

AROMA CUM ANTIBACTERIAL FINISHING OF COTTON WITH INCLUSION COMPLEX OF β -CYCLODEXTRIN-CO-CHITOSAN AND OIL OF LAVENDER

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Abstract: Host-guest complex of β -cyclodextrin-co-chitosan and lavender was employed for the combined aroma cum anti-bacterial finishing of cotton textiles. The modified host viz. β -cyclodextrin-co-chitosan was synthesized with the use of β -cyclodextrin citrate as the intermediate for the achievement of the satisfactory yield of the host. FTIR analysis was carried out for the characterization of β -cyclodextrin-co-chitosan and also the surface modification of the functionalized cotton with the modified host. ¹HNMR spectra of the inclusion complex of β -cyclodextrin-co-chitosan and Lavender had confirmed the formation of a strong complexation between the two compounds. The functional as well as physical properties as aroma retention, anti-bacterial assessment, tensile strength, crease recovery, bending length and air permeability were investigated both for the host-guest complexed cotton and lavender alone treated cotton. The aroma retention and a strong inhibition zone was found more pronounced in the functionalized cotton instead of the oil alone treated cotton, which can be a promising and potential cadre of application of multi faceted textiles.

Keywords: Host-guest, β -cyclodextrin-co-chitosan, anti-bacterial, β -cyclodextrin citrate, FTIR, ¹HNMR

1. INTRODUCTION

Aromas have been used widely in different fields such as textile, medicine, food, tobacco, leather, papermaking, cosmetics and many more because of their antibacterial effect, sedative effect and tranquillization[1-3]. Nowadays, finishing a textile with aroma is an important commercial target and an engineering challenge. Pure aroma compounds and essential oils have been used traditionally in folk medicine for a long time. Aroma finishing has been increase in demand since last one decade, as well as tremendous competition in the market, opens up opportunities for value addition to all forms of textile materials[4]. Over the last 50 years, plants are utilized as a potential source of natural aromas in the form of essential oils. It is reported that the essential oils with high aromatic index offers advantage of insect

repellency and prohibit the growth of microorganisms[5]. Aroma finished textiles are an important entry into the field of textiles for trend conscious people[6]. A fragrant finished fabric is the one, where, the aroma is attached and incorporated into the fabric surface in a way that the aroma is slowly released, refreshing the mood and emotions of human being. Finishing a textile with long term aroma and with not much change in physical properties of textile is a desirable commercial goal as well as a textile chemistry and engineering challenge with two aspects; the amount of aroma applied to the textile must have good durability and the aroma is released in a slow and controlled manner. The aromatic matter was added in traditional dyeing, printing or finishing processes producing a perfumed fabric. However, the durability of aroma imparted was analyzed to be poor. Addition of aromatic matter into synthetic fiber polymer during fiber formation can create a fragrant fiber that exhibit good aroma durability. But, the limitations of this method was the time from fiber formation to final product, loss of fiber properties due to the presence of aroma, loss of aroma during textile processing, difficulty in changing the aroma, and inability to use this technique on natural fibers[7]. Many other techniques are also available to impart aroma into textiles, however, the need and development of the techniques which not only enhance the slow release property but also not affect the properties of textile is yet to be clearly defined.

The aroma compounds are volatile substances that can easily vaporize to give sense of smell. The most difficult task in preparing the aroma therapeutic textile is to prolong the lifetime of aroma. Micro-encapsulation technique is an effective solution of this difficulty[8,9] but it suffers from a number of limitations as external stimuli is needed to activate the microcapsules, loading of microcapsules can be carried out once in their life time and fabric hand gets stiffer as binders are a necessity for adhering and

fixing microcapsules to the textile surfaces. Instead, the macromolecular containers as cyclodextrins can be preferentially loaded with the newer guests every time after the depletion of the guest molecules and fabric handle is also maintained softer without any adverse effect on other functional properties. Also, β -CDs considered as the environmental friendly microcapsules because they are biodegradable and non-toxic[10].

The cyclodextrins are produced by certain micro-organisms of cultivated starch and are non-reducing cyclically linked oligosaccharides, which are capable of forming inclusion compounds with molecules that fit into their cone-shaped hydrophobic cavity[11]. These cavities of cyclodextrins can hold liquid, gases and solid particulates properly and can act as a slow releasing host with the prospects of refilling the cavities again with a variety of guests. As a result of the inclusion compounds between the host-guest associations, the physico-chemical properties of the compounds are changed, i.e. the vapour pressure of volatile substance is reduced, stability against light or air is enhanced, harmful and/or unpleasant odour in the surrounding may be eliminated[12]. No hydrogen and covalent bonds are formed or broken during the formation of such host-guest complexes[13]. β -Cyclodextrin (β -CD) is the most commonly used derivative of cyclodextrins as these are the cheapest, nontoxic for oral use, causes no skin irritation, no skin sensitisation, no mutagenic effect, does not cause any problems in water and are also biodegradable[14]. β -CD can be incorporated onto textile by means of spraying, printing, padding, grafting, surface coating, impregnation and ink jet printing[15] but it suffers from a major limitation that it has no inherent affinity for any fibre. Different mechanisms are used to fix β -CD on textile substrates viz. grafting with the use of crosslinking agents such as polycarboxylic acids onto cotton, [15,16,17] wool,[15,18] polyester [19,20] polyamide [21] and polyacrylonitrile fibres [22]. Resins such as epichlorohydrin can also be used to fix β -CD to cellulose [23,24]. The derivatives as Monochlorotriazine β -CD and hydroxypropyl β -CDs are also gaining importance among durable host-guest complexes. Chitosan is utilized in the textile industry as an antimicrobial agent due to its inherent ability to provide protection against allergies and infections, diseases, coupled with moisture retention and wound healing capabilities [25].

Thus, an attempt was carried out to synthesize β -CD-co-chitosan, characterization with FTIR and HNMR for the development of inherently durable fragrant cum anti-microbial textiles for hi-end niche market with the infusion of lavender essential oil.

2. EXPERIMENTAL

2.1 Materials

Thoroughly pre-treated 2/1 twill woven cotton fabric possessing epi (64), ppi (78), warp (2/40 Ne^s), weft (2/20 Ne^s) and gsm (236) was used as control for finishing with Lavender essential oil for aroma as well as anti-microbial finishing. All the analytical grade chemicals, viz. β -cyclodextrin (β -CD; M.W~1134.98 g/mol), citric acid anhydrous (M.W 192.129 g/mol), formic acid, sodium hypophosphite, silicon softener, sodium hydroxide, ethanol, oil, Isopropanol and lavender were supplied by SDFL, Mumbai. Chitosan (M.W 50000 g/mole) was sourced from India sea food (regd.) from Kochi, Kerala. Water bath (Laboratory glassware co. Ambala), Electronic pH meter (PH-009(I)), Electronic weighing balance (CAS Model MW -11 series), UV/Visible spectrophotometer (Lab India analytical UV 3000⁺), Orbital shaker (Bio-Technology Lab, M.D.U Rohtak). Padding mangle (Electronic and Engg. Company), Drying oven (Kaypee udyog, Ambala), Laundrometer (RBE, Mumbai), Digital Tensile strength tester (Globe Tex Industries), Shirley Stiffness tester, Crease recovery tester (Shirley), Air permeability tester (Prolific) and FTIR (Perkin Elimer) were used for the assessment of aroma stability; anti microbial activity (AATCC: 147-2004) and effect on physical properties as Tensile strength (IS: 1969-1968), Stiffness (IS: 6490-1971), Crease recovery (IS: 4681-1968) and Air permeability (IS: 11056-1984).

2.2 Methods

2.2.1 Calibration and Optimization of lavender for aroma analysis

Calibration of lavender was done by measuring the aroma of different concentrations of lavender as (1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 %) in 100% ethanol. Maximum wavelength (λ_{\max} ~344nm) of lavender was measured by taking any concentration of oil in ethanol as a blank sample through UV/Visible spectrophotometer. Concentration versus absorbance curve was plotted on UV/Visible spectrophotometer according to Beer Lambert's law. A calibration curve was thus obtained and it was used for the detection of unknown concentration of aroma in the sample.

Ten oil concentrations (1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 %) in 100% ethanol were prepared. Ten cotton samples were independently immersed in each concentration of oil for 1 min, followed by padding (2dip, 2nip) and Drying (at room temperature). The release of oil was investigated for the optimization of oil concentration with the extraction of oil as- Extraction of oil was done by taking 0.1g of each

treated sample, immersing in 100 % ethanol with 1:50 ML followed by heating of test tube at 50°C in water bath for 5 minutes, cooling and finally removing the samples from the solutions. Absorbance of each concentration of lavender was measured with the help of UV/Visible spectrophotometer at $\lambda_{\text{max}} \sim 344\text{nm}$. The same procedure was repeated after a time span of 2 hours each up to 72 hours by exposing samples in dirt and dusty environment. Effect of time on aroma retention was also measured by calculating unknown concentration of aroma.

2.2.2 Synthesis and characterization of β -CD-co-chitosan

The synthesis of β -CD-co-chitosan was carried out according to the previous procedure without any major modification i.e. by a two step process-synthesis of β -CD citrate followed by its modification for the production of β -CD-co-chitosan. β -CD citrate concentration (0.54g) was introduced into a solution containing chitosan (0.6g) dissolved in formic acid concentration (0.4ml/1g chitosan) using M:L (1:15). The reaction mixture was then magnetically stirred and heated at 100°C for 3 hour at the end the reaction, the product was precipitated by adding 100 ml of NaOH solution (0.2 N). The sample was thoroughly washed with distilled water till natural (pH- 7) to ensure the removal of un-reacted β -CD-C. Finally, treated cotton was washed with acetone and oven dried at 60°C for 24 hours²⁶. FTIR was used to characterize the synthesized component.

2.2.3 Application and characterization of β -CD-co-chitosan on cotton

Cotton was treated with 80 gpl of synthesized β -CD-co-chitosan in the aqueous medium of formic acid to aid its solubility via pad, dry, cure and rinsing technique on cotton. FTIR was used to characterize the presence of β -CD-co-chitosan on cotton as a modification of control.

2.2.4 Treatment of control and β -CD-co-chitosan functionalized cotton with lavender

Control and β -CD-co-chitosan functionalized cotton were treated with lavender by taking its optimized concentration as discussed in section 2.2.1. Cotton was immersed in the optimized concentration with 100 % ethanol (v/v) for 1 minute, padded at 1 Kpa (2dip and 2 nips) and finally air dried at room temperature. ¹H-NMR spectra was investigated for the inclusion complex formation between β -CD-co-chitosan and lavender.

2.2.5 Performance evaluation of oil treated cotton

The treated cotton was evaluated for the aroma release; Qualitative and Quantitative assessment, laundering durability, tensile strength, bending length, crease recovery, air permeability and antimicrobial activity. Comparative analysis of physical and functional properties was performed for oil treated non-functionalized and β -CD-co-chitosan functionalized cotton. **Qualitative Aroma release** was carried out as – Intensity of scent was performed by a group of 5 persons in which, standard sample of each recipe was first given to the panel to rank the test specimen. Evaluation was done for the treated samples which were packed in an airtight polythene bag so that there was no release of aroma in between evaluation due to air or light. Panel was allowed to take 3-4 whiffs for each sample in an open corridor and rank them in prepared rating scale from 1 to 5. Coding of preference was done and analyzed to obtain relative results of scent intensity. The rating was between ‘5’ which meant the strongest scent and ‘0’ which meant no scent. The higher the rating, the stronger the aroma intensity on the finished sample would be. The aroma retention was observed for 72 hours after the treatment. **Quantitative Aroma release-** UV/Visible spectroscopy was used for the quantitative evaluation of % retained aroma as- The aroma was extracted from 0.1g of cotton samples (oil alone and host- oil complex) immersed in 100 % ethanol with 1:50 ML ratio then test tube heated at 50°C in water bath for 5minutes, cooled it, followed by removing the cotton samples from the ethanol solutions. Absorbance of the extracted aroma was measured with UV/Visible spectrophotometer at λ_{max} . The observations were taken at 0, 2, 4, 6, 12, 24, 48 and 72 hours to find the % retained aroma intensity after predetermined time span and to evaluate the aroma release rate. Release rate was calculated using Equation 1.

Equation 1:

$$\text{Release rate of fragrance} = \frac{\text{Immediate concentration} - \text{concentration after hours}}{\text{Immediate concentration}} \times 100 \quad \text{-- 1}$$

Laundering durability was assessed as- 4 cm x 10 cm strips of oil treated cotton were washed according to ISO 105-C01:1989 with 5 gpl of soap solution with M:L of 1:50 at 40°C ± 2°C for 30 minutes. Washed cotton were rinsed with running tap water for 10 minutes and dried at room temperature. % retained aroma was observed according to the procedure earlier mentioned. **Tensile strength** of cotton was carried with a gauge length of 8” inches (IS: 1969-1968). **Bending length** was measured with IS:6490-1971, **Crease recovery** was measured with a test

method (IS:4681-1968), **Air permeability** was determined by testing using (IS:11056-1984) and AATCC Test Method 147-2004 standard was followed for **anti-bacterial testing**. Parallel streak method is a qualitative method to determine antibacterial activity of diffusible antimicrobial agents on treated textile materials. The average width of a zone of inhibition along a streak on either side of the test specimen was calculated using the following equation: $W = (T - D)/2$; where: W = width of clear zone of inhibition in mm, T = total diameter of test specimen and clear zone in mm, D = diameter of the test specimen in mm.

3. Results and Discussion

3.1 Calibration and Optimization of lavender

The lavender was required to be calibrated against blank at λ_{max} for the evaluation of % retained aroma on cotton. The graph was plotted between absorbance values (Abs) and concentrations of lavender (Conc.) in mol/l. The slope of the linear line was calculated. The equation is: $y = mx + b$ or $Abs = (Slope \times Conc.) + 0 = 0.088 \times concentration$ as shown in Figure 1. The optimization of lavender was obtained at λ_{max} using UV/Visible spectrophotometer after time spans of 2 hours each up to 72 hours as shown in Table 1 and retained fragrance oil with time in Figure 2.

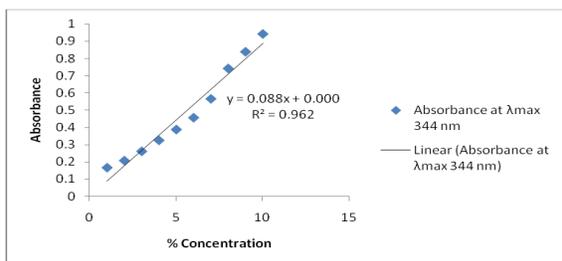


Figure 1. Calibration curve of Lavender oil

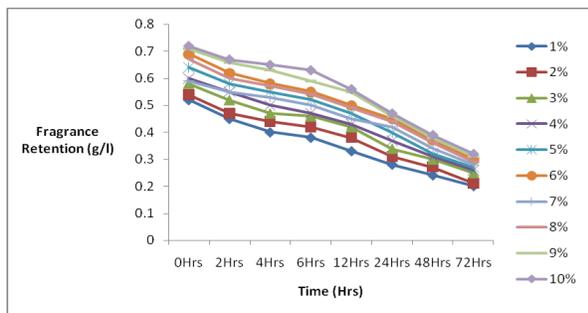
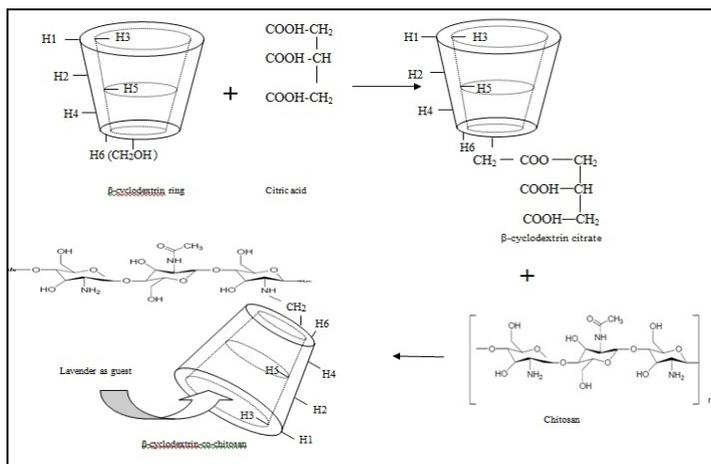


Figure 2. Effect of time on fragrance retention at various concentrations

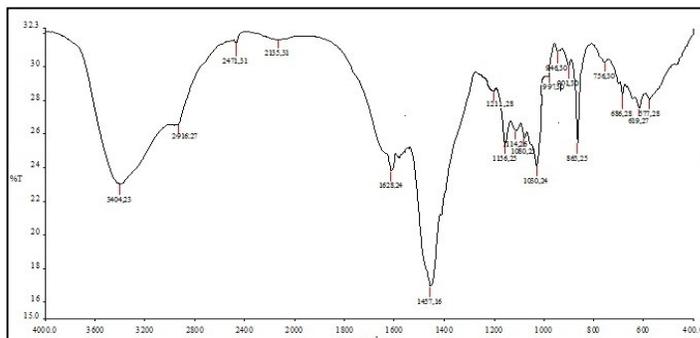
It was observed that at 6% lavender, maximum absorbance was obtained which indicated that at this concentration, maximum lavender was deposited on cotton. Higher absorbance indicated more quantity of lavender extracted into the solution and hence confirmed the presence of more lavender on the cotton too.

3.2 Synthesis and Characterization of synthesized β -CD-co-chitosan

The reaction mechanism of β -CD-co-chitosan synthesis is shown below in Reaction 1. It can be inferred that the β -CD citrate was used as the intermediate for the attachment of chitosan to the β -CD ring, which was later removed as citric acid in the form of by-product and further neutralized in the presence of an alkali. The degree of substitution of chitosan with β -CD rings varied as per the citric acid concentration.



Reaction 1. Synthesis mechanism of β -CD-co-chitosan



a)

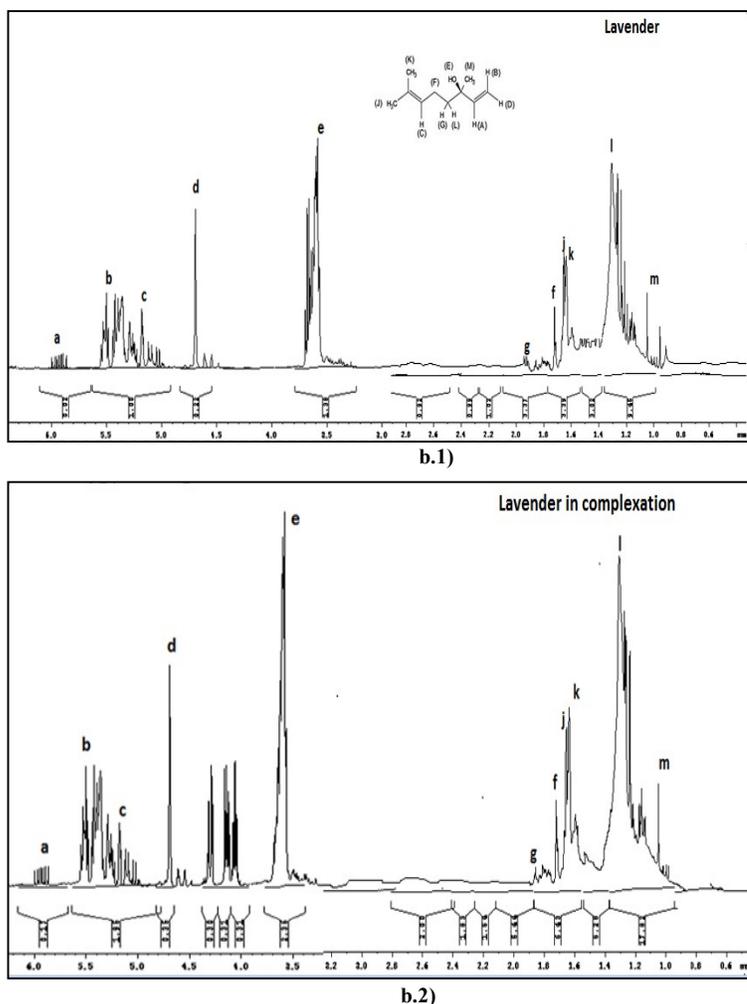


Figure 5. HNMR of a) β -CD in pure and complexed form with lavender b.1) lavender in pure state b.2) lavender in complexed state with β -CD (concentration of pure compounds ~ 10mM; concentration of β -CD: lavender ~ 5mM: 5mM)

The respective chemical shifts ' δ ' and induced chemical shifts ' $\Delta\delta$ ' (The induced shift, is defined as the difference in chemical shifts in the absence and in the presence of the other reactant) as detailed in Table II. The chemical and induced chemical shifts of β -CD and lavender in free as well as in compounded state had revealed evident shifts in the β -CD and oil in free and complexed state. This signified the formation of dynamic complexes between the anchor and the guest. The chemical shifts had shown significant entrapment of oil molecules into the cavities as the induced shifts of proton H5 > H3 was found for lavender.

The above table shows that induced chemical shifts were pronounced in both the host (β -CD-co-chitosan) and the guest (Lavender) during complexation process. H3 and H5 protons in β -CD-co-chitosan and H_j and H_k protons in lavender had shown the maximum shift when complexed together. This had

also shown that no newer peaks were formed in the complexed spectra of host-guest system; that could be assigned to the fact that complexation was a dynamic process, the included oil being in a faster exchange between the free and bound states.

3.5 Performance evaluation of lavender treated control and functionalized cotton

3.5.1 Qualitative analysis-

Aroma evaluation was done qualitatively by panel judgment for scent intensity as shown in Table III. Cotton finished with both the techniques gave noticeable scent intensity after 24 hours but cotton treated with lavender alone did not retain any aroma after 24 hours, on the other, oil treated functionalized cotton was scented even after 72 hours. The reason might be the cavities of β -CD that were capable of holding aroma of lavender. Along with this, ester linkages between cotton and β -CD-co-chitosan also stabilize as well as suppress the release of lavender. This was attributed to the slow release of lavender from the surface of cotton.

3.5.2 Quantitative analysis-

For the Quantitative assessment of the aroma retention, it was observed that there was an increase in release rate of fragrance from both categories of finished cotton with span of time i.e. about 91.72% oil was released from *lavender alone cotton* but only 36.33% was sublimated from the *Host guest-complexed cotton*. The initial release of oil was primarily dependent upon the presence of oil either alone on the fabric or in the complex form. Minimum aroma oil's release rate was observed from cotton with host-guest complex (β -CD-co-chitosan and lavender) as per the quantitative evaluation of extracted lavender from the lavender treated cotton as it retained maximum fragrance even after 72 hours. The reason might be the linkage of β -CD chitosan and cotton to retain the fragrance even after long span of time as seen in Figure 6.

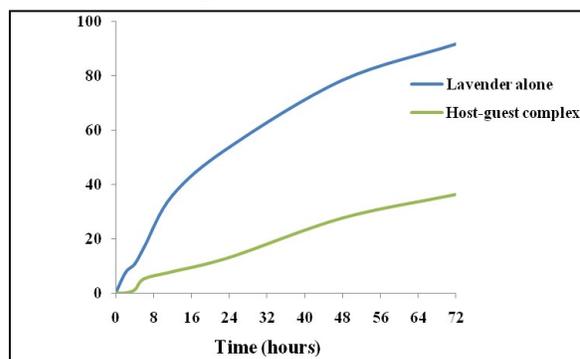


Figure 6. % Release of lavender from treated cotton with time

3.5.3 Laundering durability

The durability to laundering of the lavender treated cotton was investigated as shown in Figure 7. Lavender when applied alone on cotton gave significant release in fragrance after three washing cycles. Aroma was released by almost 25.8 % after 3rd subsequent wash due the weak interactive forces between cotton and lavender resulting in its quicker release. On the other, host-complexed cotton had shown a significant improvement in aroma durability as aroma release rate was significantly low (10.53% after 3rd wash). This was attributed to the cavities of β -CD which were capable of forming inclusion complex with the lavender as guest. The lavender dimensionally fitted into the cavities of host molecules, thereby, releasing the aroma in a suppressed manner. Thus, to improve the retention of fragrance for longer duration, lavender was enclosed with β -cyclodextrin as host.

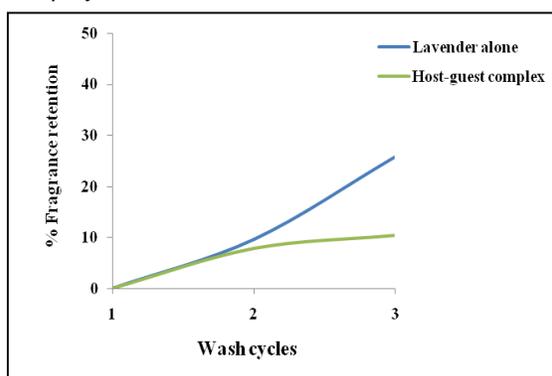


Figure 7. Effect of wash treatments on % fragrance retention of treated cotton

3.5.4 Effect of aroma treatment on physical properties of cotton

The effect of aroma treatment on physical properties of cotton alone and host-complexed cotton is detailed in Table IV.

Tensile strength (TS) had shown an increase when cotton was finished with lavender alone and also with the complex of β -CD-co-chitosan and lavender. The tensile strength of cotton finished with β -CD-co-chitosan and lavender shows higher tensile strength of the two treatments due to the ester linkage between modified host and hydroxyl groups of cotton which lessened the restriction of segmental movement of cellulosic chains in the fiber and prevented the fiber from being tendered severely. The above results had shown that host-complex treatment had added bit **stiffness**

(**BL**) to the treated cotton due to mechanical deposition of finishing chemicals in the open interstices of the fabric or due to cross linking reaction between cellulosic molecules of fiber and β -CD-co-chitosan; due to this, extra cross linking chains were developed between fiber segments that increased the abrasion resistance of fiber segments, thereby restricting the bending of fibers which made the fabric more rigid. The change in the **crease recovery (CR)** angle of treated cotton is elucidated in the above Table. Crease recovery angle of cotton treated with β -CD-co-chitosan shows minimum recovery angle as compared to the other treatment due to the deposition of finishing chemicals in the pores of cotton and physical bonding between adjacent molecular chains of cotton and chitosan. There was a noticeable change in the **air permeability (AP)** of treated cotton due to the closing of open interstices due to mechanical deposition of β -CD-co-chitosan as a film on cotton. In addition, swelling of hydrophilic cotton would had changed the fabric porosity and thickness that resulted in decrease of air permeability of both the treated cotton as compared to control.

3.5.5 Assessment of anti-bacterial activity of control and treated cotton

The three specimens of control, lavender treated cotton and lavender treated functionalized cotton were investigated for ant-bacterial activity. Nil activity was seen for the control (A), little was observed for the lavender alone treated cotton (B) and maximum inhibition zone was observed for cotton finished with host-guest complex of β -CD-co-chitosan-Lavender (C). The reason might be the presence of chitosan, as it is a cationic antimicrobial agent particularly for external disinfection and the target site of the cationic biocides is the cell envelope of bacteria. Chemical modification of chitosan via it's coupling with β -CD improves its antimicrobial activity due to the enhancement of dissolution of chitosan and the creation of carboxyl groups along with chitosan molecules serve in the dissolution of phospholipids area and causing leakage of intercellular components and finally the death of the microorganisms as shown in Figure 8.

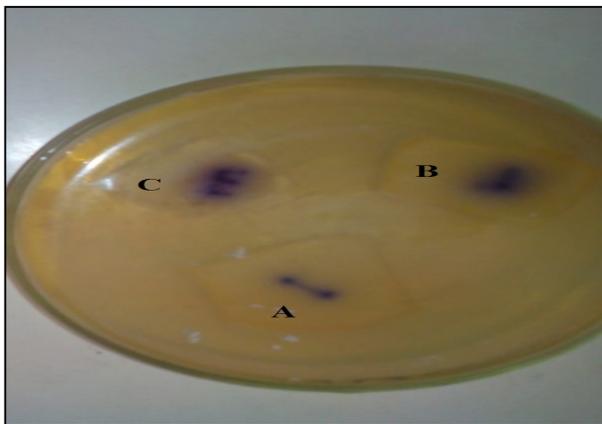


Figure 8. Inhibition of control and treated cotton A. control B. Lavender alone treated cotton C. Lavender treated on host-guest complexed cotton

4. CONCLUSIONS

β -CD-co-chitosan was synthesized and characterized for the development of modified host-guest system to impart aroma and anti-bacterial finishing. Aroma release (Qualitative and Quantitative) and laundering durability was excellent for the β -CD-co-chitosan and Lavender inclusion complex rather than when lavender treated alone on cotton. The physical properties viz. tensile strength had shown an improvement due to the use of the modified host, on the other, bending, crease recovery and air permeability were only marginally affected. The combination of chitosan and lavender had shown appreciable antibacterial activity and inhibition zone for the development of antibacterial cum aroma finished textiles.

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Table I. Absorbance index of lavender at various concentrations

Oil%	Absorbance Index							
	Time span (hours)							
	0	2	4	6	12	24	48	72
1	0.029	0.023	0.018	0.016	0.012	0.007	0.003	0
2	0.031	0.025	0.022	0.02	0.016	0.01	0.006	0.001
3	0.035	0.029	0.025	0.024	0.02	0.013	0.009	0.004
4	0.037	0.032	0.027	0.025	0.021	0.015	0.01	0.005
5	0.04	0.035	0.032	0.029	0.025	0.018	0.011	0.006
6	0.045	0.038	0.035	0.032	0.027	0.023	0.015	0.009
7	0.036	0.032	0.03	0.027	0.023	0.02	0.013	0.007
8	0.043	0.037	0.034	0.031	0.026	0.022	0.014	0.008
9	0.047	0.042	0.039	0.036	0.032	0.024	0.016	0.01
10	0.048	0.043	0.041	0.039	0.033	0.025	0.017	0.011

Table II. Chemical shifts of Protons of β-CD and Lavender

Protons	β-CD-co-chitosan			Lavender oil			
	CS		ISA Δ δ	CS		ISA Δ δ	
	δ _{β-CD free}	δ* _C		δ _{L free}	δ* _C		
H3	3.8322	3.666	-0.1659	H_B	5.4424	5.5032	-0.0608
H5	3.7438	3.612	-0.1314	H_C	5.1804	5.1196	-0.0708
H6	3.7158			H_D	4.9653	4.6949	-0.0004
H2	3.5336			H_E	3.6036	3.5969	-0.0067
H4	3.4469			H_F	1.7231	1.7226	-0.0005
		H5> H3		H_G	1.8531	1.8591	0.006
				H_J	1.6580	1.6686	0.0106
				H_K	1.6486	1.6569	0.0083
				H_L	1.3742	1.3739	0.0003
				H_M	1.050	1.0498	-0.0002

CS= Chemical shift; IS= Induced Shift; δ_{β-CD free}= β-CD in free state; δ*_C= β-CD/ Lavender in complex; δ_{L free}= Lavender in free state

Table III. Qualitative evaluation of fragrance through scent intensity rating

Techniques	Scent intensity rating							
	0 hrs	2 hrs	4hrs	6hrs	12hrs	24hrs	48hrs	72hrs
Lavender alone	+++++	++++	+++	++	+	+	-	-
Host-guest complex	+++++	+++++	++++	++++	++++	++++	+++	+++

Table IV. Effect of aroma treatment on physical properties of cotton

Type of cotton treatment	TS Warp (kgf)	BL Warp (cm)	CR (θ)	AP (lph)
Control	50.1	2.275	99.1	39.1
Lavender alone treated	51.7(3.19%)*	2.4(5.49%)	100.5(1.41%)	35.00 (10.66%)
Host-guest complex treated	63.5(26.74%)	3.21(41.1%)	100.2(1.1%)	31.67 (19.16%)

*Data in Parenthesis(θ) shows the % change in the physical property subject to treatment