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Eco-friendly Antimicrobial Finish for Wool Fabric

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ABSTRACT Eco-friendly antimicrobial finish has been imparted to the wool fabric using methanolic *neem* extract by exhaust and pad dry cure method in order to reduce the bacterial growth on fabric. Finish was applied in two concentrations (3 and 5gpl) on grey as well as enzymatically scoured wool fabric and compared. The antimicrobial activity of the finish was accessed by quantitatively by AATCC-100 test method in terms of bacterial reduction. Effectiveness of finish was also accessed after predetermined number of washing cycles. It was observed that *neem* has been found effective against bacterial growth on wool fabric even after 20 washing cycles (79.28%). *Neem* treated scoured wool fabric shows very good antibacterial activity than *neem* treated grey wool fabric. Since the active *neem* ingredient extracted from *neem* leaves can serve as appropriate alternative for eco-friendly antimicrobial finish to conventional harmful chemicals in present use.

1. INTRODUCTION

Usually textile substrates are deficient in one or more properties or improved properties are desired for the substrate. Textile finishing provides a method whereby deficiencies in the textile can be corrected or specific properties can be introduced. Textiles are excellent substrate for bacterial growth and microbial proliferation under appropriate moisture, nutrients and temperature conditions (Ramachandran et al. 2004). An antimicrobial finish is also applied to the textile material in order to protect the skin of the wearer and the textile substrate itself. Antimicrobial treatments have become more important for the textile material especially used in sportswear, active wear and casual wear since they can easily be contaminated by perspiration leading to bacterial growth and body odour. Wool has some resistance to bacteria. However, bacteria may attack stains left on wool, and if it is stored in a damp conditions bacteria will formed eventually and destroy the fibre. They damage the textile substrate and also may promote skin contamination and inflammation in sensitive people (Haug et al. 2006). Therefore, there is a need to inhibit the microbiological growth on textile substrate, in industrial as well as apparel usages. The wool fibre surface is covered by a covalently-bonded fatty layer, being responsible for the hydrophobicity of wool. Protease which can catalyse the degradation of different component of wool fibre is the most common enzyme used for wool fabrics. Pre-treatment of wool fabric with enzyme leads to increase in hydrophilicity nature of the wool fibre and increased swelling in nature (Julia et al. 1998).

Present day's textile processors preferred eco-friendly chemicals to impart antimicrobial finishing on textiles. *Neem* (*Azadirachta indica)* is well known in India and its neighbouring countries for most versatile medicinal plants. *Neem* possesses a wide spectrum of antibacterial action against Gram-negative and Gram-positive microorganisms (Chopra 1952; Almas 1999). At present, little has been reported of its use in woollen textiles as an antimicrobial agent. Few studies concerning application of *neem* extracts to cotton and cotton/polyester blends have been reported (Joshi et al. 2007, 2009; Vaideki et al. 2007). In this paper *neem* has been used for fixing on the wool fabric. This paper has been planned to study the effect of *neem* treatment on wool fabric with the following objectives:

To study the antibiotic properties of neem treated fabric.

To study the effectiveness of neem treatment on treated fabric.

2. MATERIAL AND METHODS

2.1 Materials

100% wool fabric with a weight of 260g/m^2 was used to carry out the experiment. For scouring of wool fabric, protease enzyme was used.

Methanol, citric acid and acetic acid were used for extraction and application of *neem* extract on the wool fabric. The test was carried against gram positive bacteria.

2.2 Preparation of *Neem* Leaf Extract

Fresh mature green *neem* (*Azadirachta indica*) leaves were collected, washed and dry in hot air oven at temperature 40°C. After complete drying they were made into a fine powder by crushing and grinding. Powder was then subjected to organic solvent (methanol) to get the concentrated methanolic extract. Extraction was carried out by SOXHLET method as per standard described by Mukherjee (2002).

2.3 Enzymatic Scouring

Enzymatic scouring was carried out by following the optimized standard conditions which were already standardized through research studies in the department. The standard conditions were concentration -0.5gpl; temperature-40°C; pH- 9 for 30 minutes and material to liquor ratio 1:30. Objective of enzymatic scouring was to remove impurities from the grey wool fabric with minimum strength loss and weight loss.

2.4 Antimicrobial Finish Application

Antimicrobial finish was applied on wool fabric with methanolic *neem* leaf extract. Finish was applied on fabric by two methods, that is, exhaust and pad dry cure method.

2.4.1 Exhaust Method: *Neem* extract in two concentrations (3 gpl and 5gpl) was set in a bath. The material to liquor ratio was taken as 1:20. The sample was entered into the antimicrobial bath with pH 5-6 adjusted with acetic acid. The bath temperature was raised to 70°C and it was kept at this temperature for 30 minutes. A post treatment was given with citric acid (8% on the weight of fabric) at room temperature. Finally samples were washed with cold water and dried. *Neem* treatment was given to enzymatically scoured as well as grey wool fabric.

2.4.2 Pad Dry Cure Method: The sample was immersed in 3 gpl and 5 gpl concentration of methanolic extract of neem for thirty minutes. After this sample was taken out and padded on two-bowl pneumatic padding mangle at a pressure of 2.5 psi with two dips and nips to give a wet pick up of 85%. The fabric was then dried at 80°C for 3 min and cured at 120°C for 2 min on a lab model curing chamber. A post treatment was given with citric acid (fixing agent) at room temperature. The samples were then again padded on a two-bowl pneumatic padding mangle at a pressure of 2.5 psi, dried at 80 °C and cured at 120 °C.

2.5 Determination of Add-on (%)

To estimate the actual amount of *neem* extract absorbed by the fabric, the total weight add-on % of the treated fabric was calculated using the following formula.

Add-on (%) = $[(W_2 - W_1)/W_1] \times 100$

Where W_1: weight of fabric before treatment (g)

W_2: weight of fabric after treatment (g)

2.6 Assessment of Antimicrobial Activity of Wool Fabric

The antimicrobial activity of control and finished samples was determined using AATCC-100-2004 test method. To assess the bacterial count of treated and untreated samples, bacterial growth in inoculated and incubated samples was determined through serial dilution (10^{-1}, 10^{-2} and 10^{-3}). From each of prepared dilutions, 0.1 ml was transferred onto the prepared petri dish and spread with the help of spreader under laminar flow. The petri dishes were placed in the incubator set as 30°C for 24 hours for the growth of bacteria. After 24 hours the colonies of bacteria were counted manually and with mean values percent reduction was calculated.

2.7 Assessment of Durability of Finish to Washing

The durability of the finish to washing was analyzed by washing all finished samples in the 'Launder-o-meter' by using standard ISO: 6330-1984E. Soap solution was prepared by taking the following materials-

Standard soap (powder form) - 4 gpl

Sodium carbonate (Na CO) - 2 gpl

The fabric samples were then subjected to microbial agar and the bacterial growth was analyzed by serial dilution carried out after incubation.

Table 1: Add-on (%) of antimicrobial treatment on wool fabric by different methods

Methods of application		*Exhaust method*		*Pad dry cure method*	
		Increase in weight (%)		*Increase in weight (%)*	
Name of extract	*Conc.(gpl)*	*Neem treated grey fabric*	*Neem treated scoured fabric*	*Neem treated grey fabric*	*Neem treated scoured fabric*
Neem extract	3	1.78	1.92	2.83	2.98
	5	2.92	3.08	3.36	3.52

3. RESULTS AND DISCUSSION

It is evident from the results obtained from Table 1 that when grey wool fabric treated with 3gpl *neem* extract by exhaust method, add on weight was observed 1.78 % which increased to 2.92 % when treatment was given with 5gpl extract concentration. But add on weight was observed 1.92 % when herbal treatment was given to enzymatically scoured wool fabric with 3 gpl *neem* extract and increased up to 3.08 % with 5 gpl *neem* extract concentration by same method.

Data illustrates that when *neem* treatment was given by pad dry cure method on grey wool fabric then add on weight was 2.83 % for 3gpl extract concentration which increased to 3.36 % for sample treated with 5gpl concentration. Whereas, add on weight was obtained 2.98 % when *neem* treatment was given to enzymatically scoured wool fabric with 3 gpl *neem* extract and increased up to 3.52 % with 5 gpl *neem* extract concentration.

It can be concluded from the data presented in Table 1 that as the concentration increases add on percentage was also increased. This may be due to the reason that more amount of extract was absorbed by the fabric in more concentrated finish bath. Add on percentage was also increased when treatment was given on enzymatically scoured fabric as compared to grey fabric. It may be due to the fact that enzymatic scouring increases the absorbency of the fabric by breaking the covalently-bonded fatty layer on the wool fibre. The results are supported by Feldtman and Mcphee (1964). They reported that enzymes increase the absorbency of wool fiber. The findings are in accordance with Lmmayappan and Jeyakodi Moses (2007).They also reported that enzyme treatment improves the finish add-on (%).

Table 2 shows that wool fabric treated with *neem* extract was found to have good resistance to bacterial attack. It was observed that there was confluent lawn of growth in control sample. After the treatment of grey wool fabric with 3gpl *neem* extract by exhaust method, percent bacteria reduction value was 90.75% which increased to 95.02 % with 5gpl concentration. Percentage reduction value was further increased to 96.72 and 96.67% with 3gpl and 5gpl *neem* concentration respectively, when treatment was given to enzymatically scoured wool fabric.

Data also indicates that percent bacteria reduction value of *neem* treated grey wool by pad dry cure method follow the same pattern as followed by samples finished with exhaust method.

From the above observations it can be concluded that *neem* treated scoured wool samples showed very good percentage of bacterial reduction as compared to *neem* treated grey wool samples. It may be due to reason that enzymatic

Table 2: Bacterial reduction of treated and untreated wool fabric by quantitative method

Method of application	*Conc. (gpl)*	*Percentage bacterial reduction(%) in neem treated wool fabric*									
		Neem treated grey fabric					*Neem treated scoured fabric*				
Dilutions →	-	10^{-1}	10^{-2}	10^{-3}	*Mean (10^2)*	*% reduction*	10^{-1}	10^{-2}	10^{-3}	*Mean (10^2)*	*% reduction*
Exhaust	3	107	9	1	9.9	90.75	14	Nil	Nil	4.6	96.72
	5	101	5	Nil	5.03	95.02	9	Nil	Nil	3.0	96.67
Pad dry cure	3	119	18	1	13.3	88.83	14	Nil	Nil	4.6	96.70
	5	100	7	Nil	5.66	94.34	10	Nil	Nil	3.3	96.72

Grey untreated fabric (control) Confluent lawn of growth

* Percentage reduction was calculated on the bases of mean value

scouring remove the natural impurities and increase the absorption rate of antimicrobial agent. Ammayappan and Jeyakodi Moses (2011) supported the finding that pre-treated wool fibre with formic acid prior to aloe vera and turmeric application shows better antimicrobial activity as compared to untreated wool. Ash Demir et al. (2010) also reported that enzymatic treatment remove the fatty bonded layer of wool fibre and promote the chitosan absorption rate, hence antimicrobial activity.

It was also concluded from the above results that by increasing the extract concentration percent bacterial reduction progressively increased. These findings are in line with the results reported by Joshi et al. (2007) that as the concentration of *neem* seed extract increases the zone of inhibition increase up to 5 % extract concentration and then decreases. It was also observed that exhaust method was more effective as compared to the pad dry cure method. It was supported by the findings of Mahesh et al. (2011) they reported that for coating of herbal extract, the exhaust method was found to be effective and suitable than dip coating method.

Table 3 shows the effectiveness of *neem* treated wool fabric by exhaust method in respect to washing. It was observed that when grey wool fabric was treated with 3gpl *neem* extract by exhaust method, % bacterial reduction was found 90.75 % which increased to 95.02 % with 5gpl concentration. Percentage reduction value for *neem* treated grey wool fabric remained same up to 10 wash cycles. After 15 washes % bacterial reduction was decreased. At the end of 20 wash cycles the % bacterial reduction value reached to 67.25 % for 3gpl and 74.61 % for 5gpl neem treated grey wool fabric.

It was further indicated that when the *neem* treatment was given to enzymatically scoured wool fabric, very good resistance to bacteria was found. Effectiveness of *neem* treated scoured wool fabric was assessed after different number of washing cycles and found that good resistance to bacterial attack up to 15 wash cycles. After 20 wash cycles the % reduction value decreased to 72.11 % for 3gpl and 72.60 % for 5gpl *neem* treated scoured wool fabric.

The data in the Table 4 shows the effect of washing on *neem* treated wool fabric by pad dry cure method. It was observed that samples finished with pad dry cure method follow the similar trend as observed by samples finished with exhaust method. When grey wool fabric was treated with 3 gpl and 5gpl *neem* extract concentration, percentage bacterial reduction was remained same up to 10 washes. After 10 washes % bacterial reduction was gradually decreased. At the end of 20 wash cycles the % bacterial reduction value was further decreased and reached to 74.24 % for 3gpl and 79.48 % for 5gpl *neem* treated grey wool fabric.

When the *neem* treatment was given to enzymatically scoured wool fabric exceptionally good resistance to bacteria was found. When enzymatic scoured wool fabric treated with 3gpl and 5gpl *neem* extract concentration, % bacterial reduction was found 96.70 and 96.72%

Table 3: Effect of washing cycles on neem treated wool fabric by exhaust method in terms of % bacterial reduction

		Percentage of bacterial reduction (%) by exhaust method									
Treatments→		*Neem treated grey fabric*					*Neem treated scoured fabric*				
Dilutions→		10^{-1}	10^{-2}	10^{-3}	*Mean (10^2)*	*% reduction*	10^{-1}	10^{-2}	10^{-3}	*Mean (10^2)*	*% reduction*
Washing cycles	*(conc.) gpl)*										
0	3	107	9	1	9.90	90.75	14	Nil	Nil	4.6	96.72
	5	101	5	Nil	5.03	95.02	9	Nil	Nil	3.0	96.67
5	3	107	9	1	9.90	90.75	14	Nil	Nil	4.6	96.72
	5	100	5	Nil	5.03	95.02	9	Nil	Nil	3.0	96.67
10	3	107	9	1	9.90	90.75	14	Nil	Nil	4.6	96.72
	5	101	5	Nil	5.03	95.02	9	Nil	Nil	3.0	96.67
15	3	121	10	2	14.03	88.41	16	Nil	Nil	5.3	96.69
	5	116	8	1	9.86	91.5	11	Nil	Nil	3.6	96.67
20	3	145	38	6	47.5	67.25	19	4	1	53.0	72.11
	5	142	34	6	36.06	74.61	18	3	1	49.3	72.60

Grey untreated fabric (control) Confluent lawn of growth

* Percentage reduction was calculated on the bases of mean value

Table 4: Effect of washing cycles on neem treated wool fabric by pad dry cure method in terms of % bacterial reduction

Treatments →		*Percentage of bacterial reduction (%) by pad dry cure method*									
		Neem treated grey fabric					*Neem treated scoured fabric*				
Dilutions →		*10^{-1}*	*10^{-2}*	*10^{-3}*	*Mean (10^2)*	*% reduction*	*10^{-1}*	*10^{-2}*	*10^{-3}*	*Mean (10^2)*	*% reduction*
Washing cycles	*(conc.) (gpl)*										
0	3	119	18	1	13.3	88.83	14	Nil	Nil	4.6	96.70
	5	100	7	Nil	5.66	94.34	10	Nil	Nil	3.3	96.72
5	3	119	18	1	13.3	88.83	14	Nil	Nil	4.6	96.70
	5	100	7	Nil	5.66	94.34	10	Nil	Nil	3.3	96.72
10	3	119	18	1	13.3	88.83	14	Nil	Nil	4.6	96.70
	5	100	7	Nil	5.66	94.34	10	Nil	Nil	3.3	96.72
15	3	132	22	2	18.4	86.07	18	Nil	Nil	6.0	96.67
	5	106	16	1	12.2	88.50	14	Nil	Nil	4.6	96.72
20	3	162	49	6	41.73	74.24	25	3	1	51.6	79.39
	5	159	42	4	32.63	79.48	23	2	1	47.6	79.28

Grey untreated fabric (control) Confluent lawn of growth

* Percentage reduction was calculated on the bases of mean value

respectively. Percentage bacterial reduction remained same up to 15 washes. After 20 wash cycles the % reduction value decreased and observed 79.39 % with 3gpl and 79.28 % with 5gpl *neem* extracts concentration for *neem* treated scoured wool fabric.

It can be concluded from the data presented in Tables 3 and 4 that *neem* treated scoured wool fabric showed very good antibacterial activity as compared to neem treated grey wool fabric. These findings are in line with the results reported by Ammayappan and Jeyakodi Moses (2009). They found that there were no bacterial and fungal growth in the finished fibrous substrates up to 20 washings and after 25 washings two bacterial and two fungal colonies were observed in wool/ rabbit hair substrate. Ash Demir (2010) also reported that wool samples pretreated by enzymes prior to finish application showed much better antimicrobial activity and washing stability than untreated wool fabric. Enzymes treatment promoted absorption, leading to improved durability. As the concentration of extract increases, % bacterial reduction also increased for *neem* treated grey wool sample, whereas in *neem* treated scoured wool fabric concentration did not affect the % bacterial reduction value up to 15 washing cycles.

CONCLUSION

Neem application has been done successfully by exhaust and pad dry cure method on wool fabric. Antibacterial activity of *neem* treated wool fabric was studied by quantitatively. Effectiveness of finish was also accessed after different washing cycles. From the present study following conclusions were derived–

1. As the concentration of extract increases % bacterial reduction was also increased.
2. Enzymatically scoured wool fabric treated with *neem* extract shows very good antimicrobial activity than *neem* treated grey wool fabric.
3. The antimicrobial effect on the *neem* treated wool fabric sample was durable even after 20 washing cycles.
4. Since the active *neem* ingredient extracted from *neem* leaves can serve as appropriate alternative for eco-friendly antimicrobial finish to conventional harmful chemicals in present use.

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