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Research Paper

Antibacterial finishing of cotton fabric with clove oil microcapsules

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■ **ABSTRACT :** The present study was conducted to develop a microencapsulated antibacterial finish using clove essential oil for cotton fabric. Microcapsules of clove essential oil (*Syzygium fragranceticum)* were developed with phase separation - complex coacervation technique using five concentrations of essential oil for application on cotton fabric. The finished cotton fabric was tested for SEM analysis and anti-bacterial finish using standard test methods. Findings of SEM analysis revealed that small spherical shape microcapsules in the treated samples were located at interstices between the fibres and on the fibre surface. The zone of inhibition was found maximum at concentration 5 of MCCL treated cotton fabric before wash with 12.5 mm ± 1.5 and 12.33 mm ± 0.57 (Mean and SD) against *S. aureus* and *E. coli*. MCCL oil treated cotton fabric showed the good wash durability against *S. aureus* as compared to *E.coli*. The antimicrobial property decreased with increasing number of wash cycles.

■ **KEY WORDS:** Antimicrobial, Finishing, Cotton, Clove, Oil, Microcapsule

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Nature has gifted the mankind with variety of plants and herbs. Most of the plants have medicinal properties and they are used as natural curing substance in treating many illnesses even today in ayurvedic and naturopathy. The plant extracts possess many functional properties, they can be used as natural source of dye for textile colouration and also as finishing agent to impart herbal finishes on textile substrate. Microbial infestation poses danger to both living and non-living matters. Textile materials are good media for growth of microorganisms. Obnoxious smell from the inner garments such as socks, spread of diseases, staining and degradation of textiles are some of the detrimental effects of bad microbes. Though the use of antimicrobials has been known for the decades, it is only in the recent couple of years several attempts have been made on finishing textiles with antimicrobial compounds (https://www.textiletoday. com.bd). Herbal treatment of textiles is growing in popularity across the globe as they have no side effects. Microencapsulation of essential oil is a process in which tiny particles or a coating to give small capsules with many useful properties surrounds droplets. Herbal Textile is finished entirely with herbal extractions, without using any sort of chemicals. These herbs are applied directly to the fabric with the help of natural ingredients, so that the medicinal value of the herbs can

be kept intact. The antimicrobial bioactive agents have become highly important for bio-functionalization for textile materials because they impart safe nontoxic and environment-friendly properties (Sathianarayanan, 2010). Herbal plant extract for antimicrobial finishing in textiles are being used because of the excellent antimicrobial and ecofriendly properties exhibited by them (El-Shafei *et al.*, 2018).

Biotechnology is a frontier area in the field of science and technology having significant commercial applications in healthcare, agriculture, Textile and service sectors. There is a good deal of demand for the fabrics having functional/specialty finishes in general antimicrobial finishes in particular to protect human being against microbes (Kumar, 2014 and Bouchekrit *et al.*, 2016). These Antibacterial extracts can be used as textile finishing agents in solvent form or microcapsules to enhance the durability and controlled release of the extracts (Kathirvel and Ramachandran, 2014). Clove oil has very good antimicrobial properties. In order to increase clove oil eugenol, eugenol acetate utilization, it is important to protect and maintain its stability by microencapsulation process.

■ RESEARCH METHODS

Present research is follow-up study of the researcher on 'Development of clove oil microcapsules by process optimization using coacervation technique'. The best microcapsules of clove oil were developed when the ratio of oil:gum:gelatin was kept 2:4:4, at optimized 50ºC temperature having initial pH 4.0 and final pH 9.0. These developed microcapsules were used as anti-bacteial finish on cotton fabric.

Application of developed microcapsules on cotton fabric:

Developed microcapsules of clove oil was applied on cotton fabric using padding mangle with pad dry cure process. Fabric sample was first immersed in the finishing solution containing microcapsules, M:L ratio, binder and softener. Then the immersed fabric was padded for better absorption of microcapsules. Fabric was then passed between the rollers to squeeze out excess liquid from the fabric, dried in shade and cured at 110°C for 30 to 120 seconds in curing chamber.

Assessment of finished cotton fabric :

The finished cotton fabric samples treated with varied concentrations of MCCL oil (microencapsulated clove oil) were tested for SEM analysis and anti-bacterial finish using standard test methods. The surface of the fabric when photographed showed the presence of developed microcapsules of essential oil fixed in to the yarns of the fabric to a maximum amount as revealed by Krishnaveni and Mani (2012). The SEM photographs of finished samples were taken under different magnifications to ensure the presence of microcapsules in the fabric structure and images were captured.

The fabric samples treated with MCCL oil at selected concentration 5 was subjected to different wash cycles (5, 10, 15, 20 and 25) in launder-o-meter using standard test (AATCC: 43-1995) for wash durability of finish using AATCC standard test method *i.e.* Parallel streak method (AATCC-147). The width of zone of inhibition was calculated by taking three replicates of each concentration of MCCL oil treated samples and their mean and SD was taken out as width of inhibition zone for corresponding concentration.

■ RESEARCH FINDINGS AND DISCUSSION

Findings of the study revealed that best results of microcapsules formation using clove oil were obtained when the ratio of oil:gum:gelatin was kept 2:4:4, at optimized 50ºC temperature having initial pH 4.0 and final pH 9.0. The microcapsules formed with these optimized conditions were medium having good uniformity in size and distribution with sharp and thick walls of capsules.

SEM analysis of MCCL oil treated fabric samples:

Before wash:

The fabric samples treated with MCCL oil at five different concentration using microencapsulation methods were checked for presence of microcapsules as shown in Plate 1 under Scanning Electron Microscope. MCCL oil treated fabric surface was found coated with numerous uniform size microcapsules, located at interstices between the fibre and on the fibre surface.

According to SEM analysis, concentration 1 of MCCL oil treated sample contains very few small and very few large sized microcapsules, concentration 2 was found having few medium sized microcapsules, concentration 3 was found to possess some medium and

few small sized microcapsules and concentration 4 was observed with many medium and very few large sized microcapsules. At concentration 5, too many medium and small sized microcapsules showed their presence on the fibre surface. Sathianarayanan *et al.* (2010) found small spherical shape microcapsules with a fairly uniform size distribution in microencapsulated fabric through SEM analysis.

After wash:

The fabric samples treated with MCCL oil at concentration 5 were subjected to different wash cycles (5, 10, 15, 20 and 25) for observing presence of microcapsules under scanning electron microscope.

SEM analysis of wash durability of finished cotton fabric with concentration 5 of MCCL oil:

Plate 2 shows SEM analysis of wash durability of

Plate 1 : SEM analysis of MCCL oil treated fabric samples at different concentrations before wash

SEM analysis of MCCL oil treated fabric samples before wash

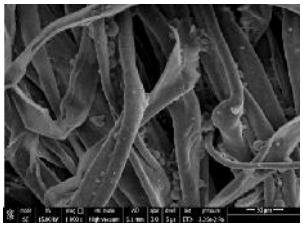
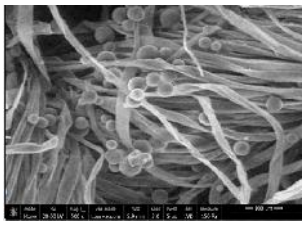
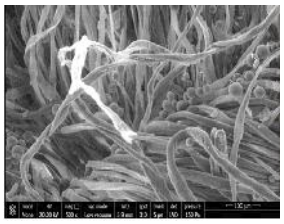
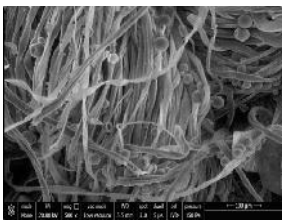
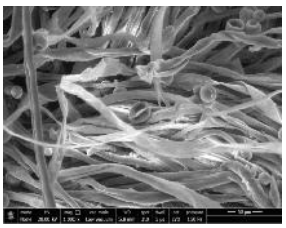
Treated fabric samples at different concentration	SEM Images	Inference
Concentration 1 (10 g/litre)		Presence of very few small and very few large sized microcapsules
Concentration 2 (20 g/litre)		Presence of few medium sized microcapsules
Concentration 3 (30 g/litre)		Presence of some medium and few small sized microcapsules
Concentration 4 (40 g/litre)		Presence of many medium and very few large sized microcapsules
Concentration 5 (50 g/litre)		Presence of too many medium and small sized microcapsules

Plate 2: SEM analysis of wash durability of treated fabric samples with concentration 5 of MCCL oil

SEM analysis of MCCL oil treated fabric samples after wash

Wash cycles (Concentration 5)	SEM Images	Inference
5 Wash cycle		Presence of too much medium and some small sized microcapsules
10 Wash cycle		Presence of some medium and some small sized microcapsules
15 Wash cycle		Presence of few medium and few large sized microcapsules
20 Wash cycle		Presence of few medium and very few large sized microcapsules
25 Wash cycle		Presence of very few medium sized ruptured microcapsules

finishes cotton fabric samples with concentration 5 of MCCL oil. According to SEM analysis, after 5 wash cycles, MCCL treated sample with concentration 5 was observed with too much medium and small sized microcapsules and at 10 wash cycles, sample showed presence of some medium and some small sized microcapsules, after 15 wash cycles, few medium and few large sized microcapsules, after 20 wash cycles, few medium and very few large sized microcapsules and at wash cycle 25, very few medium sized ruptured microcapsules were found present on the fibre surface. The size and quantity of microcapsules was evaluated on visual comparative basis.

Antimicrobial efficiency of MCCL oil treated fabric samples before wash:

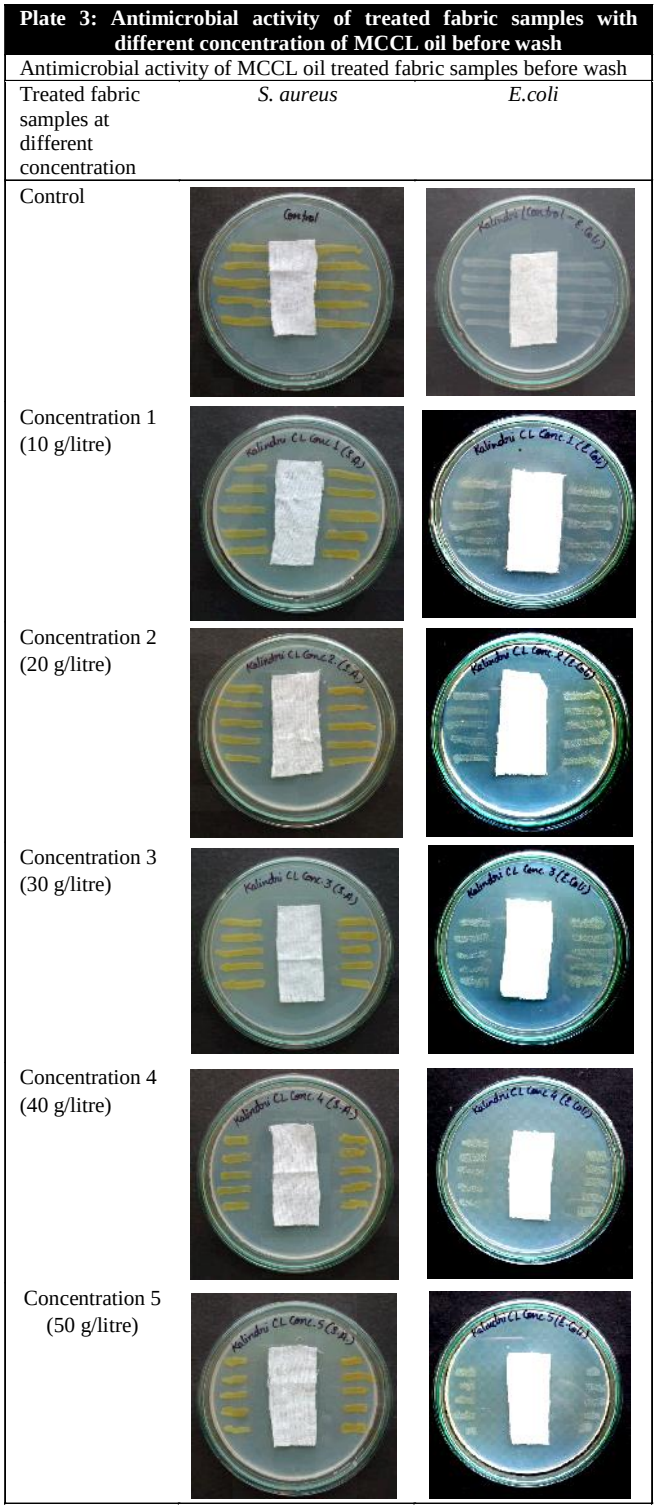
The finished fabric samples were tested for Anti-microbial activity using AATCC standard test method *i.e.* Parallel streak method (AATCC-147). The width of zone of inhibition was calculated for each replicates of each concentration of MCCL oil treated samples and their mean and SD was taken as width of inhibition zone for corresponding concentration. The results are shown in the Table 1 and Plate 3.

Table 1 : Antimicrobial activity of the MCCL oil treated fabric samples against *S. aureus* and *E. coli* (before wash)

Concentration of MCCL oil treated samples	Zone of inhibition (mm) in treated samples	
	S.aureus	*E.coli*
	Mean ± SD	Mean ± SD
Concentration 1 (10g/litre)	3.16 ± 0.76	2.83 ± 0.28
Concentration 2 (20g/litre)	4.83 ± 0.76	4.33 ± 0.76
Concentration 3 (30g/litre)	7.16 ± 0.76	6.33 ± 1.2
Concentration 4 (40g/litre)	10.0 ± 1.32	9.50 ± 0.5
Concentration 5 (50g/litre)	12.5 mm ± 1.5	12.33 mm ± 0.57

Samples treated with concentration 1 showed 3.16 mm ± 0.76 (Mean and SD) zone of inhibition against *S. aureus* and 2.83 mm ± 0.28 (Mean and SD) against *E. coli*. Concentration 2 showed 4.83mm ± 0.76 (Mean and SD) inhibition zone against *S. aureus* and 4.33 mm ± 0.76 (Mean and SD) against *E. coli*. The zone of inhibition at concentration 3 was 7.16 mm ± 0.76 and 6.33mm ± 1.25 (Mean and SD) and at concentration 4, 10.0 ± 1.32 and 9.50 ± 0.5 (Mean SD) against *S. aureus* and *E. coli*, respectively. The zone of inhibition was maximum at concentration 5 with 12.5 mm ± 1.5 and 12.33 mm ± 0.57 (Mean and SD) against *S. aureus* and *E. coli*. Untreated fabric sample showed clear growth of the pathogens under them with no inhibition against *S. aureus* and *E. Coli* as shown in Plate 3.

Plate 3: Antimicrobial activity of treated fabric samples with different concentration of MCCL oil before wash



It was observed that by increasing the microcapsules

Table 2 : Effect of wash cycles on antimicrobial activity of MCCL oil treated fabric samples at conc. 5 against *S.aureus* and *E.coli*

Wash cycles	Zone of inhibition (mm) of treated samples at Concentration 5	
	S.aureus	*E.coli*
	Mean ± SD	Mean ± SD
0 WC (Control)	12.5 ± 1.5	12.33 ± 0.57
1 WC	12.0 ± 1.5	11.67 ± 0.76
5 WC	10.67 ± 1.52	10.33 ± 1.04
10 WC	8.5 ± 1.80	7.67 ± 1.15
15 WC	5.5 ± 0.86	4.83 ± 1.44
20 WC	2.33 ± 0.57	1.5 ± 1.73

concentration, width of inhibition zone increases in the MCCL oil treated samples. This may be due to more microcapsules gets attached to the substrate through bond formation on the surface in higher concentration as compared to low concentration. Test control did not show any antibacterial effect against the test bacteria as inhibition zone was not found.

Assessment of wash durability of MCCL oil treated fabric samples:

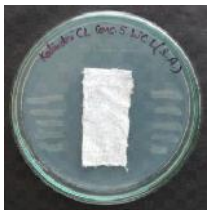
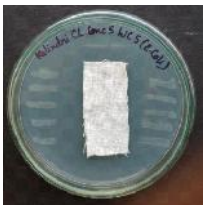
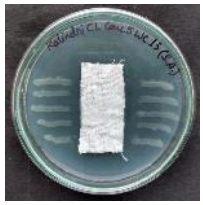
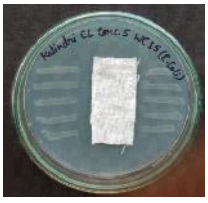
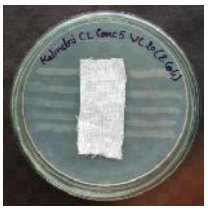
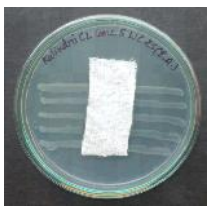
The selected concentration 5 of MCCL oil treated samples was tested for wash durability using AATCC standard test method *i.e.* Parallel streak method (AATCC-147). The width of zone of inhibition was calculated by taking three replicates of each concentration of MCCL oil treated samples and their mean and SD was taken out as width of inhibition zone for corresponding concentration.

It is clear from Table 2 and Plate 4 that fabric treated with MCCL oil with concentration 5 showed inhibition zone 12.5 mm ± 1.5 and 12.33 mm ± 0.57 for *S. aureus* and *E. coli*, respectively at 0 wash cycle. After 1 wash cycle inhibition zone was shown 12.0 mm ± 1.5 and 11.67mm ± 0.76 for *S. aureus* and *E. coli*, respectively. The zone of inhibition of samples of 5 wash cycle showed 10.67mm ± 1.52 and 10.33 mm ± 1.04 (Mean and SD) against *S. aureus* and *E.coli*, respectively, while, 8.5 mm ± 1.80 and 7.67 mm ± 1.15 (mean and SD) against *S. aureus* and *E. coli* at wash cycle 10 sequentially. The zone of inhibition at 15 was cycle, the sample showed 5.5 ± 0.86 and 4.83 ± 1.44 (Mean and SD). At the end of 20 wash cycle the value reaches to 2.33 mm ± 0.57 (Mean and SD) and 1.5 mm ± 1.73 for *S. aurous* and *E. coli*, respectively.

The antimicrobial activity is retained upto 20 washes indicates that the rate of release is quite slow with higher concentration of Clove oil microcapsules. From the table it can be concluded that fabric treated with concentration 5 gives good antimicrobial property till 15 washes. After 15 washes, inhibition zone was shown less under the

Plate 4: Antimicrobial activity of wash durability of treated fabric samples with concentration 5 of MCCL oil

Antimicrobial activity of MCCL oil treated fabric samples at concentration 5 after wash

Wash cycles (Concentration 5)	*S. aureus*	*E.coli*
1 Wash cycle		
5 Wash cycle		
10 Wash cycle		
15 Wash cycle		
20 Wash cycle		
25 Wash cycle		

fabric. Antimicrobial property decreases with increase in number of wash cycles, because MCCL oil destroyed and become inactive by increasing washing cycles. Findings are in confirmation with Vyas *et al.* (2011) who observed that there was no bacterial growth in the finished samples upto 15 washings. On 20 washings there was less bacteria observed in Aloe vera finished cotton and silk samples.

Conclusion:

The antibacterial efficiency was found greater in the MCCL oil treated sample as compared to control sample in case of *S. aureus* and *E-coli*. The developed microcapsules gel of clove oil at concentration 5 showed good anti-bacterial activity upto 15 washes and thus can effectively be applied on cotton fabric to impart herbal anti-bacterial finish.

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