

Synthesis, Characterization and Chemical Attachment of Fragrance Releasing Microcapsules on Cotton

Jinal K. Rohit

M.Tech Scholar, Department of Textile Chemistry, Maharaja Sayajirao University of Baroda, Gujarat, India

Abstract

The review provides an overview of research findings on microencapsulation for functional textile coatings. The Essential Oils of Clove oil, Sandalwood Oil and Cinnamon Oil is widely used as a raw material for fragrances with volatile active compounds at room temperature. It is easily affected by environmental changes and this problem can be solved by microencapsulation using coacervation methods to protect active compounds. The Essential Oils was encapsulated as coating material with 1,3,5-trichlorotriazine as the crosslinking agent. The purpose of this research was to study the effect of the composition of polymers and the mass of Essential oils on encapsulation characteristics. The resulting microcapsules were analysed using, fragrance loaded microcapsules, Anti-Bacterial Activity and digital microscope of SEM encapsulation efficiency. The result showed that microcapsules have an irregular shape with a textured surface.

Keywords: microcapsules, coatings, functional textiles, biodegradability, Essential Oils of Clove oil, Sandalwood Oil and Cinnamon Oil, Cotton Fabric.

1. INTRODUCTION

In recent years, scientists have developed smart fabrics that react to stimuli such as light, temperature or mechanical stress and respond in certain ways, such as by changing colour or conducting an electrical signal. Researchers have also explored different methods to release fragrances from fabrics. The encapsulated material is released in a gradual and controlled manner giving long lasting effects. If these chemicals are grafted to textiles, various special properties can be introduced to it which can be called as special finished products. Insertion of Fragrance to textiles is one such immaculate magnanimous entry into any textile culture. Insertion of Fragrance to the textiles is the process where we enhance the value of the product by adding some incentives to it. Fragrance is both an art and the science. It can also be called “An Emotional Catalyst” [1]. Odours are also called scents, which can refer to both pleasant and unpleasant odours. The terms fragrance, scent, and aroma are used primarily by the food and cosmetic industry to describe a pleasant odour, and are sometimes used to refer to perfumes. In contrast, malodour, stench, reek, and stink are used specifically to describe unpleasant odours [2]. The all your senses, only the sense of smell acts directly with the brain [3]. Sense of smell gives rise to the perception of odours. Smell is mediated by the olfactory nerve [3].

Odour perception is a complex process involving the central nervous system that can evoke psychological, the detection of stimuli by receptors in the nose and physiological responses, the stimuli are processed by the region of the human brain which is responsible for olfaction [2,3]. When you perceive an odour, it is because the molecules of that odour activate specific receptors in the nasal epithelium and then in the olfactory bulb. Although olfactory sensitivity generally declines with age [4]. Hazardous odours are extremely objective and the human body has been wired to be very accurate to prevent you from killing yourself. The scent is immediately assumed as not “natural” and is used effectively to save lives [5]. The perfumes date back more than 4,000 years [6]. Dawn to dusk and from womb to the tomb we are enveloped by odiferous substances and Our emotions and behavior are influenced by smells [4,7]. The Vedas that are more than 3000 years old mentions the use of different herbs, barks, shrubs, and flowers as Yagna offerings to please god almighty. Use of fragrance water for bathing and freshening has been described in detail in the Indian Epics, the Ramayana, and the Mahabharata. Sanskrit texts written during the Mauryan rule describe the method of fragrance preparation and its development [4].

Microencapsulation, a process in which tiny particles or droplets are surrounded by a coating to give small capsules with many useful properties. The material inside the microcapsule is referred to as the core, internal phase or fill, whereas the wall is sometimes called a shell, coating or membrane. Most microcapsules have diameters of few micrometres. The reasons for microencapsulation are countless. In some cases, the core must be separated from its surroundings, as in isolating vitamins from the deteriorating effects of oxygen, retarding evaporation of a volatile core, improving the handling properties of a sticky material, or isolating a reactive core from chemical attack. In other cases the objective is not to separate the core completely but to control the rate at which it leaves the microcapsules as in the controlled release of drugs or pesticides. Manufacturing costs are based on coating material, solvent, equipment and labour. Coatings that can be applied without solvent or water are preferred. Environmental and safety regulations greatly increase the cost of process that use volatile organic solvents. Microcapsule consists of two parts, viz. the core and the wall material. The typical range of capsule content is 70 to 99% nucleus material by weight. This corresponds to a capsule wall thickness of about 0.1-200mm. Different shapes of micro capsules is shown in Figure 1 and 2 [8].

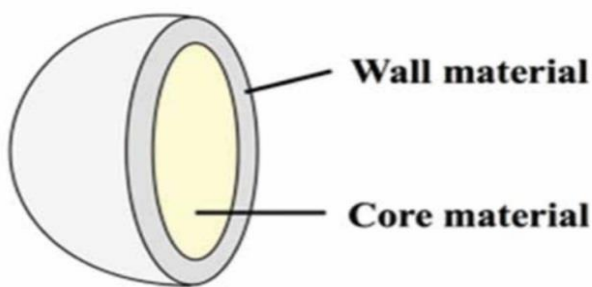


Figure 1: The schematic cross section of microcapsule

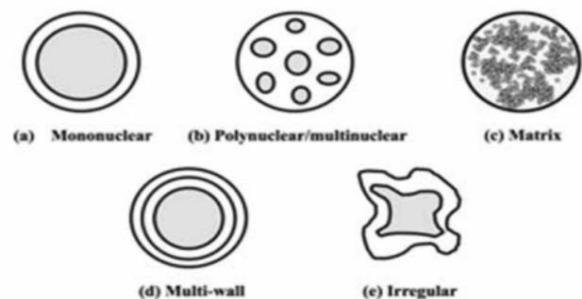


Figure 2: Morphology of Microcapsules

The content of capsules can be made available by mechanical rupture of the capsule wall, by causing its disintegration by electrical or mechanical means or by leaching action carried out in an appropriate liquid environment. Microcapsules range in diameter from 1 to 1000 mm; capsules greater than 1000 mm can be called microcapsules and those smaller than 1 mm are called nanocapsules.

2. MATERIALS & EXPERIMENTAL METHODS

2.1 Material

Desized, scoured and bleached i.e. ready for dyeing 100% cotton fabric and purchased from local market were used for the study. The fabric was purified by scouring at 100 °C for 60 min. using a solution containing Na₂CO₃, to remove dust particles and finishing chemicals, then all the fabric were neutralized, thoroughly washed with water and dried at ambient conditions.

2.2 Specifications of Essential oils used

1. Clove oil:

It contains a chemical called eugenol. The clove tree, known as *Syzygium aromaticum*. Clove oil is produced by distilling the dried flower buds that are collected from the clove tree. Other parts of the tree, such as the stem and leaves, may also be used. Clove oil, which ranges in color from colorless to light yellow. The major component of clove oil is usually considered to be eugenol, with β -caryophyllene and lesser amounts of other components such as benzyl alcohol, but the proportions vary widely [9]. Clove oil has antifungal properties against yeast, filamentous fungi, and pathogenic fungi in human [10]. Phenolic compounds as clove oil constituents have antimicrobial and phytotoxic properties, and can kill insects [11].



Figure 3: Essential Clove oil

2. Sandalwood Oil:

The essential oil of East Indian Sandalwood (*Santalum album* L., Santalaceae) contains more than 90% of sesquiterpenic alcohols, the santalols. The major component, with approximately 50-60%, among these alcohols is the tricyclic-santalol, β -Santalol makes up for about 20-25 % [12]. In aromatherapy, Sandalwood oil is deemed aphrodisiac, antide-pressant, relaxing, and sedative [13]. sandalwood oil has been used as an antiseptic and astringent, and for the treatment of headache, stomachache, and urogenital disorders. Sandalwood oil has also demonstrated repellency against the crop pest, *Tetranychus urticae* (two-spotted spider mite), with santalol suggested as the effective component.



Figure 4: Essential Sandalwood Oil

3. Cinnamon Oil:

The botanical name "Cinnamomum" is derived from the Hebraic and Arabic term amomon, meaning fragrant spice plant. The aroma of essential oil has a sweet, spicy, slightly woody, and clove-like aroma. The major chemical constituents are cinnamaldehyde (65-80%) and eugenol (5-10%). The cinnamyl group such as cinnamic acid and cinnamyl acetate, compounds containing endocyclic double bond as α -thujene, α -terpineol, α -cubebene, unconjugated exocyclic double bond eugenol, β -caryophyllene, terpinolene and hydroxyl-substituted aliphatic compounds. Biological activities, for instance, antioxidant, antimicrobial, antifungal, antidiabetic among others [14].



Figure 5: Essential Cinnamon Oil

a. Synthesis of Microcapsule

Few drops of essential oils were dispersed in ethyl cellulose dissolved in ethyl acetate and the dispersion was rotated in an aqueous bath with dispersing agent using high speed stirrer for 10 minutes. The microglobules so formed were filtered, washed and dried at room temperature.

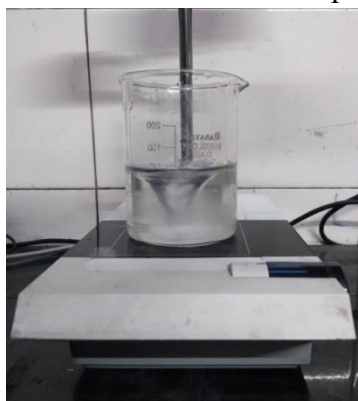


Figure 6: Synthesis of Microcapsules



Figure 7: Filtered product

b. Treatment of Cotton Sample with Synthesized microcapsules

In the beaker 200 ml of water was taken. Synthesized micro capsules, 0.25gm 1,3,5-trichlorotriazine and cotton fabric sample were treated together at room temperature for half an hour using a magnetic stirrer. The treated sample were washed, dried at room temperature and stored.

c. Dyeing

The cotton samples were dyed using hetero bi-functional reactive dyes using 0.25% and 2% shades. The sample dyed with dye stock solution, Glauber's salt & soda-ash in Introduced the given fabric sample in prepared dye-bath and treat in this dye-bath at room temperature for 10-15 min. Then gradually raise the temperature of dye-bath up to the boil. Add calculated amount of sodaash after 30 min. The dyeing is continuing at this temperature for further 30 min. Then cool down the temperature of dye-bath and take out the cotton sample & wash thoroughly with water. The dyed samples then rinsed, washed and soaped followed by drying.

d. Characterization of microcapsule

a. Analysis of particle size:

The particle size and size distribution of the micro capsules were analysed on particle size analyzer (Malvern instrument, MAL501131, DTS version 5.03, U.K.) Atomic Force Microscope (easy Scan 2 Nanosurf AG, Switzerland.) operating in a contact mode (cantilever force constant 3 N/m) was employed for visual observation of the particles.

b. Scanning Electron Microscopy (SEM) Analysis:

Treated and untreated sample were examined in Scanning electron microscope (SEM). The image view of treated cotton sample was shown the application of extracted amino acids. Treated and untreated samples were analysed on Scanning electron microscope using JEOL JSM -5610 LV equipment.

c. Repeated washing of the treated samples

The treated samples were examined for fragrance retention after number of washings which has given idea of the fragrance retention on clothing . The washings were given according to the ISO 105-C10, 2006 standards using 5gpl soap solution and 2gpl soda ash solution at 60° for 30 minutes to examine the fragrance retention after the washing procedure. After the repeated washing, the samples were subjected to olfactory analysis by the panelists for fragrance intensity ratings.

d. Olfactory Analysis :

It is one kind of sensory analysis to establish the odour concentration which employs a group of panelists. The odor concentration categories according to the intensity. The odour grading scale was 0

to 5. Interpretation; 0 → No odour, 1 → Very weak odour, 2 → Weak odour, 3 → Medium odour, 4 → Strong odour and 5 → Very strong odour.

e. Testing of Antibacterial property : [AATCC 147]

Microbiological studies of many types have been carried out according to American Type Culture Collection (AATCC, USA, US Pharmacopeia Norms). The objective of this test method is to detect bacteriostatic activity on textile materials, according to test method AATCC 147 (Parallel streak method). The method is useful for obtaining a rough estimate of activity in that the growth of the inoculums organism decreases from one end of each streak to the other and from one streak to the next resulting in increasing degrees of sensitivity. The size of the zone of inhibition and the narrowing of the streaks caused by presence of the antibacterial agent permit an estimate of the residual antibacterial activity after multiple uses. The antibacterial activity was checked against both gram positive bacteria *S. aureus* and gram negative bacteria *E. Coli* obtained from an overnight culture were suspended in nutrient broth. The samples were then placed on the inoculated medium and the plates were kept for incubation for 24 hours at 37°C. The inhibition zones were then observed.

f. Determination of K/S of Dyed Samples

The colour strength in terms of K/S value was measured using computer colour matching system interfaced with Spectro photo meter.

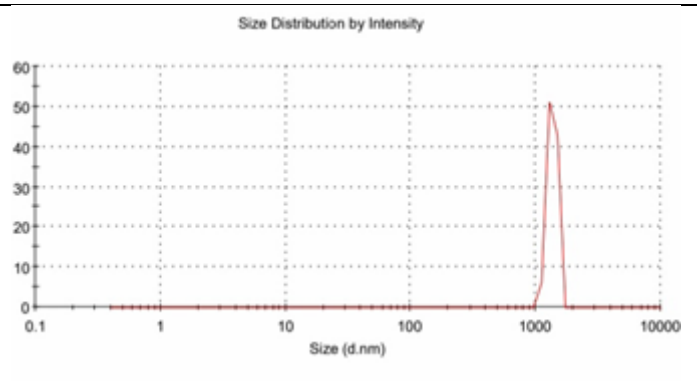
4. RESULTS AND DISCUSSION

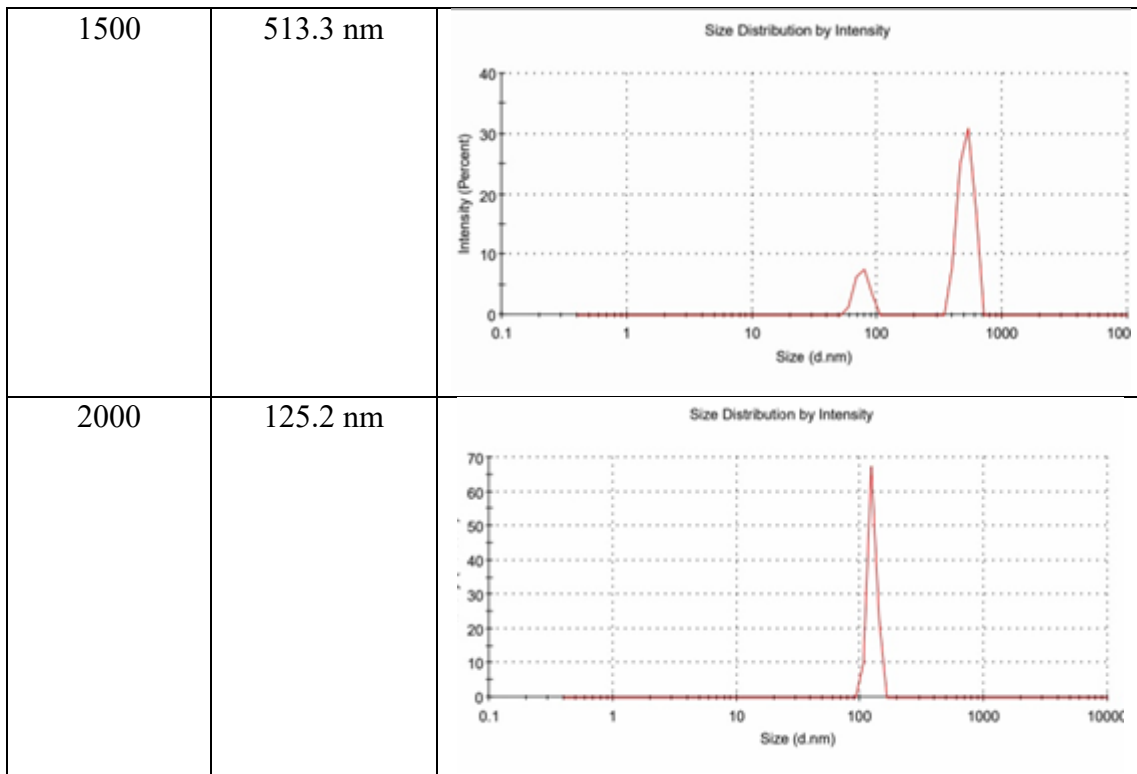
Micro-capsulation provides a means to enable us to have controlled loading and retaining of active substances. In this study microcapsules containing essential oils were encapsulated by phase separation method. Essential oils such as clove oil, cinnamon oil and sandal wood oil were micro-capsulated for fragrance release applications.

a. Optimization of stirring speed on the size of the microcapsules

During the preparation of the microcapsules the stirring speed of stirrer was varied to have a control on the size of the resultant capsules. The size of the capsules was measured using Malvern particle size analyser at three levels of the stirring speed viz. 500 rpm, 1500rpm and 2000 rpm. It is seen from the table that as the speed of the stirrer was increased the size of the capsules was also reduced. At a speed of 2000rpm the size of the microcapsules were reduced to 125.2 nm. Thus, by this method a very small size of the microcapsules were synthesized.

Table 1: Effect of stirring speed on the size of the microcapsules

Stirring Speed (RPM)	Size of the Microcapsules	Size Distribution
500	1357 nm	



b. Application of Fragrance loaded microcapsules on cotton fabric

Cotton fabric samples were anchored to fragrance loaded microcapsules using 1,3,5-trichlorotriazine as anchoring agent. After the completion of treatment time the samples were cold rinsed and dried and stored. After storing the samples in open air for 48 hours. The samples were distributed to five male judges for odour evaluation. Individual scoring sheets were provided to the judges. The odour grading scale was 0 to 5. Interpretation 0 → No odour, 1 → Very weak odour, 2 → Weak odour, 3 → Medium odour, 4 → Strong odour and 5 → Very strong odour.

It is seen from the table 2 that the fabric samples treated with clove oil encapsulated capsules scored highest, followed by the sample finished with cinnamon oil micro capsules and sandal wood oil microcapsules.

Table 2: Evaluation of fragrance feel of the fabric finished with fragrance loaded microcapsules by organoleptic evaluation method

Sr. No.	Sample	Judge-1	Judge-2	Judge-3	Judge-4	Judge-5	Avg Score
1	Sample finished with Clove oil encapsulated microcapsules	5	5	4	5	5	4.8 (Strong-Very Strong)
2	Sample finished with sandal oil	2	3	1	1	3	2 (Weak)

	encapsulated microcapsules						
3	Sample finished with cinnamon oil encapsulated microcapsules	3	4	3	3	4	3.4 (Weak-Medium)

The fabric samples were subjected to repeated washing and then given again to the previous judges for grading the score. It is seen from the table 3 that repeated washing of the samples reduced the fragrance level but still contains about 75% , 60% and 70% fragrance for samples finished with clove oil, sandal wood oil and cinnamon oil encapsulated samples.

Table 3: Effect of repeated washing on fragrance score of the finished samples

Sr. No.	Sample	Judge-1	Judge-2	Judge-3	Judge-4	Judge-5	Avg Score
1	Sample Finished with clove oil encapsulated microcapsules	3	4	3	4	4	3.6
2	Sample finished with sandal oil encapsulated microcapsules	1	2	0	1	2	1.2
3	Sample finished with cinnamon oil encapsulated microcapsules	2	2	2	3	3	2.4

c. Examination of scanning electron microphotographs of the sample finished with microcapsules.

The fabric sample after the treatment was examined using Scanning electron microscope JEOL JSM -5610 LV equipment. The microphotograph of the sample is displayed in Figure 8. Since the microphotographs of the microcapsules treated sample with limited magnification was not detectable, the microcapsules were pasted on black paper and visualized as shown in Figure 9.

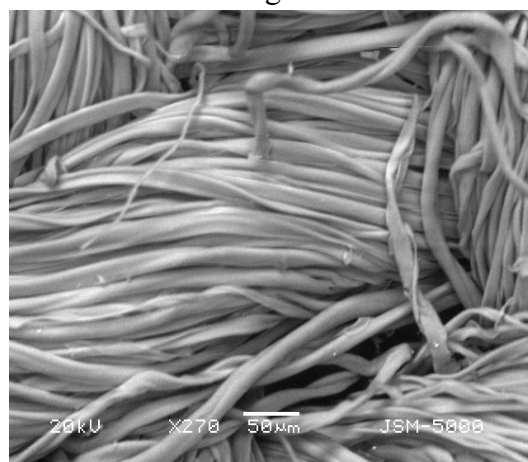


Figure 8: Scanning electron microphotographs of the sample treated with clove oil encapsulated microcapsules

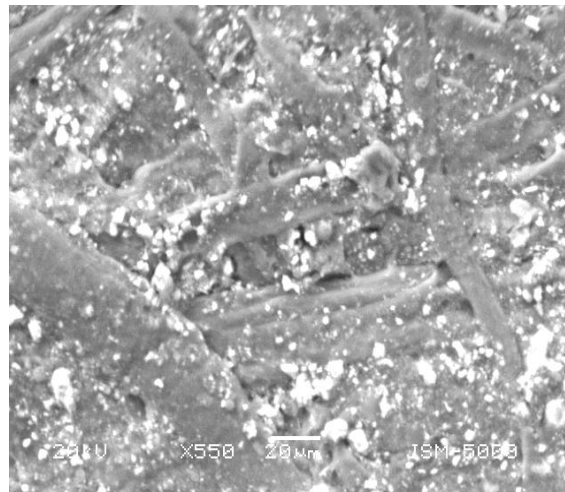
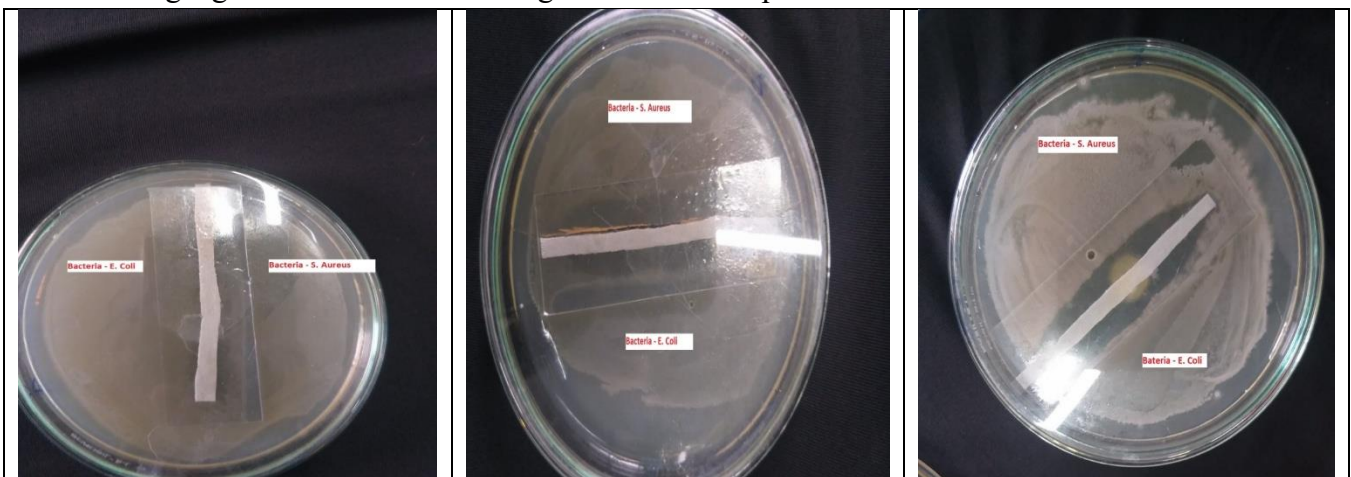


Figure 9: Scanning electron microphotographs of the sample treated clove oil encapsulated microcapsules on paper

d. Evaluation of the Anti-Bacterial Activity of the Fabric by Qualitative Test Method (AATCC 147)

The results of agar diffusion method against the standard test organisms *S. aureus* (Gram positive) and *E. coli* (Gram negative) are given in figure 10 (a), (b) & (c) There is a clear zone of inhibition around the fabric treated with clove microcapsules against both the test organisms in contrast with untreated fabric sample which allowed the growth of organism. The effect of sandal oil was found to be less effective compared with the clove oil. Clove oil has biological activities, such as antibacterial, antifungal, insecticidal and antioxidant properties. It is well know that both eugenol and clove essential oil phenolic compounds can denature proteins and react with cell membrane phospholipids changing their permeability and inhibiting a great number of Gram-negative and Gram-positive bacteria.



<p>Figure 10 (a): Evaluation of the Anti-Bacterial Activity of the untreated sample</p>	<p>Figure 10 (b): Evaluation of the Anti-Bacterial Activity of the sample finished with sandal wood oil encapsulated microcapsules</p>	<p>Figure 10 (c): Evaluation of the Anti-Bacterial Activity of the sample finished with clove oil encapsulated microcapsules</p>
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e. Assessment of Dye Ability of the samples

The samples were dyed and then treated with Essential oils. The results are shown in Figure 11 and Table 4 . Interestingly the samples treated with the microcapsules have found to give slightly higher colour depth compared with the untreated samples.

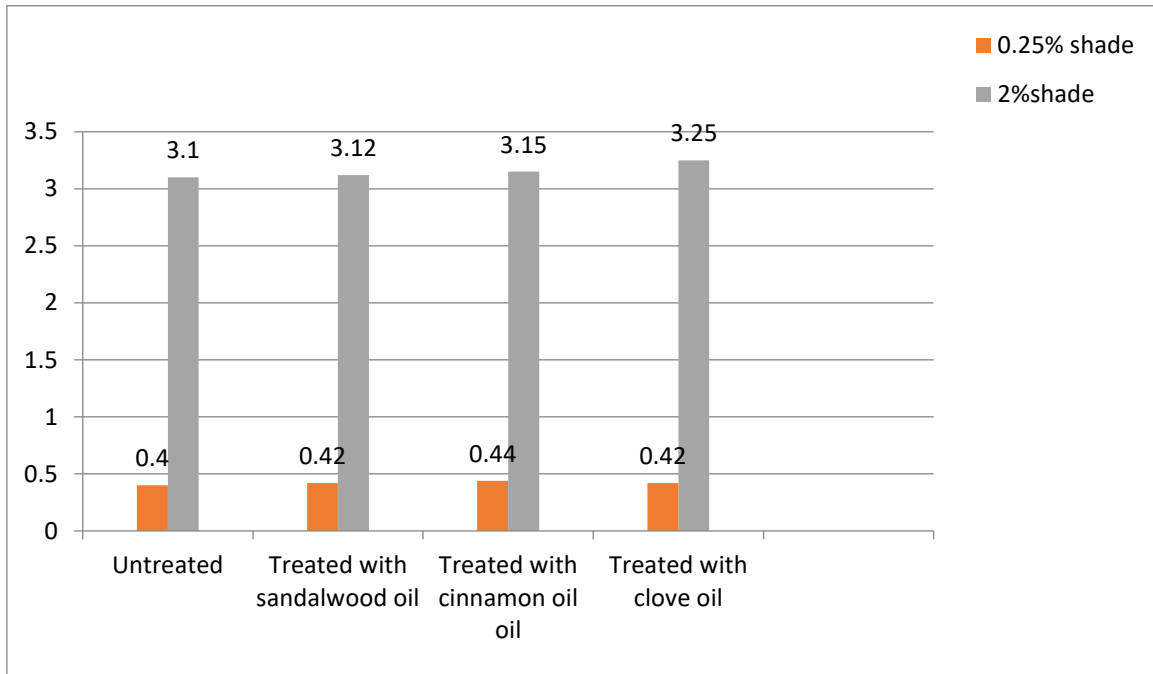










Figure 11: Dyeing results of the samples for Corafix N. Blue SFB

Table 4: Actual dyed samples [Dye: Corafix N. Blue SFB]

Sample	Percentage Shade	
	2%	0.25%
1. Untreated		
2. Treated with sandalwood oil		
3. Treated with cinnamon oil		
4. Treated with clove oil		

5. CONCLUSIONS

The Microcapsules with encapsulated fragrance oil were successfully synthesized. The size of the microcapsules was found to be highly dependent on the stirrer speed. A stirrer speed of 2000 rpm could generate as small as 125.2 nm microcapsules. Cotton fabric samples were anchored to the microcapsules using 1,3,5-Trichlorotriazine and the fragrance intensity was judged by five panellists. The average odour intensity as judged by the judges was as follows: Sample finished with Clove oil > Sample finished with Cinnamon oil > Sample finished with Sandal wood oil. The odour intensity of the samples was checked after repeated washing and it was found that the sample could retain about 60-75% of the smell as judged by the panellists. Characterization of the synthesized microcapsules were studied using particle size analyser and scanning electron micrographs. The antibacterial activity of the samples was qualitatively assessed for the clove and sandalwood oil finished samples. There was a clear zone of inhibition found around the fabric treated with clove microcapsules against both the test organisms in contrast with untreated fabric sample.

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