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Application of ecofriendly antimicrobial finish on cotton and khadi fabrics using periwinkle leaves

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An attempt has been made to develop microcapsules for the antimicrobial finishing of cotton and khadi fabrics using periwinkle leaves. Extract of leaves has been prepared by using methanol solvent and applied on fabric directly as well as through the micro-capsulation method. The treated and untreated samples are then subjected to various tests, such as SEM analysis, FTIR analysis, antimicrobial efficacy and wash durability. The durability of antimicrobial properties to washing is found to be better for samples treated with the microencapsulation method than the direct method.

Keywords: Antimicrobial finish, Cotton, Khadi fabric, Microencapsulation, Periwinkle leaves, Wash durability

1 Introduction

In textile manufacturing, finishing plays a vital role in improving the aesthetic, performance and functionality of the product or material. Finishing processes are carried out to improve the functional properties of the fabric and its serviceability as well. Natural fibres especially cellulosic fibres are very susceptible to microbes that deteriorate textile material because cellulose provides a favourable environment for microbial growth. The bacteria and fungi present on the surface of textile material use natural textile material as their food and grow rapidly, which results in damage to textile material as well as the development of bad odour and stains. Antimicrobial garments are now gaining the attention of consumers. Further, they are an excellent choice in the medicinal and healthcare sectors, as they prevent and reduce infection by various pathogenic microbes and bacteria. There are various types of chemicals that are used as antimicrobial agents, such as quaternary ammonium compounds, triclosan, and metal salts. Some natural antimicrobial agents are also found in various plants, such as neem leaves, tulsi plants, lemon grass, turmeric, clove and periwinkle.

Periwinkle (*Catharanthus roseus*) is an evergreen plant that belongs to the *Apocynaceae* family. It has some other common names, such as Madagascar periwinkle, rosy periwinkle, old maid, vinca, etc. The plant has oblong leaves ½ to 2 inches long, simple and glossy with short petioles.

Catharanthus roseus possesses antibacterial, antimicrobial, antifungal, antidiabetic, anticancer and antiviral properties[1].The extract has demonstrated significant anticancer activity against numerous cell types. The leaf extract contains many indole alkaloids, and some phenolic compounds, which are known for their antimicrobial properties[2]. The extract of periwinkle leaves which have medicinal value can be used on textiles through various application methods during processing.

Microencapsulation may be defined as a micro packaging technique, wherein an active core material is encapsulated in a polymer shell of limited permeability. The objective of microencapsulation is either to protect the active core material from the external environment till required or to affect the controlled release of the active core to achieve desired delay until the right stimulus is encountered. Microencapsulation is a physiochemical technique that provides textiles with resistance to microorganisms and insects. In this technique, a substrate reservoir contains an antibacterial activity sandwiched between two layers of protective polymers, so that the active agent migrates to the outer layer as needed[3].

Antibacterial fabrics and clothes are important alternatives to avoid cross-infection by pathogenic microorganisms. Antimicrobial finish is considered an important parameter for functional textiles which find

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a variety of applications, such as health and hygiene products especially, the garments worn close to the skin and several medical applications, such as infection control.

The present study has been undertaken with the objective to apply the antimicrobial finish on cotton and khadi fabrics, and to evaluate the antimicrobial efficacy of treated fabric against Gram-positive (*Staphylococcus aureus*) and Gram-negative (*Klebsiella pneumonia*) microorganisms. Extract of Periwinkle leaves has been used as an antimicrobial agent using two application methods, viz. direct and microencapsulation. Treated fabrics are subjected to various tests for assessing the percentage of quantitative bacterial reduction and qualitative assessment of antimicrobial activity.

2Materials and Methods

2.1 Materials

Cotton (EPI 104 and PPI 77) and khadi fabrics (EPI 42 and PPI 37) were selected for the application of an antimicrobial finish using an extract of periwinkle leaves. Periwinkle (*Catharanthus roseus*) leaves extract was used as core material for the antimicrobial finish on selected cotton and khadi fabrics. Leaves were collected from the campus of SHUATS, Prayagraj, Uttar Pradesh, India.

Various chemicals, such as chitosan, silicon oil, citric acid, amylase, hydrogen peroxide (H_2O_2), NaOH, etc. ofLR grade were used for specific purposes. Chitosan was used as wall material for microcapsules, citric acid as a cross-linking agent and silicon oil for the preparation of emulsion. Amylase served as a desizing agent, H_2O_2 as a bleaching agent and NaOH as a scouring agent.

2.2 Methodology

2.2.1 Pre-treatment of Fabrics

Before applying an antimicrobial finish on cotton and khadi fabrics, the entire length of fabric was desized, scoured and bleached using standardized recipes, as given hereunder.

The recipe given by Parmar[4] was used for the desizing of cotton and khadi fabrics. Ten grams of amylase enzyme solution per 1000 mL of water was prepared maintaining the material: liquor ratio 1:50. The solution was heated to 50-60°C. Cotton and khadi fabrics were dipped into the solution separately and stirred gently till the fabric turned slightly yellowish in colour. Then, fabrics were kneaded and squeezed and finally rinsed under tap water and dried in sunlight[3].

Scouring and bleaching were carried out simultaneously by using the standard recipe given by Parmar[4].A detergent solution containing 3g of NaOH, 5g of Na_2CO_3, 1g of wetting agent (soap) and 3g of H_2O_2 were added to 1000 mL of water. The solution was prepared maintaining the material: liquor ratio 1:50. It was heated to 100°C, and then cotton and khadi fabrics were dipped into this solution separately and stirred gently for about 60 min. After pretreatment, the fabrics were thoroughly washed with hot water and finally rinsed with cold water. Then samples were dried in sunlight.

2.2.2 Application of Antimicrobial Finish

2.2.2.1 Extraction of Plant Material

Periwinkle leaves were plucked and washed with water to remove dust particles and were dried under shade at room temperature (25°C). After that, dried leaves were crushed into small pieces and stored in airtight containers for extraction. Some glass wool was added into the Soxhlet tube for filtration purpose; 20g of dried and crushed leaves were taken and added to the Soxhlet tube which has round bottom glass fitted at the end. Methanol (300 mL) was added as a solvent and fixed in the Soxhlet extractor for 3 h at 60°C temperature. After 3 h, the extract was filtered into a beaker and incubated at 40°C till the required consistency is achieved. The extract was finally stored in the refrigerator at 4°C for the formation of microcapsules to be used as core material. Extraction was carried out in the Soxhlet apparatus.

2.2.2.2 Application on Fabric

The extract has been applied to the fabrics using the following two methods, namely direct application and microencapsulation.

(i) Direct Application

The above methanol extract of periwinkle leaves was directly applied to the cotton fabric by pad–dry–cure method. The methanol extract (500 mL) and citric acid (6%) as a cross-linking agent were taken and stirred completely. Samples were immersed in the stirred extract of periwinkle leaves for 30 min. After that, samples were taken out and padded on padding mangle at a pressure of 2.5 psi with 2 dips and nips to give a wet pick-up of 85%. Drying and curing were carried out in a hot air oven which was used for drying, condensation and fixation. The samples were dried and cured at 102°C for 3 min.

(ii) Microencapsulation

To prepare the microcapsules, 2.5 g chitosan (as a wall material in microencapsulation) was dissolved in 100 mL distilled water with 1% acetic acid by mechanical stirring; then kept overnight till a clear solution was obtained. Silicon oil (0.5 mL) and 2 g of core material (extract of periwinkle leaves) were slowly added with continuous stirring for an emulsion. Stirring was continued for another 15 min. Coacervation of chitosan was done by the addition of sodium hydroxide (1 g in 100 mL). Stirring was stopped and the mixture was allowed to rest for a period of 30 min at 0-5°C in the refrigerator to develop microcapsules. After that, the solution was kept at 5-10°C for 24 h, filtered, and then washed thrice with distilled water.

For the application of microcapsules, a solution of filtered microcapsules and distilled water was prepared, and samples were immersed in microcapsule solution for 30 min and then padded through padding mangle at a pressure of 2.5 psi. Treated samples were dried at 80°C for 5 min in a hot air oven to cure the samples. The cured samples were evaluated for antimicrobial efficacy against Gram-positive (*Staphylococcus aureus*) and Gram-negative (*Klebsiella pneumonia*) microorganisms.

2.2.3 Functional Analysis of Finish

2.2.3.1 Quantitative Analysis

Assessment of quantitative bacterial reduction was carried out by AATCC test method 100:2014 for both treated and control samples. Those showing activity were evaluated quantitatively. Treated and control samples were inoculated with test organisms (*Staphylococcus aureus* and *Klebsiella pneumonia*). 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°dilution were reported as "less than 100". The percentage reduction (R) of bacteria by the specimen treatments was calculated using the following formula:

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

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, is the number of bacteria recovered from the inoculated treated test specimen swatches in the jar immediately after inoculation (at '0' contact time).

2.2.3.2 Qualitative Method

A qualitative assessment of antimicrobial activity was carried out under AATCC 147-2004 test method. Sterilized nutrient agar was dispensed by pouring 15±2 mL into each standard (15×100 mm) flat-bottomed petri dish. Agar was allowed to get firm before inoculating. Inoculum was prepared by transferring 1.0±0.1 mL of a 24 h broth culture into 9.0±0.1 mL of sterile distilled water contained in a small flask. It was mixed well using appropriate agitation. Using a 4 mm inoculating loop, one loopful of the diluted inoculum was loaded and transferred to the surface of the sterile agar plate by making five streaks approximately 60 mm in length, spaced 10 mm apart covering the central area of the standard petri dish without refilling the loop. It was ensured that no surface breakage should occur while making the streaks.

2.2.4 FTIR Analysis

Fourier transform infrared (FTIR) spectra of the extract of periwinkle on cotton and khadi fabrics were recorded using Perkin Elmer FTIR Spectrometer (SPECTRUM TWO). The wave number range was kept at 450-4000 cm^{-1} and the resolution was 1 cm^{-1}.

2.2.5 SEM Analysis

Scanning electron microscopy (SEM) was used to analyse the presence, binding and availability of microcapsules on the fabric surface. All the treated, untreated and washed samples were observed under instrument 'Carl Zeiss EVO 18' at a magnification of × 50 to ×2500,000 and acceleration voltage of 20 kV. Before the assessment, all the samples were sputtered under a vacuum with gold.

2.2.6 Wash Durability

After the functional analysis of treated samples, wash fastness was performed on treated samples to evaluate the fastness against treated samples up to 10 cycles of wash using AATCC 100 test method.

3 Results and Discussion

The results obtained from the study as well as relevant discussion are summarized hereunder.

3.1 Quantitative Bacterial Reduction

Assessment of quantitative bacterial reduction has been carried out and the results are shown in Table 1 and Fig. 1.

Table 1 — Assessment of bacterial reduction

Sample	Bacteria	No. of bacteria per sample (CFU)		Bacteria reduction, %
		0 h (B)	24 h (A)	
		Untreated (control)		
Cotton	*S. aureus*	1.35×10^5	1.44×10^7	NS*
	K. pneumonia	1.41×10^5	1.27×10^7	NS*
Khadi	*S. aureus*	1.38×10^5	1.55×10^7	NS*
	K. pneumonia	1.47×10^5	1.26×10^7	NS*
		Treated with periwinkle extract		
Cotton				
Direct	*S. aureus*	1.17×10^5	0.036×10^5	96.92
	K. pneumonia	1.38×10^5	0.023×10^5	98.33
Microcapsulation	*S. aureus*	1.71×10^5	0.136×10^5	92.04
	K. pneumonia	1.85×10^5	0.121×10^5	93.45
Khadi				
Direct	*S. aureus*	1.43×10^5	0.036×10^5	97.48
	K. pneumonia	1.50×10^5	0.021×10^5	98.6
Microcapsulation	*S. aureus*	1.30×10^5	0.112×10^5	91.38
	K. pneumonia	1.80×10^5	0.11×10^5	93.88

*NS–Non-significant.

The result clearly reveals that all the treated fabric samples (cotton and khadi both) show good antimicrobial properties against Gram-positive (*Staphylococcus aureus*) and Gram-negative (*Klebsiella pneumonia*) microorganisms. This is because the treated fabrics do not allow the growth of microorganisms.

In the case of cotton samples, a higher percentage of antimicrobial efficacy is obtained against *Klebsiella pneumonia* (98.33% direct and 93.45% microencapsulation) in both application methods. On the other hand, the percentage of antimicrobial efficacies against *Staphylococcus aureus* is a bit less as compared to *Klebsiella pneumonia* reduction efficacy.

Similar results are obtained on khadi samples. Both direct and microencapsulation methods show higher efficacy of reduction against *Klebsiella pneumonia* as compared tothat against *Staphylococcus aureus*. However, no bacterial reduction is observed in the case of untreated samples of cotton and khadi.

3.2 Qualitative Method for Assessing Antimicrobial Activity

A qualitative assessment of antimicrobial activity has been carried out and the results are reported in Table 2.

It is observed that both untreated samples (cotton and khadi) do not show antimicrobial properties and hence microbial growth has been observed on these samples. However, treated samples show the presence of antimicrobial properties, thereby no microbial growth on these samples is observed.

Table 2 — Assessment of the presence or absence of bacteria (zone of inhibition)
[Zone of inhibition–No zone]

Sample	Bacteria	Microbial growth	Antimicrobial activity
	Untreated (control)		
Cotton	*S. aureus*	Present	Absent
	K. pneumonia	Present	Absent
Khadi	*S. aureus*	Present	Absent
	K. pneumonia	Present	Absent
	Treated with periwinkle extract		
Cotton			
Direct	*S. aureus*	Absent	Present
	K. pneumonia	Absent	
Microcapsulation	*S. aureus*	Absent	Present
	K. pneumonia	Absent	
Khadi			
Direct	*S. aureus*	Absent	Present
	K. pneumonia	Absent	
Microcapsulation	*S. aureus*	Absent	Present
	K. pneumonia	Absent	

It is also found that qualitative test is good for testing the main agent or treated fabrics, provided the antibacterial agent used can leach out. It is observed from Table 2 that all the treated fabrics show antibacterial activity against Gram-positive (*Staphylococcus aureus*) and Gram-negative (*Klebsiella pneumonia*) microorganisms. On the other hand, untreated fabrics show the growth of microorganisms and no antibacterial activity.

In the case of both treated fabrics (cotton and khadi), there is no zone of inhibition for both organisms. The zone of inhibition indicates that periwinkle extract not only prevents the growth of microbes on the fabrics but also helps in leaching out the bacteria.

3.3 SEM Studies

Both treated and untreated cotton and khadi samples are analysed using the SEM test and the results are shown in Fig. 2. Figures 2 (a) and (c) show that both untreated samples (cotton and khadi) have a smooth and clean surface, because of the pre-treatment process all impurities including starch get washed off. However, treated samples [Figs 2 (b) & (d)] show rough and uneven surfaces due to the deposition of microcapsules made by chitosan as wall material.

3.4 FTIR Studies

The overlapping spectrum between control samples of cotton and khadi, as well as samples treated directly and with the microencapsulation method, are shown in Fig. 3. Figure 3 (a) reveals the presence of a typical group of *Catharanthus roseus* on the surface of treated samples. The peaks at 3336.6 cm^{-1} observed in the direct application and at 3338 cm^{-1} in microencapsulation application indicate the occurrence of stretch-free vibration type alcohol with O-H; 2906.5 cm^{-1} and 1749.4 cm^{-1} peaks represent the organic aromatic group

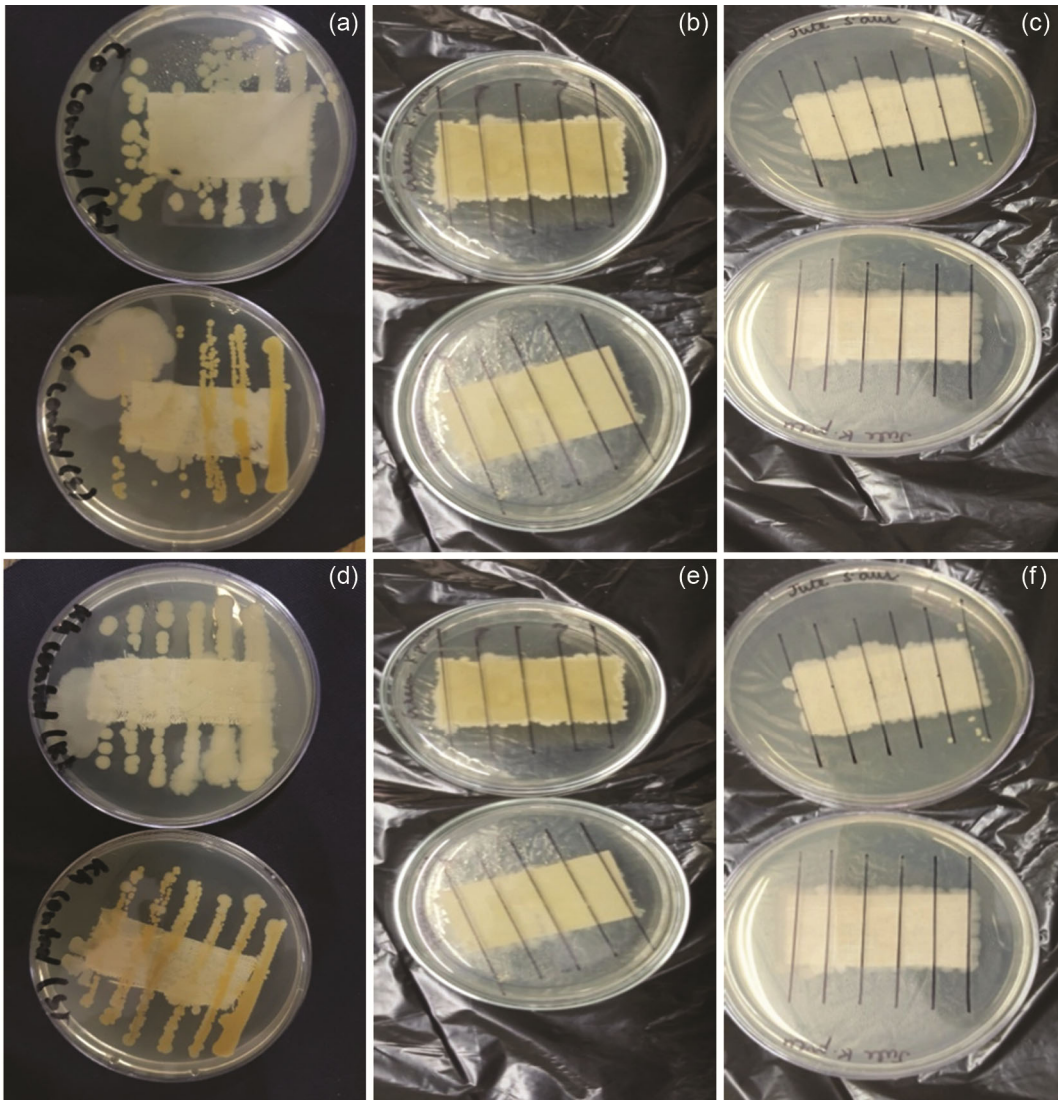

Fig. 1 — Antimicrobial activity of treated and untreated cotton and khadi fabrics against *Staphylococcus aureus* and *Klebsiella pneumonia* [(a) untreated khadi sample, (b) khadi sample treated with direct application, (c) khadi sample treated with microencapsulation method, (d) untreated cotton samples, (e) cotton samples treated with direct application method and (f) cotton Sample treated with microencapsulation method]

and carbonyl acid with C=C and O-H correspondingly. The presence of C-H stretch organic amine is confirmed due to the presence of a 1035 cm^{-1} band. Similarly, Fig. 3 (b) reveals the presence of a typical group of *Catharanthus roseus* on the surface of treated samples. Peaks at 3318.8 cm^{-1} indicate the occurrence of stretch-free vibration type alcohol with O-H; peaks at 2915.8 cm^{-1} and 1718.4 cm^{-1} represent organic aromatic group and carbonyl acid with C=C and O-H correspondingly. The presence of functional groups, like organic (=C-H bending), carbonyl acid (C-O stretch), organic (alkane; C-H stretch), and carbonyl (amide N-H stretch) are confirmed with the occurrence of infrared band at 996.9, 1319.3, 1749.4, 2908.5, 3336.6 cm^{-1}, while peaks at 874, 1238.7, 1718.4, 2915.8, 3318.8 cm^{-1} indicate the presence of phenolic group on treated samples, thus indicating the reason for antimicrobial efficacy.

3.5 Wash Durability of a Fabric Having Antimicrobial Property

Wash durability test has been performed on both cotton and khadi fabrics treated with periwinkle extract and the results are reported in Table 3.

It is clear from Table 3 that both the samples treated with direct application show very good antimicrobial properties against both the microorganisms,but after a few washes, the bacterial reduction is dropped, which means the fabric loses its antimicrobial property on subsequent washing. However, samples treated with themicroencapsulation method show a slight drop in antimicrobial properties, indicating that on each successive washing, the bacterial reduction is sustained.

In the case of samples treated with the microencapsulation method for both cotton and khadi fabrics, a good microbial reduction is observed on

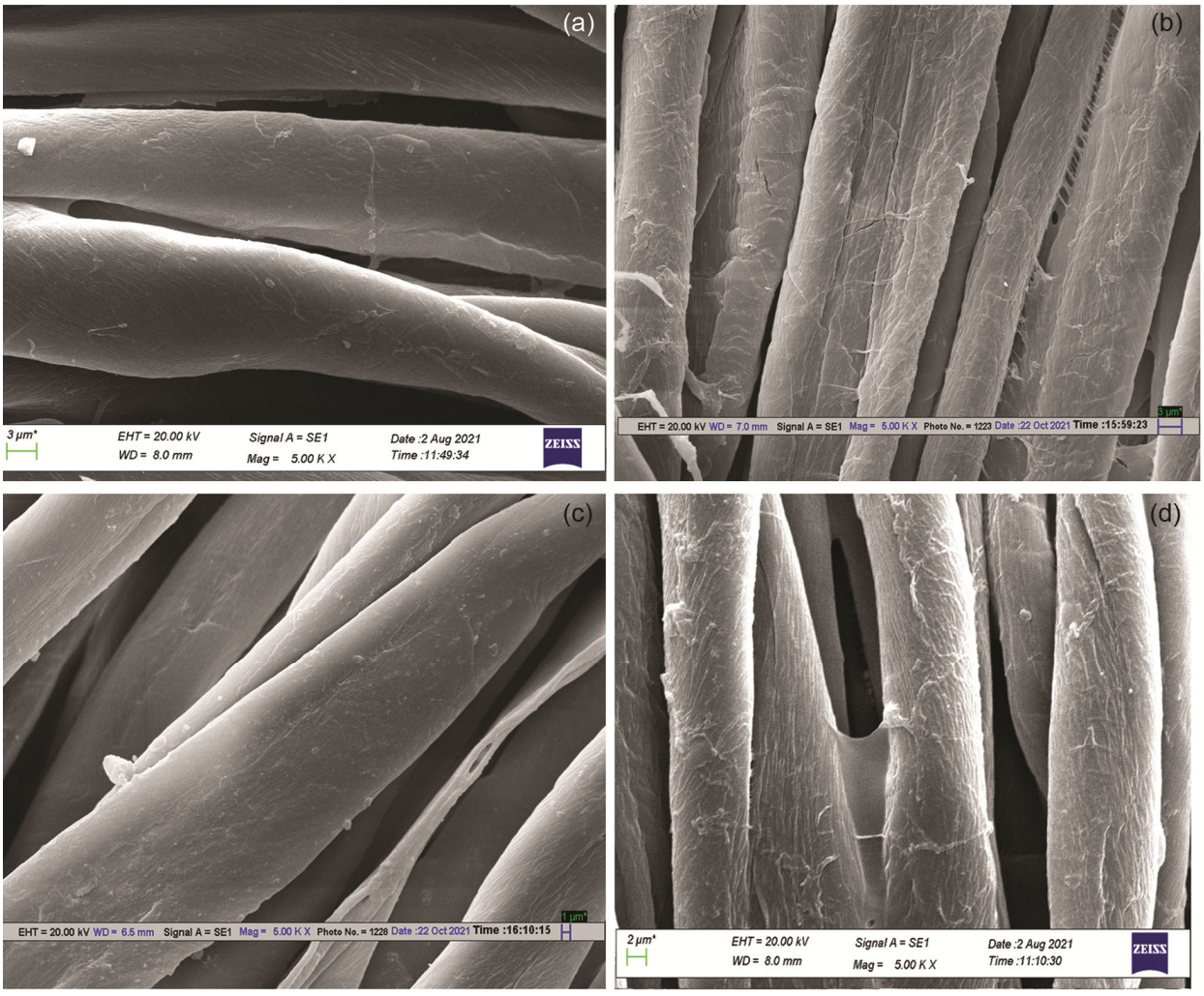

Fig. 2 — SEM photographs of cotton and khadi fabrics [(a) Untreated cotton, (b) microencapsulated cotton, (c) untreated khadi and (d) microencapsulated khadi]

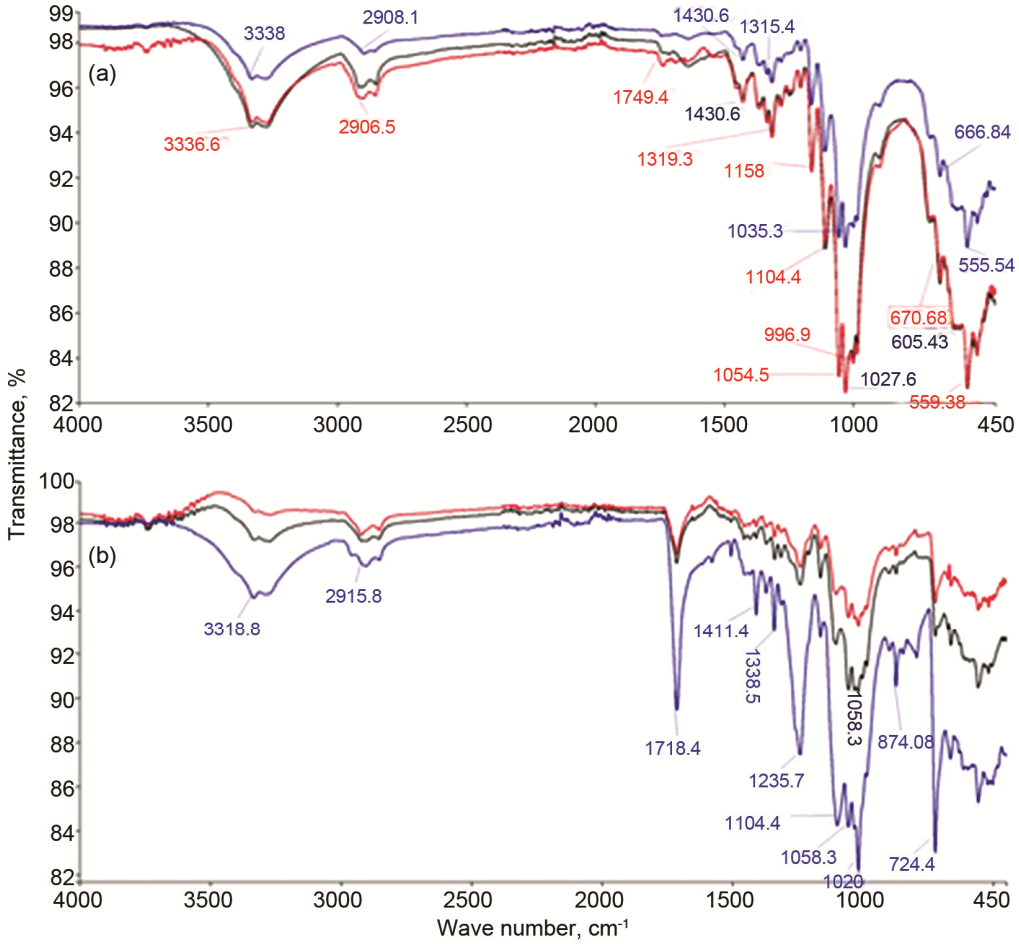

Fig. 3 — Overlapping of FTIR spectra of control, periwinkle-treated (Direct) and periwinkle-treated (microencapsulation) samples [(a) cotton and (b) khadi]

Table 3 — Wash durability of treated samples

No. of washes	% of bacterial reduction							
	Direct method				Microencapsulation method			
	Cotton		Khadi		Cotton		Khadi	
	Sa	*Kp*	*Sa*	*Kp*	*Sa*	*Kp*	*Sa*	*Kp*
0	96.92	98.33	97.48	98.6	92.04	93.45	91.38	93.88
1	31.0	23.3	29.2	22	90.6	86.4	90.7	86
3	26.4	20.1	21.3	18	89.2	85.2	84.5	81.9
5	22	17.3	15	12	88.1	84.0	79.4	76
7	12.8	8.6	8	6	79	76.9	63.4	70.2
10	6.3	4	3.3	2.5	64.3	52.8	57.7	53.9

Sa- Staphylococcus aureus and *Kp- Klebsiella pneumonia*

zero wash as well as the capacity of bacterial reduction is sustained up to 10 wash cycles against both the microorganisms as compared to the direct application method.

Wash durability of microencapsulated samples is found better as compared to samples treated with the direct method. The finish applied on cotton fabric is found more effective than khadi fabric.

4 Conclusion

Microencapsulation of herbal extract (periwinkle leaves) has been done successfully by phase

coacervation method using herbal extract as core material and chitosan as wall material, followed by its application onto fabric using pad-dry-cure and direct method. It is found that the treated cotton sample shows a higher percentage of antimicrobial efficacy in both the application method against *K. pneumonia* (98.33 and 93.45%). On the other hand, the percentage of antimicrobial efficacy against *S. aureus* is found less as compared to *K. pneumonia* reduction efficacy. A wash durability test comparing microencapsulated and direct applied herbal extract for both cotton and khadi fabric reveals that microencapsulated samples retain their activity up to 10 wash cycles.

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