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Combined antimicrobial and aroma finishing treatment for cotton, using micro encapsulated geranium (*Pelargonium graveolens* L' Herit. ex Ait.) leaves extract

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The methanolic extract of geranium, *Pelargonium graveolens* L' Herit. ex Ait. leaves was used to impart antimicrobial and aroma finishing to the cotton fabric. The extract was microencapsulated using two different methods namely coacervation-spray drying and direct spray drying techniques. The results indicated that the direct spray drying method produced smaller capsules with 5 µ size in powder form and 12.5 µ in aqueous form compared to the 7.5 µ and 17.5 µ respective capsule sizes produced by coacervation-spray drying method. The microencapsulated geranium leaves extract treated cotton fabrics showed antimicrobial efficacy against both *Staphylococcus aureus* and *Escherichia coli*. However, the Gram positive *Staphylococcus aureus* was more susceptible to geranium extract compared to *Escherichia coli*. The microcapsule treated fabric retained 50-60% aroma after 10 washes compared to the nil retention property of pure geranium extract treated fabric.

Keywords: Antimicrobial, Aroma finish, Geranium, Micro encapsulation, Cotton fabric, Spray drying.
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Introduction

Textile materials are good carriers of various types of microorganisms and can cause health related problems to the wearer. In order to protect the wearer from such infection, the textile fabrics can be finished with antimicrobial agents. Several antimicrobial agents like Polyhexamethylene biguanide (PHMB), nano silver, triclosan, quaternary ammonium compounds, etc., are available for textile application. However, these are synthetic chemicals with prohibitive cost and also the durability of such chemicals on the textiles is not satisfactory. There are several studies in literature in which natural materials such as neem, *Aloe vera* and essential oils[1-3] have been used to give antimicrobial finishing to the textile materials. One of the major limitations in the natural materials based antimicrobial finishing is the non-durability of the finish since they cannot form any bond with the textile materials. Micro encapsulation of natural materials is one of the methods used to increase the durability of the antimicrobial finishing on the textile materials. In this technique, the active compounds are encapsulated using a wall material like modified starch, sodium alginate, gum acacia, etc. and applied on the textile materials.

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Aroma finish is the process by which textile materials are treated with the pleasant odour producing essential oils and aromatic compounds so that the wearer gets some beneficial effects. Various essential oils like lavender, rosemary and jasmine were used to give this type of finish. The pleasant smells created by the essential oils have pharmacological effects like antibacterial, antifungal, antiviral, etc. and mood elevating effects[4-6]. *In vitro* studies have shown that geranium based essential oil has antimicrobial as well as mind elevating effects. In this study, the possibility of using geranium leaf extract as such and upon micro encapsulation with gum acacia for giving antimicrobial and aroma finishes to the cotton fabric has been explored.

Materials and Methods

Fabric

Plain, 100% cotton woven fabric, made up of Ne60 combed yarn with 110 ends/cm and 80 picks/inch was desized (0.5% H_2SO_4), scoured (3% NaOH) and bleached (3% H_2O_2) prior to the application of the antimicrobial finish and blood repellent finish.

Collection of Geranium

The geranium (*P. graveolens*) leaves were collected from Kodaikanal hills during the month of December-January. It was shadow dried to the moisture content of below 10% and ground to powder. 20 g of the

geranium crude powder was then dissolved in 100 ml of methanol and kept for 24 h. It was then sonicated using Sonics Vibracell for 5 h to facilitate the complete extraction of the ingredients.

Micro encapsulation

Micro encapsulation of geranium extract was done by two methods using gum acacia as a wall material. In the first method, the geranium extract was encapsulated by coacervation technique followed by spray drying in to powder. In the second method, the core and wall materials were spray dried to get the microcapsules.

Coacervation-spray drying method

Ten gram of wall material was allowed to swell for half an hour by mixing with 100 ml of water. To this mixture, 50 ml of hot water was added, stirred for 15 min maintaining the temperature between 40°C and 50°C. Core material (10ml) was added and stirred at 300-500 rpm for further 15 min followed by drop wise addition of 20% sodium sulphate solution(10 ml) for 5-10 minutes. The stirrer speed was reduced and then 5ml of 17% formaldehyde was added.

The microencapsulated geranium extract was then converted to powder using a laboratory spray dryer (Model – LU222 Advanced of M/s Lab Ultima, India) operated in the co-current mode. The liquid feed rate was 2 ml/min through 0.7 mm diameter nozzle at 3 psi air pressure. The atomization was done in the aspirator with the vacuum of 35 mm water column. Spray drying was performed at an inlet air temperature (T_{inlet}) of 70°C, corresponding to an outlet air temperature (T_{outlet}) of 40°C. The powder was collected from I & II cyclones of the spray dryer and stored in air tight container for further applications.

Direct spray drying method

In this method, 10 g of wall material was allowed to swell for half an hour by mixing with 100ml of water. To this mixture, 50 ml of hot water was added, stirred for 15 min maintaining the temperature between 40°C and 50°C. Core material (10 ml) was added and stirred at 300-500 rpm for further 15 min. This solution is then directly spray dried into powder using similar processing condition mentioned in the coacervation-spray drying method.

Measurement of size of the microcapsule

The light microscope is used to measure the size and core-wall ratio of the microcapsules. The size is determined using a graduated ocular micrometer using 40× magnification.

Application on the fabric

The cotton fabric was padded with 10% aqueous solution of microencapsulated geranium powder and to attain a wet pick-up of 75%, dried and then cured at 100-120°C for 2 min. In order to fix the microcapsules 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. A control cotton fabric was produced by padding with pure geranium extract as per the above procedure followed by fixing with 10% citric acid.

Testing of antimicrobial efficacy of the fabrics

In this work, the qualitative agar diffusion test and the quantitative bacteria reduction through AATCC 100 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 bacteriostasis agar, 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.

AATCC 100 method

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. *S. aureus* (ATCC 6538) was used as a representative Gram positive organism and *E. 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 10º dilution was reported as "less than 100". The % reduction of bacteria by the specimen treatments was calculated using the following formula:

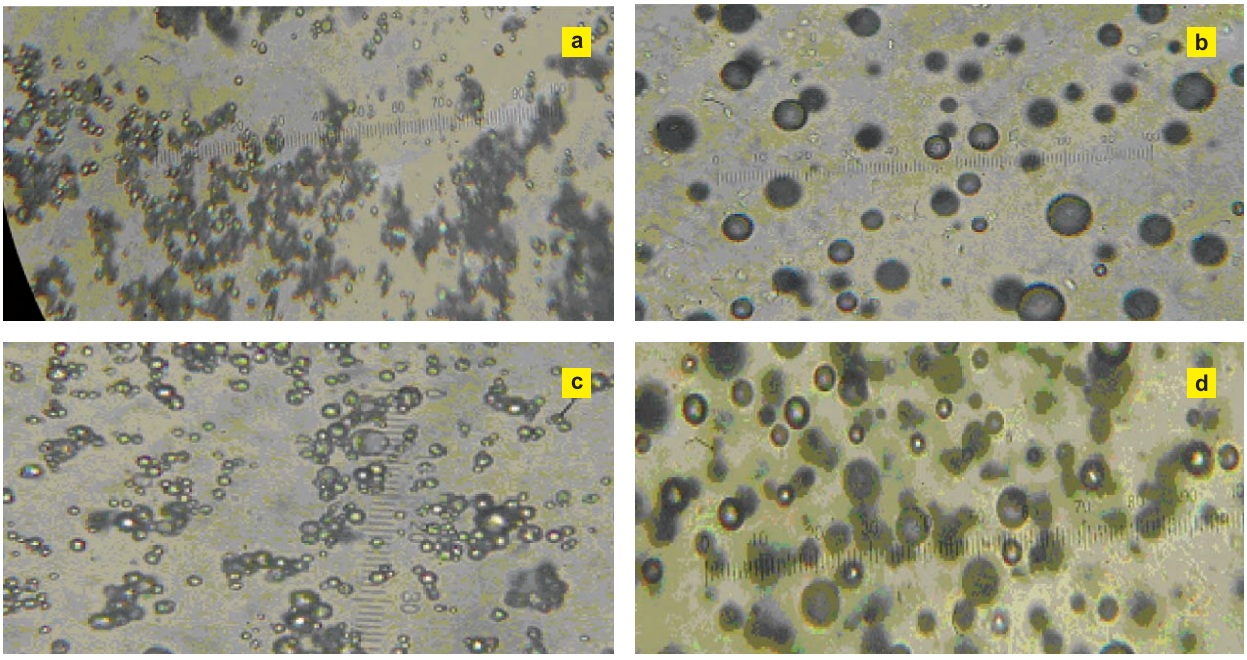

Plate 1—Geranium microcapsules (a) in powder; (b) in aqueous solution by coacervation-spray drying method; (c) in powder by direct spray drying method; (d) in aqueous solution by direct spray drying method

$100\ (\underline{B}\text{-}\underline{A})/\underline{B} = \underline{R}$

Where, $\underline{R}$ = % reduction

$\underline{A}$ = number of bacteria recovered from the inoculated treated test specimen swatches in the jar incubated over the desired contact period.

$\underline{B}$ = 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.

Durability of antimicrobial finishing

The durability of the finish is tested after 5, 10 and 15 washes as per AATCC 124-2001 method using home washing machine and compared with the fabric treated with pure geranium extract.

Aroma Testing

Aroma testing of treated fabric was done subjectively using 16 judges. The judges were graduate students with prior training to assess the odour. The judges were asked to rate each fabric on a likert scale of 0 (nil aroma) to 4 (highest aroma intensity). The overall rating for aroma intensity was then calculated by the following equation[7].

$$\text{Percentage aroma intensity} = \sum_{i=0}^{4} (S_i n_j/4n) \times 100$$

where n = Total number of judges

S_i = Judge's rating

4 = Highest rating

n_j = Number of judges giving rating S_i

Results and Discussion

Size of the microcapsules

Both the micro encapsulation methods, produced microcapsules of geranium effectively. However, there is a difference in the size, core to wall ratio and capsule concentration in the microcapsules produced by these methods. The microcapsules produced by the coacervation-spray drying method are shown in Plate 1a & b. The Plate 1a represents the capsules in powder form and Plate 1b represents the capsules in aqueous solution. Similarly, the microcapsules produced by direct spray drying method are shown in Plate 1c & d. SEM micrographs are shown in Plate 2. The results indicate that the coacervation-spray drying method produce 7.5 µ size capsule in powder form and 17.5 µ in aqueous solution. The direct spray drying method produces smaller capsules than the previous method with 5 µ size in powder form and 12.5 µ in aqueous form. The wall to core ratio of the capsules in aqueous form formed by coacervation-spray drying method is 1:2.3 compared to 1:1.6 of direct spray drying method. The direct spray drying method produces more number of capsules per unit surface in comparison to coacervation-spray drying method due to the formation of smaller capsules and optimum core to wall ratio of the capsules.

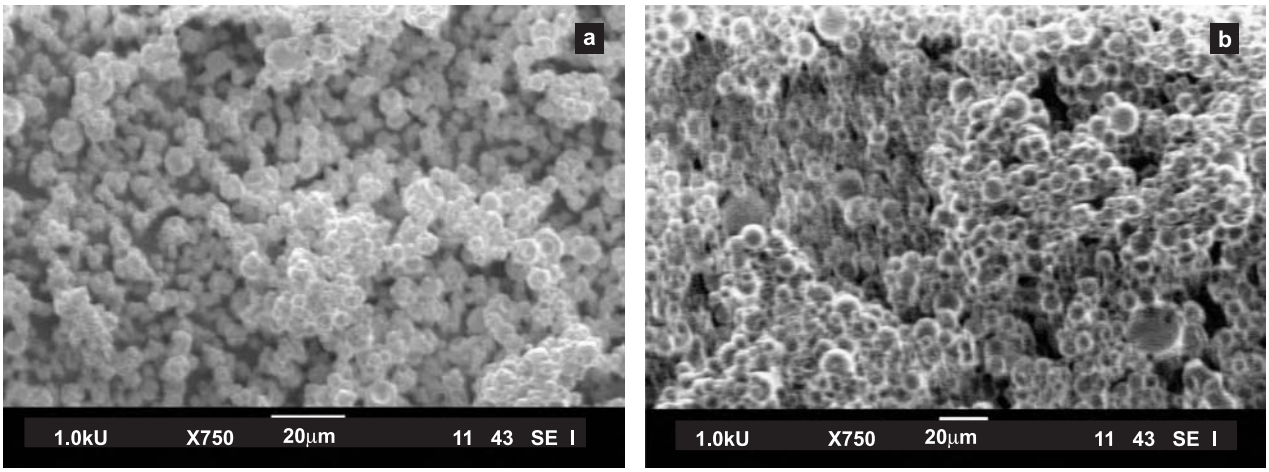

Plate 2—SEM images of microencapsulated Geranium powder-(a) At 750 × Magnification; (b) At 1000 × Magnification

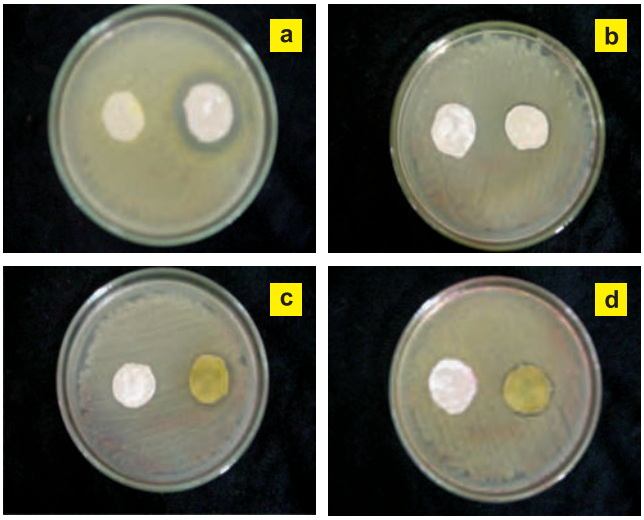

Plate 3—Agar diffusion test: Antimicrobial activity-(a) Pure geranium leaves extract treated fabric against *S. aureus*; (b) Pure geranium extract treated fabric against *E. coli*; (c) Microencapsulated (Direct spray dried) Geranium treated fabric against *S. aureus*; (d) Microencapsulated (Direct spray dried) Geranium treated fabric against *E. coli.*

Antimicrobial efficacy of the treated materials

The results of the agar diffusion test carried out on the treated and control fabrics are shown in Plate 3 and Table 1. The microencapsulated geranium extract and pure geranium extract treated fabrics showed antimicrobial efficacy against both *S. aureus* and *E. coli*. However, the *S. aureus* is more sensitive to geranium extract compared to *E. coli*. The Gram positive bacteria are more susceptible to natural herbal plant extracts compared to Gram negative bacteria. This fact is well documented by the previous studies also[8-10]. The presence of outer cell wall membrane in the gram negative bacteria acts as a barrier to the many antibiotics. This may be the reason for the low efficacy of the geranium extract against *E. coli*. The wash durability in terms of antimicrobial efficacy of the microencapsulated geranium extract treated fabrics is shown in Table 2. The result shows that the microencapsulated geranium extract based antimicrobial finish is durable up to 15 washes in comparison to direct geranium extract

Table 1—Bacterial zone of inhibition and reduction percentage of treated fabrics

Sample	Zone of Inhibition (mm) (Fabric Diameter = 20mm)	
	Staphylococcus aureus	*Escherichia coli*
Pure geranium leaves extract	36	32
Microencapsulated geranium extract-by coacervation-spray drying method	32	24
Microencapsulated geranium extract-by spray drying method	32	26

Table 2—Wash durability of the antimicrobial efficacy of the treated fabrics

Sample		Bacterial Reduction Percentage (%)			
		No wash	5 washes	10 washes	15 washes
Pure geranium extract	*S aureus*	99.99	65	20	0
	E. coli	78	40	0	0
Microencapsulated geranium extract-by coacervation-spray drying method	*Staphylococcus aureus*	92	95	80	70
	Escherichia coli	55	65	40	25
Microencapsulated geranium extract-by spray drying method	*Staphylococcus aureus*	94	97	85	77
	Escherichia coli	60	70	50	40

Table 3—Aroma retention capacity of the geranium leaves extract treated fabrics at different washes

Sample	Aroma retention intensity (%)			
	No wash	5 washes	10 washes	15 washes
Pure geranium extract	100	32.81	0	0
Microencapsulated geranium extract-by coacervation-spray drying method	75	80	56.25	15.62
Microencapsulated geranium extract-by spray drying method	82	85	60.94	25

treated fabric which shows activity only up to 5 washes. The micro encapsulation process of geranium extract leads to slow release of active core materials and preserve the durability for 15 washes. Between the two micro encapsulation processes employed, the direct spray drying method provide durable and effective antimicrobial efficacy due to the presence of higher concentration of capsules. Hence, it is possible to produce microcapsules of geranium without the use of chemicals like sodium sulphate and formaldehyde by direct spray drying method in comparison to coacervation-spray drying method.

Aroma retention

The percentage of aroma intensity of the treated and control fabrics are given in Table 3. The aroma intensity of direct geranium extract treated fabric is considered as 100 per cent. Based on the above, the aroma retention of treated fabrics is determined subjectively by the panel of experts. The results show that the direct spray dried microcapsule treated fabric retain as much as 50 to 60% of aroma even after 10 washes compared to the nil retention ability of the pure geranium extract treated fabric. The aroma of the core materials is released slowly in the microcapsules treated fabrics. The direct spray dried capsules treated fabric shows 25% higher aroma retention capacity after 15 washes in comparison to coacervation-spray drying method.

Conclusion

A combined antimicrobial and aroma finishing method using microencapsulated geranium leaves extract is developed for textile application. The direct spray drying technique of micro encapsulation provides uniformly-sized capsules with smaller size and optimum core to wall ratio compared to the coacervation-spray drying method. The treated fabrics showed antimicrobial efficacy against both *S. aureus* and *E. coli*. However, the geranium extract possesses effective antimicrobial efficacy against *S. aureus* compared to *E. coli*. The durability of the antimicrobial finish based on geranium leaves extract is increased from 5 washes to 15 washes by micro encapsulation technique. The use of geranium extract provides aroma finishing in addition to its antimicrobial finishing to the treated textile material in a single process.

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