

Microencapsulation of herbal extracts for microbial resistance in healthcare textiles

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Antimicrobial finish has been imparted to the cotton fabric using extracts of neem and Mexican daisy by direct application and by microencapsulation using pad-dry-cure method. To enhance the durability of antimicrobial finish to number of washes, the microencapsulation of herbal extracts has been done using phase separation / coacervation. Microcapsules are produced using herbal extracts as core and acacia as wall material. Structure of microcapsules has been evaluated using light microscopy with image analysis technique, the presence of microcapsules by scanning electron microscopy, the antimicrobial efficacy by quantitative method in terms of bacterial reduction, and the wash durability of antimicrobial activity by AATCC 124. It is observed that the microencapsulated herbal extracts possess a very good resistance for microbes even after 15 washes.

Keywords: Antimicrobial textiles, Cotton, Mexican daisy, Microencapsulation, Neem, Wash durability

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1 Introduction

Health and hygiene are the primary requirements for human beings to live comfortably and work with maximum efficiency. To protect the mankind from pathogens and to avoid cross infection, a special finish like antimicrobial finish has become necessary. As consumers have become more aware of hygiene and potentially harmful effects of microorganisms, the demand for antimicrobial finished clothing is increasing.^{1,2} In selecting the active substances for hygienic finishing, it must be ensured that these substances are not only permanently effective, but also compatible with the skin. Microbial infestation is problematical with clothing worn especially next to the skin.

Antibacterial fibres and various antibacterial chemicals available in international market are mostly from synthetic base and are not environment friendly. Consumer preference has changed and higher demands are placed on the functional fabrics. These new requirements necessitate a production process that is environment friendly. There are many natural

plant products, which show antibacterial properties. Extracts from roots, stem, leaves, flowers, fruits and seeds of diverse species of plants exhibit antibacterial properties. These antibacterial extracts can be used as textile finishing agents in the crude form or as microcapsules to enhance the durability and controlled release of the extracts. Microencapsulation is a rapidly expanding technology and finds greater applicability in textiles in recent years. Uniqueness of microencapsulation is the smallness of coated particles and it provides a means of packaging, separating and storing materials on a microscopic scale for later release under controlled conditions.³⁻⁵

In the present work, herbal plant extracts of neem and Mexican daisy are directly applied onto cotton fabric as antimicrobial finishing agents. The microencapsulation of neem and Mexican daisy is also done and applied onto textiles by pad-dry-cure and by printing to achieve higher durability and controlled release of the active agents. Microcapsules are produced using herbal extracts as core and acacia as wall material.⁶ Comparison between directly applied and microencapsulated herbal extract is done in terms of antimicrobial efficacy and durability.

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2 Materials and Methods

2.1 Materials

Scoured and bleached 100% cotton bed linen fabric (136 ends/inch, 60 picks/inch, and satin weave) was used for the application of antimicrobial finish. The leaves and oil of neem (*Azadirachta indica*) and leaves of Mexican daisy (*Tridax procumbens*) were used for the antimicrobial finish. These plants were collected in and around Coimbatore, Tamil Nadu, India.

2.2 Methods

2.2.1 Extraction Process

The collected herbs were shadow dried within a temperature range of 37-40°C. The moisture content of the herb collected was reduced to less than 14% with proper drying since most of the herbs have moisture content of 60-80% and cannot be stored without drying. Proper drying has to be carried out otherwise important compounds may get contaminated. After drying, the grinding was carried out to break down the leaves of the plant into very small units ranging from coarse fragments to fine powder. Extraction refers to separating the desired material by physical or chemical means with the aid of a solvent. Antimicrobial active substances were extracted from the plant by methanolic extraction method. The powdered plant material was extracted with methanol by adding 20g of herbal powder in 100ml of methanol for 24 h to separate the alkaloids.

2.2.2 Direct Application Method

The fabric samples were treated with herbal extracts using citric acid as cross-linking agent. Methanolic extracts of neem and Mexican daisy were applied onto the fabric by pad-dry-cure method with material- to-liquor ratio of 1:20 at 50°C using 8% citric acid concentration. After padding for 30min, the samples were taken and dried at 100-120°C for 5 min and cured at 180°C for 3min.

2.2.3 Microencapsulation Method

Microencapsulation was done using neem extract, Mexican daisy extracts and neem oil as core material and gum acacia as wall material. Ten gram of wall material was allowed to swell for half an hour by mixing with 100ml of hot water. To this mixture, 50ml of hot water was added, stirred for 15 min maintaining the temperature between 40°C and 50°C. Ten millilitre of core material was added and stirred at

300-500rpm for further 15 min followed by drop-wise addition of 20% sodium sulphate solution(10ml) for 5-10min. The stirrer speed was reduced and then 5 ml of 17% formaldehyde was added. The stirrer was stopped and the mixture was freeze-dried. The cotton bed linen fabric was immersed in the microcapsule solution using pneumatic padding mangle, squeezed and then dried at 80-85°C in an oven. The microcapsules were also applied onto the fabric by printing using a binder.

2.2.4 Test Method

Microcapsules were examined under the $\times 50$, $\times 100$, $\times 200$ and $\times 400$ magnifications using light microscopy with image processing technique to analyze the morphology of capsules. The morphology of microcapsule-treated fabric was analyzed using high-resolution scanning electron microscope JEOL M JSM-6360 with suitable accelerating voltage and magnifications. The scanning electron microscopy was used for confirming the binding of microcapsules and alignment onto the fabric sample.

Swatches of treated and untreated materials were qualitatively assessed by Agar diffusion method (AATCC 100). Those showing activity were evaluated quantitatively. Treated and control samples were inoculated with test organisms. After incubation, the bacteria were eluted from the swatches by shaking in known amounts of neutralizing solution. The number of bacteria present in this liquid was determined and the percentage reduction by the treated specimen was calculated. 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 10^0 dilution was reported as "less than 100". The percentage reduction (R) of bacteria by the specimen treatments was calculated using the following formula:

$$R = 100 (B-A)/B$$

where, A is the number of bacteria recovered from the inoculated treated test specimen swatches in the jar incubated over the desired contact period; and B , the number of bacteria recovered from the inoculated treated test specimen swatches in the jar immediately after inoculation (at '0' contact time).

3 Results and Discussion

3.1 Evaluation of Microcapsules

The most widely used procedures to visualize microparticles are conventional light microscope and

scanning electron microscopy. Both the techniques were used to determine the shape and outer structure of microcapsules. Figure 1 shows the microcapsules of neem oil, neem extract and Mexican daisy extracts as core and gum acacia as wall material. It is clear from the photographs that microcapsules produced are of small spherical shape with a fairly uniform size distribution

3.2 Evaluation of Microcapsules-treated Fabric

The presence, binding and availability of microcapsules on the fabric were analyzed using scanning electron microscopy. The SEM photographs are shown in Fig. 2 at different magnification levels. It is clear that the microcapsules are present in interstices of the fibre assembly of fabric.

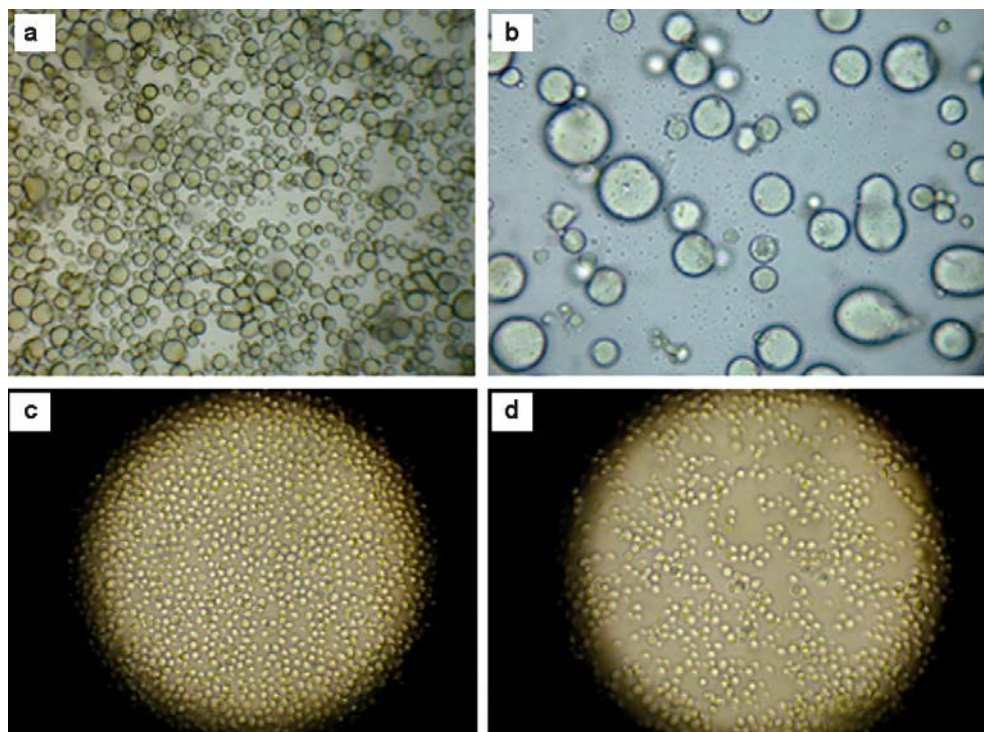


Fig. 1 — Microcapsules of neem oil (a & b), neem extract (c) and Mexican daisy extract (d) as core and gum acacia as wall material [(a) $\times 100$, (b) $\times 200$, and (c) & (d) $\times 1000$]

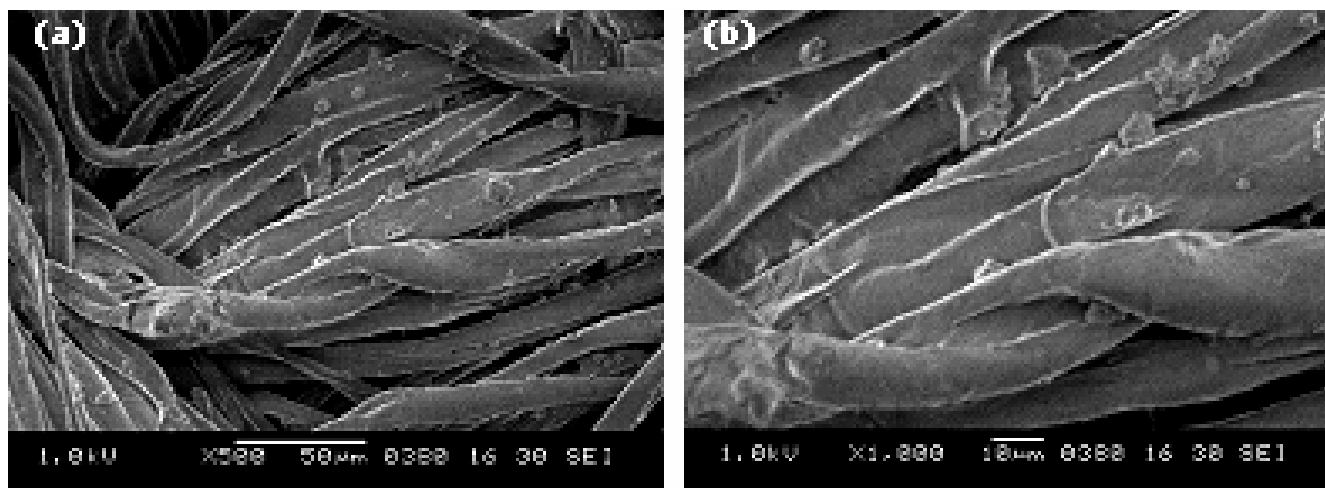


Fig. 2 — SEM photographs of microcapsules-treated fabric [(a) $\times 500$ and (b) $\times 1000$]

Table 1 — Bacterial reduction of treated fabric by quantitative method

Sample	Bacterial reduction, %	
	<i>Staphylococcus aureus</i>	<i>Escherichia coli</i>
Neem extract	100	78.44
Microencapsulated neem extract	93.45	55.21
Mexican daisy	98.75	69.25
Microencapsulated Mexican daisy	92.15	53.85

Table 2 — Wash durability of treated samples

Sample	Bacterial reduction, %			
	No wash	5 washes	10 washes	15 washes
Neem extract (without cross-linking)	100	65	45	0
Neem extract (with cross-linking)	100	82	55	41
Microencapsulated neem oil	78	75	70	59
Microencapsulated neem extract	93	87	81	78
Mexican daisy (without cross-linking)	99	72	35	0
Mexican daisy (with cross-linking)	98	85	45	37
Microencapsulated Mexican daisy extract	92	87	78	67

3.3 Antimicrobial Efficacy of Microencapsulated Herbal Extracts

Table 1 shows the antimicrobial efficacy in terms of bacterial reduction percentage for directly applied and herbal microcapsules applied fabric samples. It is clear that the herbal extracts both directly applied and microencapsulated possess better activity against *Staphylococcus aureus* than against *Escherichia coli*.

3.4 Wash Durability of Neem and Mexican daisy Treated Samples

Table 2 shows the wash durability studies carried out on treated fabrics. It is found that directly applied herbal extract samples do not show much activity after 10 washes. This is because that the extracts were coated only on the surface without any firm bonding

and gets removed by washing. In the case of both extracts applied with citric acid, the samples possess less activity after 10 washes as the active substances are cross-linked to the cotton fabric. Microencapsulated samples show higher activity even after 15 washes.

4 Conclusions

Microencapsulation of herbal extracts such as neem and Mexican daisy has been done successfully by simple coacervation technique using herbal extracts as core material and gum acacia as wall material followed by its application onto fabric using pad-dry-cure method. It is found that they exhibit potential for antimicrobial activity against *Staphylococcus aureus* and *Escherichia coli* in clearly measurable terms. The light microscopy with image processing attachment and SEM studies reveal the presence and alignment of microcapsules on the fabric. Durability test comparing microencapsulated and directly applied herbal extracts methods reveals that the microencapsulated samples retain their activity for more than 15 washes.

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