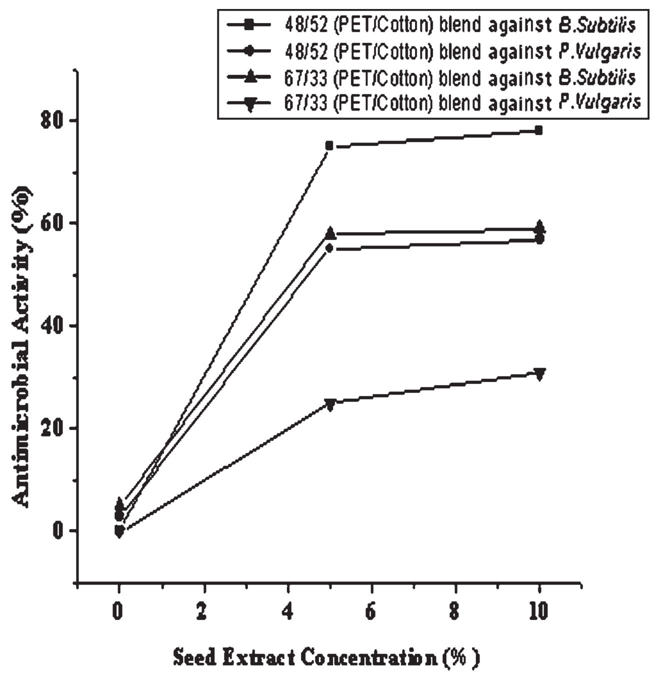

Figure 3 Effect of neem seed extract concentration on antimicrobial activity of PET/cotton blend fabrics.

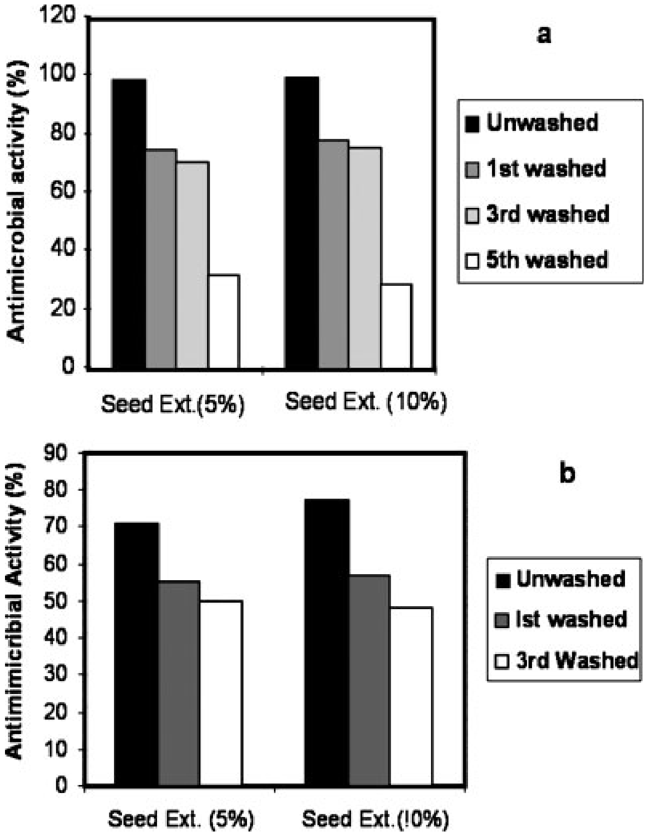

Figure 4 (a) Washing durability of neem seed extract treated PET/cotton (48/52) blend fabric in terms of antimicrobial activity against *Bacillus subtilis* (b) Washing durability of neem seed extract treated PET/cotton (48/52) blend fabric in terms of antimicrobial activity against *Proteus vulgaris*.

resin concentration (in 1 : 1*M* ratio) was used for further studies.

Performance properties of crosslinked fabrics

The performance properties of cured samples are given in Table I and it is noted that crosslinked samples offer an improved CRA as compared to the corresponding control samples. However, the increase in CRA of cured blend sample is marginally less as compared to cured 100% cotton sample ($\sim$ 275°)[26] although initial CRA for both type of blends are higher than pure cotton sample (150°). This is due to the better crease recovery property of polyester present in the blend fabrics. The low CRA in cured blends may be due to poor crosslinking as mainly the cotton part is involved in crosslink formation. This also results in the high CRA in cotton rich 48/52 (PET/Cotton) blend than in 67/33 (PET/Cotton) blend. It has been generally observed that crosslinking treatment reduces the fabric strength. Crosslinking between molecules stiffens the macromolecular network and causes fiber embrittlement, which reduces the mechanical strength of the treated fabric.[30] It is thus observed in Table I that 67/33 (PET/Cotton) cured blend sample offers a little more retention of tensile strength (1.2%) as compared to 48/52 (PET/Cotton) blend sample.

Add on percentage

To calculate the actual amount of Neem extract attached to the fabric, the wt. add-on% (Table II) was calculated before and after washing. The results indicate that 48/52 (PET/Cotton) possesses higher proportion of active ingredients as compared to 67/33 (PET/Cotton) blends. It is also seen that crosslinking treatment does not enhance the fixation of active ingredients significantly as evidenced by the significant loss of add-on after washing in both the samples. However it is believed that the availability of more —OH functional group in 48/52 (PET/Cotton) blend than in 67/33 (PET/Cotton) blend played a crucial role in enhancing the crosslinking. From the Figure 3, it is also clear that further addition of

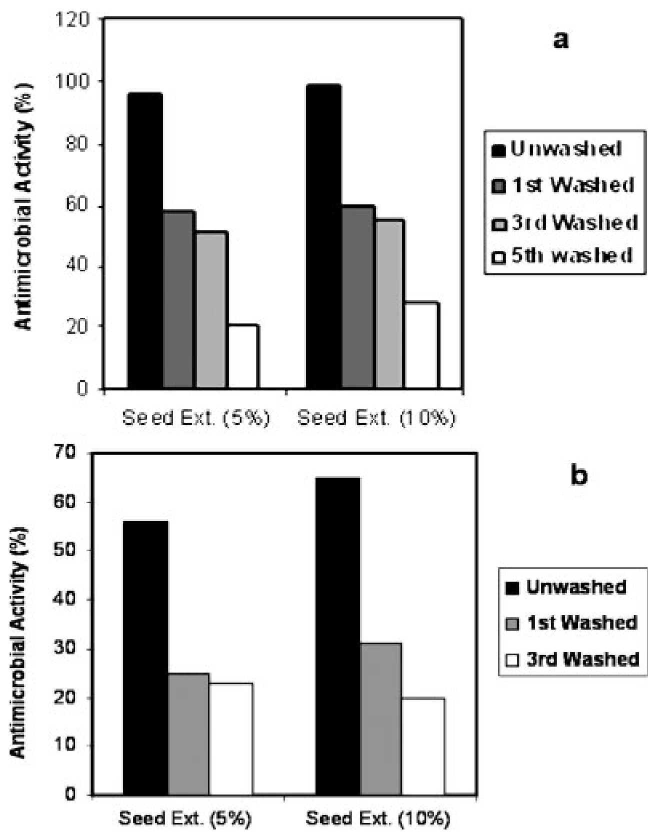

Figure 5 (a) Washing durability of Neem seed extract treated PET/Cotton (67/33) blend fabric in terms of antibacterial activity against *Bacillus subtilis.* (b) Washing durability of neem seed extract treated PET/Cotton (67/33) blend fabric in terms of antimicrobial activity against *Proteus vulgaris*.

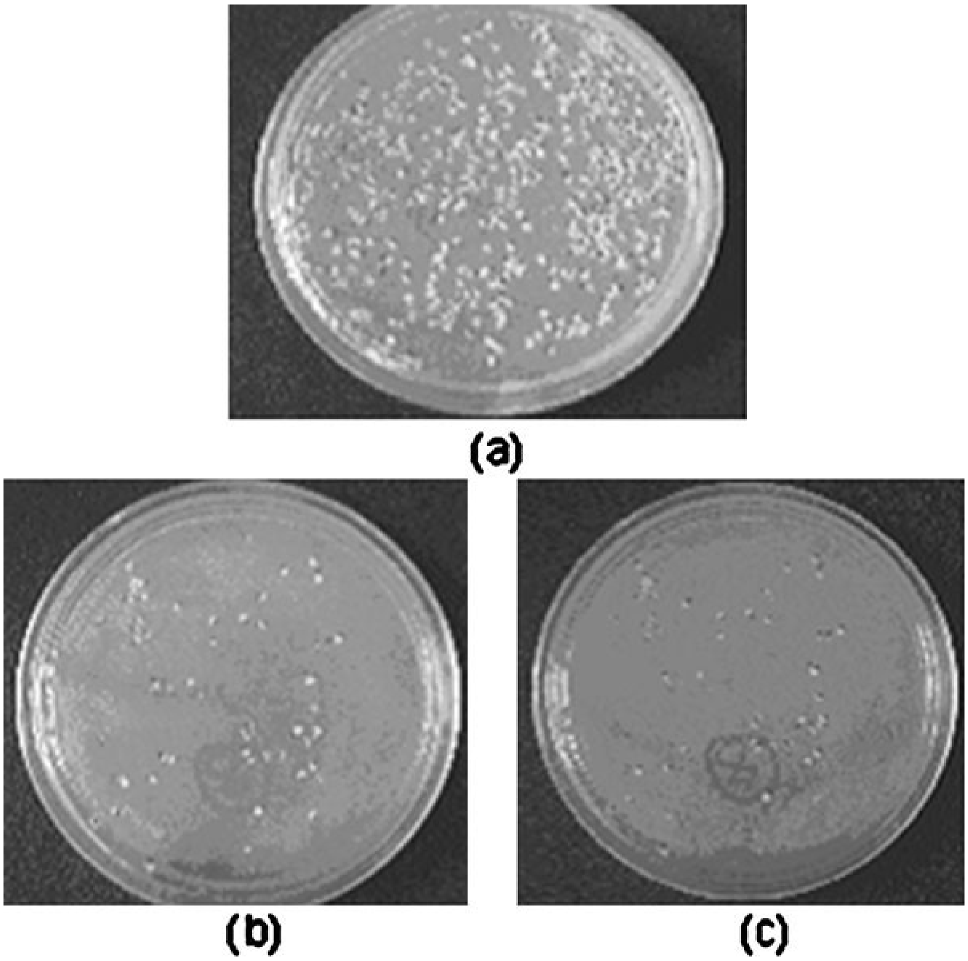

Figure 6 Photographs of untreated and Neem seed extract (5%w/v) treated cotton/PET (52/48) blend fabric (against *B. subtilis* bacteria) (a) untreated (b) neem treated, after one wash (c) neem treated, after three washes.

Neem extract (10%) does not improve the antimicrobial property of the treated fabrics.

Effect of Neem seed extract on antimicrobial activity

The antibacterial activity of treated fabric was evaluated quantitatively by measuring the number of colonies of *Bacillus subtilis* (Gram-positive) and *Proteus vulgaris* (Gram-negative) as per the test method discussed in the experimental section. Two control samples were tested along with the Neem-treated samples. Untreated fabric was taken as control-1 and crosslinked fabric without Neem seed extract was taken as control-2. Very less antimicrobial activity (<5%) was found for control-2 as compared to control-1. Therefore, antimicrobial activity for the Neem-treated crosslinked fabric was checked taking control-1 as the reference. The results [Figs. 4(a) and 5(a)] indicate that both the unwashed blends register above 95% antimicrobial activity against *B. subtilis*. After one wash the efficacy of antimicrobial activity (using 5% w/v Neem extract) of the treated 48/52 (PET/cotton) and 67/33 (PET/Cotton) reduced to 75% and 58%, respectively. Similarly, the antimicrobial activity against *P. vulgaris* (Gram-negative) is also reduced after one wash. The activity is reduced to 57% and 31% (using 10% w/v Neem extract) for

TABLE III
Whiteness Index of Neemazal-Treated PET/Cotton Blend Fabrics

Samples	Neemazal applied (%)	Avg. Whiteness Index—CIE
67/33 (untreated)	–	139.94
67/33 (unwashed)	5	86.46
67/33 (unwashed)	10	67.12
67/33 (1st washed)	5	116.93
67/33 (1st washed)	10	111.46
48/52 (untreated)	–	152.74
48/52 (unwashed)	5	112.64
48/52 (unwashed)	10	70.89
48/52 (1st washed)	5	136.35
48/52 (1st washed)	10	128.24

TABLE IV
CRA, Bending Length, and Tensile Strength of the Finished PET/Cotton Blend Fabrics

Samples	Avg. dry crease recovery angle (W+F) (degree)	CV % of dry crease recovery angle	Avg. bending length (cm)	CV % of bending length	Tensile strength retention (%)
Untreated 67/33 (PET/cotton) blend	213	0.40	2.2	0.88	–
Neem + glyoxal/glycol treated 67/33 (PET/cotton) blend	223	0.69	2.4	0.69	88
Untreated 48/52 (PET/cotton) blend	240	0.57	1.2	0.56	–
Neem + glyoxal/glycol treated 48/52 (PET/cotton) blend	255	0.58	1.4	0.80	86

CRA of only glyoxal/glycol treated 67/33 (PET/Cotton) and 52/48 (Cotton/PET) blend fabric was 235^o and 271^o, respectively.

48/52 (PET/Cotton) and 67/33 (PET/Cotton) blends, respectively.

However, the fabric blends soaked in Neem extract aqueous dispersion without any crosslinking agent did not show any activity after one wash. This shows that the active ingredients, responsible for bacteriostatic or bactericidal effect, in the Neem extract have no affinity to the fabric and therefore does not get firmly attached to the blended textile structure in the absence of crosslinking agent.

The durability of the antibacterial effect of the Neem extract treated crosslinked fabric was evaluated in term of its retention of antibacterial activity on washing (machine wash, as per AATCC test method 124-1975). Figures 4(a,b) and 5(a,b) show that the Neem extract treated fabric retains the antibacterial activity up to the three machine washes although there is a sharp decrement in antibacterial activity of Neem extract treated fabric after first wash as compared to unwashed samples. This is due to the significant loss of add-on % after first wash as discussed earlier. Figure 6 shows the photographs of bacteria colony-forming unit of *Bacillus subtilis* before and after the finishing treatment.

The cellulose part in the blend fabrics is actively involved in bond formation with the active ingredients of Neem extract. The active ingredients may be attached to cellulose by physical bonding and the crosslinking agent may act as a bridging material for chemical bond formation with the Neem limonoids. It is also possible that the Neem extract may be entrapped within the crosslinked molecular chains of the blend fabrics. This mechanism has been studied and suggested for 100% cotton fabric in a previous study by our group.[27,28]

Whiteness index of the treated fabrics

From Table III it is clear that average whiteness index (WI) value is decreased significantly for the treated and unwashed samples. Although after one wash, WI is restored but still a little less than the original value. For 5% Neemazal applied samples, WI is little more than the 10% Neemazal finished fabrics, which may be due to the relatively higher add-on% of the later.

Performance properties of the finished PET/cotton blended fabrics

The CRA of the Neem treated finished blend fabric is lower than that of only glyoxal/glycol treated fabric but still higher than the untreated blend fabrics (Table IV). It is also seen that bending lengths are more or less same before and after the treatment, which indicates, that flexibility/softness of fabric is maintained in the finished samples. Although the freshly finished fabric have a slight natural odor of the Neem extract, but it goes away after storing it for a few days.

CONCLUSIONS

Glyoxal/glycol resin concentration of 4.8%/5.2% (on the weight of the bath), and tartaric acid and aluminum sulfate concentration of 4% (on the weight of the resin) are the optimum concentrations for optimum crosslinking of both the blend fabrics used. About 5% (w/v) Neem seed extract is optimum concentration for getting good antibacterial activity for both the blend fabrics. 48/52 (PET/Cotton) blend fabric has greater antibacterial activity than 67/33 (PET/Cotton) blend fabric. The antibacterial activity of the Neem extract treated blend fabric is retained up to five and three machine-washes against Gram-positive and Gram-negative bacteria, which is equivalent to 25 and 15 home launderings, respectively. Good tensile strength and flexibility is maintained in the finished blend fabrics. Neem seed extract can thus be used as an effective eco friendly bioactive agent for imparting semi durable antibacterial finish on the polyester/cotton blend fabric using the process developed.

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