

Sustainable development of essential oils coated antimicrobial cellulosic fabric

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Novel finishing of fabrics through sustainable route has always raised demand for eco-friendly textiles and apparels intended for hygiene and medical applications. This research reports the antimicrobial activity of four essential oils (eucalyptus, clove, mint and neem oil) at 10 to 60 μ l against bacteria *S. aureus* and fungi *A. niger*. Neem oil showed minimum zone of inhibition (0.7mm) against bacteria followed by clove oil (1mm) and mint oil (3.16mm) at 60 μ l. Eucalyptus oil showed maximum zone of inhibition (7 mm) against *S. aureus*. The antifungal activity of neem oil was found to be negligible, whereas clove and mint oil had 3 to 3.16mm of zone of inhibition. Again, eucalyptus oil showed maximum antifungal activity (4.15mm) at 60 μ l. The reduction in bacterial colonies was 75% in case of eucalyptus oil, 56% by clove oil and 37% by mint oil. Neem oil efficacy was lowest against fungi. Reduction in fungal growth was found to be highest by eucalyptus oil (85%) at 60 μ l. Spray technique was done to finish fabric with selected oil and fabric properties were tested. The structural properties of finished fabric were not affected by finish although fabric thickness increased due to deposition of oil layer. The finish decreased drape coefficient of finished fabric and showed marginal effect on water vapour transmission and air permeability thus increasing comfort properties. There was marginal reduction in tensile strength of essential oil treated fabric however abrasion loss % showed better resistance against abrasion. Our findings highlight the development of sustainable cellulosic fabric using plant-based potential antimicrobial agents for finishing.

Keywords: Antimicrobial activity, Essential oils, Tensile strength, Water vapor transmission, Air permeability

INTRODUCTION

Since prehistoric times, medicinal plants have been used worldwide as natural remedy to cure various health problems including asthma, skin allergies, respiratory, urinary and cardiovascular diseases [1, 2]. According to the Botanical Survey of India, more than 8,000 indigenous medicinal plants are found in India. Medicinal plants are considered important and promising sources for traditional healing therapies, medications and have therapeutic benefits [3]. They contain several important phytochemicals such as flavonoids, alkaloids, tannins and terpenoids. These phytochemicals contain various disease-curing properties like antimicrobial, non-inflammatory, antioxidant and antiviral effects. Earlier, the crude extracts from leaves, roots, stem, flowers and fruits of medicinal plants were utilized [4]. However, recent advances in natural medicinal remedies derived essential oils as they are concentrated natural extracts with highly volatile secondary plant metabolites [5, 6]. These active bio components present in essential oils can be further utilized for diverse applications for development of functional clothing, anti-odor apparels and nano finished textiles [7, 8].

The recent global pandemic has hard hit the lifestyle. Consumers nowadays have elevated awareness to control the spread of infection through clothes or textile materials and demand hygienic or safe textiles [9]. The area of contact with skin during wear, body secretions, fabric composition and age of consumer are the determinant parameters for growth of microbes on clothes [10-12]. It is posed that susceptibility for microbial degradation of natural fibres is due to the generic composition of fabric (cellulose/ proteins/ polysaccharides), elevated body temperature, high humidity (70%–90%) and sweat secretion [13, 14]. Previous studies reported that bacteria like *Staphylococcus aureus*, *Escherichia coli* leads to body odor, skin irritation and hospital-acquired infections whereas fungi like *Aspergillus niger*, *Penicillium* cause loss in tensile strength and weight of cotton fabrics [9, 15]. Therefore, there is emerging demand for antimicrobial finished apparels that have significant impact against bacteria and viruses, environment friendly and safe [16].

A lot of chemicals mainly mineral and silver based are used to develop antimicrobial finished fabrics. These textiles are making their way into the

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global market and may affect health and environment [17]. The commonly used antimicrobial agents in textiles are triclosan, and quaternary ammonium compounds which are found to be poisonous and cause skin inflammation [9]. The plant-based antimicrobials can be the possible alternative in combating microbial growth as they are environment friendly, safe and nontoxic. Review from previous studies reported that bioactive compounds (gallic acid, cypellocarpin A, eucaglobulin, cuniloside, azadirachtinin, mentha) present in species of eucalyptus (*Eucalyptus globulus*), mint (*Mentha arvensis*), clove (*S. aromaticum*) and neem (*Azadirachta indica*) have benefits as antimicrobial, antioxidant and anti-inflammatory [18-21]. Earlier studies reported that aloe gel extract was used for treating cotton fabric for antimicrobial activity [22], chamomile, sage and green tea [23], and pine bark (*Pinus brutia*) [24], against bacteria (G+ and G-), yeasts and fungi.

The current research project tests the efficacy of antibacterial and antifungal properties of essential oils on organic cotton fabric. In addition, the antimicrobial finished fabric was evaluated on various performance parameters including structural, strength and comfort properties, adds to the literature. Therefore, this research reports the efficacy of essential oils which produce an eco-friendly antimicrobial finished organic fabric and the fabric performance finished fabric with essential oil.

MATERIALS AND METHODS

Materials

In the present study, an extensive investigation of literature was done on researches related to antimicrobial efficacy of plant-based herbs and their comparative constituents. Based on the review, eucalyptus (*Eucalyptus globulus*), neem (*Azadirachta indica*) mint (*Mentha arvensis*) and clove (*Syzygium aromaticum*) oils were procured from Central Institute of Medicinal and Aromatic Plants, Pantnagar, Uttarakhand. Plain-woven organic cotton fabric of GSM 146.34 g/m² was procured from Paramount Syntex Pvt. Ltd., Ludhiana (Punjab).

Test inocula

The strains of *Staphylococcus aureus* (Gram positive bacteria) and *Aspergillus niger* (Fungi) were taken as the representative of bacteria and fungi. These pathogenic strains are highly infectious that invade the textile materials. *Staphylococcus aureus* and *Aspergillus niger* multiply at a faster rate in the skin regions which have increased pH and prolonged wetness [25]. The antimicrobial tests were

conducted against the bacteria and fungi to evaluate the effectiveness of essential oils against microbes. The strains were collected from Department of Microbiology, College of Veterinary and Animal Sciences, GBPUAT, Pantnagar.

Antimicrobial analysis

The percentage reduction rates (% R) in growth of bacteria and fungi on the fabric was evaluated by Colony Formation Units (CFU) of selected microorganisms, i.e., *S. aureus* and *A. niger* using AATCC test method 100:2004. Both the untreated and treated organic cotton fabric was tested for antimicrobial activity against the pathogenic strains of bacteria and fungi. The test inoculum was prepared which consist of nutrient broth culture having 2.7×10⁵/ml colony forming units (CFU) of bacteria/fungi. Test inoculum of 1.0 ml was laden on fabric samples of 4.8 cm ± 0.1 cm diameter. The strains were cultivated in nutrient agar and test samples were put in contact for 24 h. The reduction in number of colonies was calculated by counting viable colonies of bacteria/fungi on the agar plate using following formula:

$$R = 100\% \times (B-A)/B$$

where A - number of bacteria recovered from the inoculated specimen after 24 h; B - number of bacteria recovered instantly after inoculation, 0 h.

For qualitative tests, agar diffusion test of textile fabrics (SN 195920-1992) was conducted where nutrient agar was prepared and test specimens (28.6mm) and control samples which were cut circularly were placed in nutrient agar for 24 h. The plates were incubated for 18 to 24 hours at 37°C. The formula used for calculating the zone of inhibition:

$$W = T-D/2$$

where T - total diameter of the test specimen and clear zone measured in mm; W - width of the clear zone of inhibition; D - diameter of the test specimen.

To determine the washing fastness of the finish, test on finished fabric samples was done in "Laundrometer" to determine the efficacy of antimicrobial finish after ten washes with the procedure ISO 2, IS 15370:2005. The recipe for laundry test was:

Laundry soap solution - 5 g/l;
Sodium carbonate (Na₂CO₃) - 2 g/l;
M:L ratio - 1:50;
Temperature - 35–37 °C ;
Time - 30 min.

Evaluation of properties of finished fabrics

Structural properties

The physical properties were tested as per their fabric construction which includes fabric count, fabric thickness, cover factor and fabric weight per unit area.

a) *Fabric count* - It was determined with calibrated square magnifying glass called "Pick glass" and expressed in ends \times picks/inch².

b) *Fabric thickness* - The samples for fabric thickness were tested as per ISI (IS: 7702-1975) method by using Shirley's thickness tester.

c) *Fabric weight* - The fabric weight of both samples (treated and untreated) was determined as per IS: 1964-1970 and calculated by the formula:

$$\text{Fabric weight (g/m}^2\text{)} = W/A$$

where W is the fabric weight measured in grams and A is the area of the fabric in meter².

d) *Cover factor* - Cover factor is determined by calculating threads/inch (both warp and weft) in the fabric as well as by counts of the thread. It is defined as the portion of the fabric area covered by both warp and weft yarns. It was calculated by the formula:

$$K = n/\sqrt{N}$$

where K= cover factor; n= threads/inch; N = cotton count.

Comfort/end use properties

The comfort properties were tested for the treated fabric finished with essential oil and comparison was done with the untreated fabric sample. The comfort properties include water vapor transmission rate, air permeability, bending length, drapability and thermal insulation.

a) *Water vapor transmission*. Water vapor transmission of the fabric samples was checked by the water dish method (ASTM: E 96-00). The distilled water was maintained in a test dish at a level of $3/4 \pm 1/4$ from the specimen in a controlled test chamber. Periodically weighing, determine the rate of water vapor which was calculated and expressed as G/m²/d.

$$WVT = (G/t)/A$$

where, WVT is water vapor transmission rate, G is weight change, t is time during which G occurred and A is the test area (cup mouth area).

b) *Air permeability*. Air permeability of the specimen was done on Eureka air permeability tester and the sample was cut in a circular shape according to the area (10 cm²). The pressure was set at 5KPa into the pressure vessel. The amount of air passed through sample was read on a manometer in cubic

centimetre/ minute for treated and untreated fabric samples.

c) *Drapability*. Drape meter was used to check the drapability of the treated fabrics and expressed as "Drape coefficient". The paper was cut according to the template and the drape coefficient was calculated with the help of following formula:

$$F = \frac{W_s - W_d}{W_D - W_d}$$

where F= drape coefficient; WD = area of specimen which is equivalent to the area of weight of the paper; Wd - area of supporting disc, which is equivalent to the area of weight of the paper; Ws = area equal to the projected area of specimen. Here, 'F' value determines the drapability, i.e., small 'F' value indicates better drapability while large value of 'F' indicates the poor drapability.

Qualitative assessment of antimicrobial activity of essential oils

Antimicrobial activity of selected essential oils of eucalyptus, clove, mint and neem was examined at 10–60 μ l against *S. aureus* and *A. niger*. The essential oil of clove, mint and neem at 10-30 μ l was not sensitive against strains of *S. aureus* and *A. niger* (Fig. 2).

d) *Thermal insulation*. The thermal insulation (clo value) of finished fabrics was assessed using Sasmira thermal conductivity apparatus. The fabric sample was cut according to the template and was placed on a hot plate. The round plate was finally used to cover the sample and the temperature was allowed to fall to 50°C. The hot plate was allowed to cool down from 51°C to 50°C and time was measured with the help of a standard stop watch. Average time was divided by two and then divided with 250 to determine the clo value.

Strength properties

The strength of the fabric is an important parameter. The strength of the treated samples was calculated for tensile strength and abrasion resistance.

a) *Tensile strength*. The tensile strength of the fabric was measured with the instrument named "Electronic tensile tester". It was based on the principle of constant rate of extension and ASTM test method D-1682-94 (1994) and Ravelled strip test method of ISI (IS: 1969-1968) was used to determine the breaking strength of samples (25 cm \times 5 cm) in both warp and weft direction.

b) *Abrasion resistance*. The fabric resistance against abrasion was measured on a "Martindale abrasion tester". The abrasion loss % of the test samples was calculated using the formula:

$$\text{Abrasion loss \%} = \frac{\text{wt. of sample before abrasion} - \text{wt. of sample after abrasion}}{\text{wt. of sample before abrasion}} \times 100\%$$

wt. of sample before abrasion after abrasion

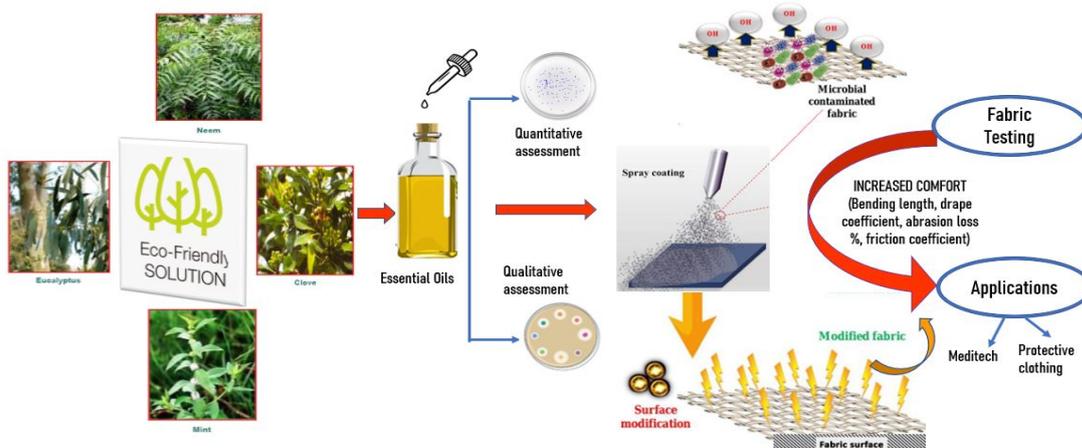


Fig. 1. Schematic development of essential oils coated antimicrobial cellulosic fabric

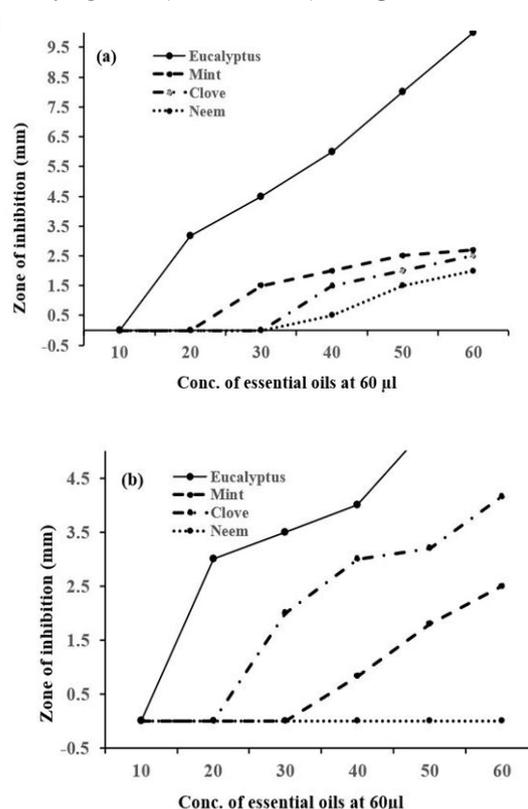
RESULTS

Fig. 1 demonstrates that the leaves of herbal plants, namely eucalyptus, clove, mint and neem were used to extract the essential oils. All essential oils were assessed for antimicrobial activity through qualitative and quantitative assessment. The essential oil which exhibited best antimicrobial activity was selected for finishing the fabric and spray method was used to apply the antimicrobial finish. The oil got adsorbed on the fabric surface and fabric was tested for comfort properties. Results indicated the increase in comfort of the fabric properties which leads to its application in medical textiles and protective clothing.

A significant zone of inhibition was formed against *S. aureus* at 60 μl by essential oils of neem (2.0 mm), clove (2.5 mm), mint (2.7 mm) and eucalyptus (10 mm) (Fig. 2). In case of *A. niger*, eucalyptus oil inhibited fungi at a minimum amount of 20 μl while clove and mint oil was not very sensitive till 30 μl . However, there was no effect of neem oil against *A. niger* even at higher concentration (60 μl). The essential oil of mint and clove oil displayed only moderate antifungal activity with zone of inhibition 2.5 mm and 4.16 mm, respectively, at 60 μl (Figs. 2 and 3). Eucalyptus oil formed highest antifungal zone of inhibition (6.5 mm) at tested amount.

Based on the results, essential oils at 60 μl amount showed maximum antimicrobial efficacy, thus, highest amount of oils (60 μl) was used for quantitative test. In general, eucalyptus oil was more effective among essential oils and showed maximum antibacterial and antifungal activity.

Fig. 2. Qualitative assessment of antimicrobial activity against a) *S. aureus*, b) *A. niger*.



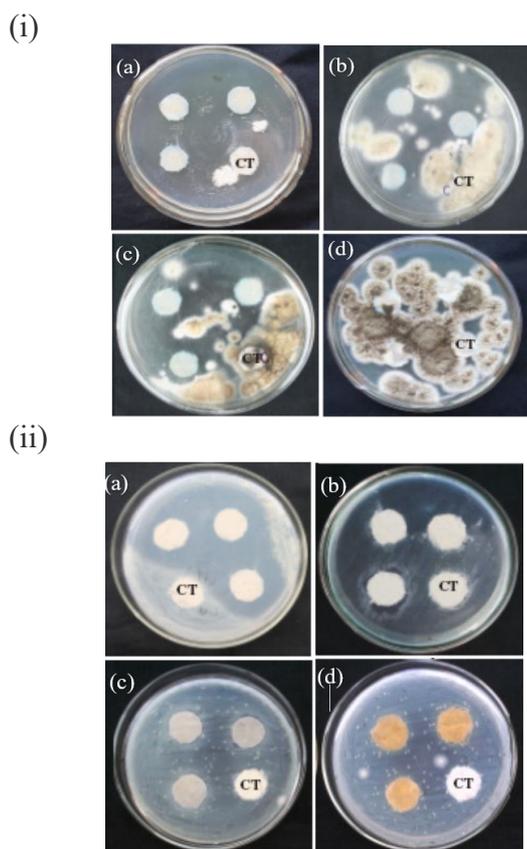


Fig. 3. Inhibition zone formed against i) *A. niger*, ii) *S. aureus* at 60 μ l by (a) eucalyptus oil (b) mint oil, (c) clove

Quantitative assessment of antimicrobial activity of essential oils

The quantitative antimicrobial test (ASTM:E 2149-01) revealed that active oils were eucalyptus, clove and mint which managed to achieve reduction in bacterial and fungal colonies. In case of fabric finished with clove oil, the percent reduction of bacteria was much higher (56%) than for mint (37%) and neem oil (14%). The oil of eucalyptus showed excellent resistance against bacteria *S. aureus* (75%) (Fig. 4). Notably, eucalyptus oil was highly effective against fungi *Aspergillus niger* with 84% fungal growth reduction. The percent reduction of fungal colonies with clove oil was marginally higher (27%) than mint oil (23%). There was no percent reduction in the growth of fungal colonies in case of neem oil.

For the fabric finished with eucalyptus oil and clove oil, the decrease in the percent reduction of

microorganism after five washes was in the range of 0.5-0.79%. This represents the good efficacy in resisting the microbial growth, i.e., against both bacteria and fungi.

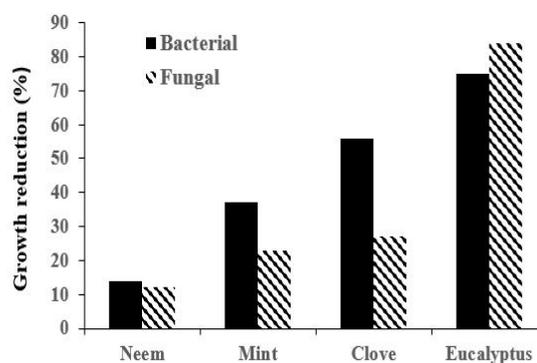


Fig. 4. Quantitative assessment of antimicrobial activity of essential oils

A marginal decrease in the percent reduction of *Staphylococcus aureus*, (Gram+ve bacteria) and *Aspergillus niger* (fungi) is noted in mint oil after laundering. In case of neem oil, the essential oil on the fabric was washed off. This results in decrease in antimicrobial reduction and less effectiveness of the finish. Overall, it can be inferred that after 5 washes of less than 1% reduction in microbial growth indicates a temporary finish.

Structural properties

Results from Table 1 indicate no difference between the fabric count of untreated and treated organic cotton samples. The fabric cover factor is good as densely woven plain weave fabric contributed to good covering power and application of essential oil did not indicate any change for the finished cotton fabric. Statistically, oil caused non-significant difference between thicknesses of fabric samples.

It is noticeable that there was increase in the weight (GSM) of finished cotton fabric (255.70 g/m^2) compared to unfinished fabric (146.34 g/m^2). The added weight or increased GSM of the fabric resulted due to the adsorption of essential oil in the interstices of fabric structure.

Table 1. Structural properties of essential oil treated fabric with untreated samples

Fabric samples	Fabric Count (ends \times picks/inch ²)	Fabric Thickness (mm)	GSM (g/m^2)	Cover Factor	
				Warp	Weft
Untreated	64 \times 64	0.41	146.34	14.44	14.26
Treated	64 \times 64	0.42	255.7	14.44	14.35

Comfort properties

a) *Water vapor transmission (WVT)*. A marginal decrease in WVT was found in the finished fabric (1575.39 Gm/m²/d) compared to unfinished fabric (1595.90 Gm/m²/d). Water vapor reduction of the fabric samples revealed that essential oils were adsorbed by the cotton fabric as the interspaces within yarn/fabric structure were filled with oils thus, reducing water vapour transmission through fabric.

b) *Air permeability*. The air permeability of untreated and treated organic cotton fabric samples was 10.75 ccf/min and 10.53 ccf/min, respectively. It is apparent from the results that there was slight decrease in the air permeability of finished sample. The reason may be that due to deposition of eucalyptus oil layer, the interstices or open spaces of woven sample were blocked or filled by oil and air permeability was decreased. Above results were supported by the study of Sarkar *et al.* (2002) that the air permeability of control fabric is higher (15.32cc/s/cm²) whereas 1% clove oil-treated fabric shows lower air permeability (12.57 cc/s/cm²) [26].

c) *Drapability*. While comparing the drape coefficient of unfinished and finished fabric samples with eucalyptus oil finish, it was found that drape coefficient of finished fabric (0.25%) was less (approx. half) than the unfinished sample (0.53%). Therefore, the drapability of treated fabric sample was more as lower the drape coefficient, higher will be drapability. The reason may be that after oil finish treatment, the smoothness and softness of fabric was increased. The woven and loop pile samples became lesser stiff as compared to untreated woven and loop thus in turn influencing improved drapability.

Statistically it was found that the effect of oil caused significant difference at 1% level between the drapability of unfinished and finished fabric samples

d) *Thermal insulation*. It was found that the thermal insulation of treated fabric (0.45 clo) was marginally higher more than the untreated fabric sample (0.39 clo).

According to Tortora (1982) [27], thermal insulation is related to heat conductivity of fabric, fabric thickness and entrapment of still air by the fabric. In the present study, the application of herbal oil finish may have sealed the interstices between the warp and weft of woven fabric, thus enhanced and trapped the level of still air. Therefore, thermal insulation of the treated sample was found more as compared to untreated fabric sample.

Strength properties

a) *Tensile strength*. The results indicate that tensile strength of finished fabric in warp direction was slightly reduced (9.43 kgf/cm) than that of unfinished fabric (9.67 kgf/cm) whereas a moderate drop in tensile strength of finished fabric (4.95 kgf/cm) when compared to unfinished cotton fabric (6.51 kgf/cm) was observed in the weft direction. The factors that lead to adsorption of oil in fabric structure are fibre length, crimp and cross-sectional shape. In the present study, staple length, bean shaped cross section and crimp of cotton fibres led to the absorption of essential oil finish on treated fabric. The pH of the eucalyptus oil was slightly acidic (5.1-5.6) and as cotton fibres are sensitive to acids, the adsorption of oil in the cotton structure anticipated reduction in tensile strength.

b) *Abrasion loss percentage*. While comparing the abrasion loss percent of samples, results indicated that abrasion loss percent of unfinished sample (2%) was marginally higher than the finished fabric (1.62%).

The reason may be that after application of oil finish on treated sample, the fabrics became soft leading to less short fibre ends on the surface than the unfinished fabric sample, hence, abrasion loss of treated sample was also reduced.

CONCLUSION

In today's competitive world, one of the top priorities for many textile manufacturers is finishing of cotton textiles with antimicrobial properties for hygiene applications. Results indicated that eucalyptus oil showed maximum antimicrobial activity against both bacteria and fungi when compared with clove, mint and neem oil. The efficacy of neem oil against bacteria and fungi was lowest and no percent reduction in microbial colonies was seen. The antimicrobial efficacy was not much reduced (less than 1%) after five washes, showing a temporary finish. The tensile strength of the finished organic cotton fabric was slightly dropped compared to that of unfinished fabrics. However, it can be inferred that statistically essential oils caused non-significant difference on count and cover factor of finished fabric. However, water vapor transmission and air permeability are little bit reduced indicating oil deposition in fabric interstices. Compared to other finishing, this essential oil finish on cotton fabric introduced suppleness and reduced the drape coefficient and abrasion loss %.

The research findings establish the possibilities in averting the transmission of microbial infections through clothes or textile materials in most safe and eco-friendly way. Furthermore, utilization of plant-based antimicrobials for finishing textiles are totally safe, both for human usage and environment when compared with synthetic chemicals or compounds. The easy availability of the plant-based herbs and method to apply or replenish finish is an add on advantage for the commercial use of the finish.

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