# **Fragrance Finishing of Cellulosic Fabrics**

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#### Abstract:

This work deals with terry towels treatment by using Reactive  $\beta$ -cyclodextrin to produce fragrance fabrics. Factors affecting on the performance properties (nitrogen content, towel wettability, braking strength and elongation) of towels such as  $\beta$ -cyclodextrin concentration, curring temperature, durability (washing) and storage time are studied. The obtained results by One-Way ANOVA (Analysis of Variance) and correlation coefficients show that the treatment of the fabric occurred by Reactive  $\beta$ -cyclodextrin concentration 30%, curring temperature 140 °C for 2 minutes, the percent loss of the fragrance was 18% and 55% after 20 washing cycles and 90 days respectively.

Keywords

- β-cyclodextrin,
- Perfume finishing,
- Terry towels,
- Wettability,
- Fragrance.

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#### 1. Introduction

Cyclodextrins compounds are novel environmentally safe complexing agents which are used to encapsulate fragrances compounds [1]. Cyclodextrins can be obtained by enzymatic degradation of starch, depending on the nature of the enzyme employed and how the reaction is being controlled; the main product is Figure 1 shows the different types of cyclodextrin. Each cyclodextrin unit has a hydrophobic cavity which can act as a host for a hydrophobic guest molecule. This property comes in useful for solubilizing and stabilizing highly hydrophobic molecules in solvents such as water. The application of Cyclodextrins to the textile industries has become more interesting, for example in dye stabilization, laser dyes [2], printing [3], color-changing compositions, longlasting perfumed cotton and the new auxiliaries [4]. Smart textiles as Perfumed, antibacterial and wound healing textiles [5-10]. Perfumed textiles are now available in the market. Perfuming of textiles can be achieved during either treatment or care. Incorporation of perfume retention agents during treatment such as cyclodextrins, elastomer latex/ synthetic resin emulsion, thermoplastic microcapsules and coating compositions were reported to bring about textile goods that can release fragrance over along period of time [1].

A terry towel is described as a textile product which is made with loop pile on one or both sides generally covering the entire surface or forming strips, or other patterns (with end hems or fringes and side hems or selvedges). The name "terry" comes from the French Word "Tirer", which means to pull out, referring to the pile loops which were pulled out by hand to make absorbent traditional Turkish toweling. Latin "vellus" meaning hair, has the derivation "velour", which is the toweling with cut loops [11]

Terry towels can be classified according to weight, production, pile presence on the fabric surfaces, pile formation, pile structure and finishing. Towels are also divided into groups according to end use and size as bath towels, hand towels, face towels, fingertip towels, kitchen towels and washcloths. In the terry towels there are four groups of yarns. These four groups are the pile warp, ground warp, weft (filling) and border weft. Towels are woven as 2, 3, 4, 5 or more pick terry weaves. The most common type is 3- pick terry toweling [12].

In the last few decades extensive researches have been conducted with regard to the physical properties of woven fabrics and terry towels [13, 14]. These works concentrated on abrasion resistance, tensile properties comfort and handle properties of woven and terry towels. This paper sheds light upon perfume finishing of terry towels and its effects on their physical prosperities. Terry towels were treated using Reactive  $\beta$  – cyclodextrin with different concentrations, the effects of Reactive  $\beta$  – cyclodextrin concentrations on towels weight, absorbency, and Nitrogen content were intended to be investigated. Also the effect of curring temperature, durability and storage time will be studied.

Cvclodextrin	Mass	Outer diameter,	Cavity d		Cavity Solubility,		Hydrate H <sub>2</sub> O [915]	
Cyclodextrim	Mass	(nm)	Inner rim	Outer rim	(mL/g)		cavity	external
α, (glucose) <sub>6</sub>	972	1.52	0.45	0.53	0.10	129.5	2.0	4.4
β, (glucose)7	1134	1.66	0.60	0.65	0.14	18.4	6.0	3.6
γ, (glucose) <sub>8</sub>	1296	1.77	0.75	0.85	0.20	249.2	8.8	5.4

# Properties of the main cyclodextrins



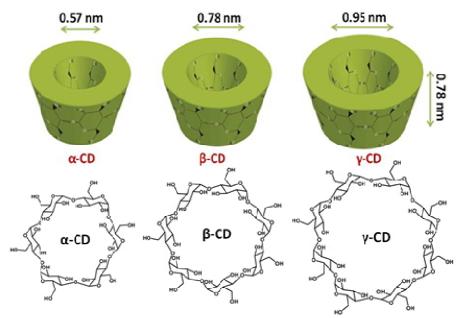


Figure 1. Structure of  $\alpha$ -cyclodextrin,  $\beta$ -cyclodextrin,  $\gamma$ -cyclodextrin [7].

### Methodology

### 2- Experimental

### 2.1 Materials

All terry towel samples were made from Ne 24/2 carded cotton yarn in the ground warp; Ne 16/1 carded cotton yarn in the ground weft; and Ne 24/2 carded cotton yarns for the pile. All warp ground, weft ground and pile yarns were made from Egyptian cotton Giza 86 whose physical properties were listed in table 1. All of the towels were 18 picks/cm weft density and 16 ends/cm warp density.

# Table 1. Characteristics of cotton used in<br/>producing the terry towels.

Mean fiber length, mm	31.7
Tenacity, g/tex	45
Elongation, %	6.2
Pressly Index	11.3
Micronaire value	3.9
Maturity, %	91
Fineness, millitex	140

All towel samples were woven on Smit terry towel Loom model TPS 600 and working width 360 cm With Electronic Staubli Dobby Type 2660. All of the terry towel samples were woven with the same weaving design. The design used in the fabrics is shown in figure 2.

Figure 2. weaving design used in the samples. G- ground warp , F: face pile warp; B: back pile warp

# 2.2. Chemicals used

Monochlorotriazinyl- $\beta$ -cyclodextrin referred here as reactive  $\beta$ -cyclodextrin (R-CD) was provided by Waker Chemie GmbH, Germany. Citric acid (CA) and sodium Hypophosphite (SHP) were of laboratory grade chemicals. Jasmine is also imported by El-Azhar, Cairo.

### 2.3. Procedures

Unless otherwise indicated, the conventional paddry-care method was applied. Thus the pad path containing R-CD, CA, SHP was prepared as follows: a small a mount of water was added to R-CD and the resulting solution warmed up until complete dissolution. The solution was then cooled down to the room temperature and the pad bath concentration was adjusted along with concurrent addition of CA and the catalyst (SHP). The concentrations of  $\beta$  – cyclodextrin were 10, 20, 30, and 40 mg/l. Terry towel samples were padded twice to a wet pick up of 100%. The treated towels were then dried at 358 K for 5 minutes, and then cured at 433K for two minutes. After that the terry towel samples were cooled to ambient temperature, then padded in an alcoholic solution of perfume oil to a wet pick up of 100% and then dried at ambient temperature for three hours before testing[1].

### 2.4. Laboratory testing

- Nitrogen content of the treated fabrics was determined by the Kjeldhal method [1].

- The ability of the terry towels to keep fragrance upon storing under ambient conditions in stagnant air for one week, two weeks, one month and three months respectively were determined by evaluating the percent loss in both weight gained and fragrance extent after the desired time as follows [1]. Percent loss in weight gained was estimated by calculating first the weight gained due to padding in the alcohol solution of the perfume oil as follows:

Weight gained = (A-B)

Where, A and B are the weights of the fabrics after and before padding in the perfume oil solution.

Percent loss in weight gained = 100 \* (C-A+B) / (A-B)

Where, C is the weight of the fabric upon storing.

The loss in fragrance extent was determined by comparing the extent of fragrance of the sample stored in air for a known time to that of a similar sample stored in a fragrance in a zip-locked plastic bag. Comparison was achieved independently by three persons using the smelling sense and the average of their estimations was recorded.

The wettability of the terry towels was determined by means of wetting time (absorbency) measurements before and after treatment according to AATCC Test Method 79 [1]. In this test method the fabric sample is placed over the top of a beaker so that the center of the fabric is unsupported. Distilled water was dropped on the fabric at 1 cm of the surface, and the time required to completely absorb the water drop was recorded as wetting time. The shorter wetting time indicates better fabric wettability. The different properties of terry towels treated with different cyclodextrin concentrations were listed in table 2. The percent loss in weight gained and the percent loss in fragrance at different storing times were also presented in table3.

Table 2. Different properties of terry towels treated with different  $\beta$  –cyclodextrin concentrations

Cycldextrin concentrations	Nitrogen content, %	Wetting times, sec	Breaking strength, Newton	Breaking extension, %
10	17.1	4	540	25
20	17.5	3	538	24
30	18.9	2	535	23.8
40	19.1	2	535	23.8

Table 3. Percent loss in weight gained and fragrance at different storing times.

Storing times, days	Percent loss in weight gained, %	Percent loss in fragrance, %
7	5	7.5
21	7	9.6
42	17	18
90	40	55

## 2.5 Statistical Analysis

Experimental results were assessed using SPSS-Statistical Package program. In order to determine how the change of  $\beta$ -cyclodextrin concentration affects the results and whether this change was significant or not, One-Way ANOVA (Analysis of Variance) and correlation coefficients were made. In order to deduce whether the parameters were significant, p values were examined. As known, if the 'p' value of a parameter is greater than 0.05 (p>0.05), the parameter will not be significant and should be ignored.

In order to derive the regression relationship between  $\beta$ -cyclodextrin concentration and the properties of the terry towel sample, a linear or non-linear regression analysis was conducted. The regression relationship has the following form:

 $Y = a + b X + c X^2$ 

Where, Y is the terry towel properties; X is the  $\beta$ cyclodextrin concentration; a, b, and c are the regression coefficients. In the case of linear regression models, the value of the regression coefficient c will equal to zero.

## 3. Results and Discussion

# 3.1. Effect of $\beta$ -cyclodextrin concentration on Nitrogen content

The results of Nitrogen content values versus the concentration of  $\beta$ -cyclodextrin were plotted in figure 3. From this figure and the results of the Analysis of variance shown in table 4, it can be noticed that  $\beta$ -cyclodextrin concentration has a profound impact on the Nitrogen content. An increasing trend was detected conforming that the nitrogen content increases concomitantly to the increase in cyclodextrin concentration. The statistical analysis also proved that increasing the  $\beta$  -cyclodextrin concentration from 10% to 40% leads to an increase of the nitrogen content from 17 % to 19%. The increase in nitrogen content can be easily explained by the fact that the terry towels are grafted onto their fibers by the esterification reaction described by Welsh [15].

The regression analysis proved that the relationship which correlates the nitrogen content to the  $\beta$ -cyclodextrin concentration is a linear of the following form:

Nitrogen content, % = 0.074 X + 16.3

The correlation coefficient between the dependent variable an\d the independent one was found to equal 0.96 which signifies that the relation between two variables is very strong and positive.



Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	11.772	3	3.924	5.848	0.011	3.490
Within Groups	8.052	12	0.671			
Total	19.824	15				

Table 4: Analysis of variance results for the effect of cyclodextrin concentration on Nitrogen content

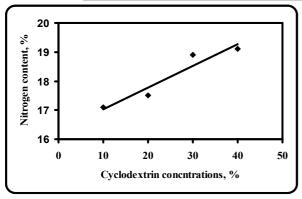


Figure 3: Effect of cyclodextrin concentration on Nitrogen content

# **3.2.** Effect of β-cyclodextrin concentration on wettability of terry towels

The effect of  $\beta$ -cyclodextrin concentration on Table 5: Analysis of variance results for the effect of cyclodextrin concentration on wetting time.

wetting time of the towels samples was illustrated in figure 4. The statistical analysis presented in table 5 shows that the concentration of cyclodextrin has a significant influence on the wettability of the terry towels at 0.01 significance level. From this figure a decreasing trend can be detected assuring that as the  $\beta$ -cyclodextrin concentration increases the wetting time of the terry towels decreases. This means that the higher  $\beta$ -cyclodextrin concentration is the higher is the towels wettability. The statistical analysis proved that increasing cyclodextrin concentration from 10% to 40% leads to an increase of towels wettability by 50%.

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	11	3	3.667	64.706	0.000	3.490
Within Groups	0.68	12	0.057			
Total	11.68	15				2 2

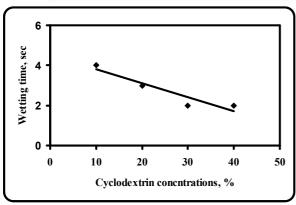


Figure 4: Effect of cyclodextrin concentration on wetting time of terry towels

The regression relationship between  $\beta$ -cyclodextrin concentration and the wetting time of the terry towels was found to have the following linear form:

Wetting time, sec = 0.074 X + 16.3

The correlation coefficient between the cyclodextrin concentration and the wetting time was - 0.99 which signifies that the relation between the two variables is very strong and negative.

3.3. Effect of Reactive β-cyclodextrin

concentration on breaking strength of terry towels

Breaking strength has been accepted as one of the most important attributes of woven fabrics. It is the main characteristic that distinguishes it from non-woven and knitted fabric. The strength of a woven fabric depends not only on the strength of constituent yarns, but also on the yarn and fabric structure and many other factors. In this study, the breaking strength of the terry towels was measured in weft direction. The effect of cyclodextrin concentration on fabric tensile strength was presented in figure 5.

The statistical results shown in table 6 showed that cyclodextrin concentration has no significant influence on the towels breaking strength. From figure 5 it can be seen that as the cyclodextrin concentration increases the towels breaking strength slightly decreases. Generally we can state that  $\beta$ -cyclodextrin concentration did not have marked effect on terry towels breaking strength. This result is in line with the findings of wang et al [16] who reported that  $\beta$ -cyclodextrin does not have a marked effect on its physical properties of the substrates and is ideal for preparing inclusion



compounds for microencapsulation of essential oils.

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The statistical analysis also proved that increasing  $\beta$ -cyclodextrin concentration leads to diminishing the breaking strength of the terry towels under study ab about 1% approximately. The slight decrement in breaking strength of perfumed terry towels is probably due to the action of alkali on cellulose at higher temperature during its functionalization with  $\beta$ -cyclodextrin.

The regression relationship which correlates the cyclodextrin concentration to the breaking strength of terry towel samples is of the following linear form:

Breaking strength, Newton = 0.18 X + 541.5

The correlation coefficient between the cyclodextrin concentration and the breaking strength is about -0.85 which signifies that the relation between the two variables is very weak and negative.

Table 6: Analysis of variance re	sults for the effect of cyclodextrin conce	entration on breaking strength.
		0

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	72	3	24.000	0.673	0.585	3.490
Within Groups	428	12	35.667		5	
Total	500	15				

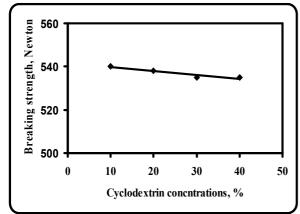


Figure 5: Effect of cyclodextrin concentration on breaking strength of terry towels

# **3.4.** Effect of Reactive β-cyclodextrin concentration on breaking extension of terry towels

Equally important to the fabric strength is its ability to extend under load. When the fabric is subjected to tension in one direction, the extension takes place in two main phases. The first phase is decrimping or crimp removal in the direction of the load. The removal of the crimp is accompanied by a slow rate of increase of the load. The second phase is the extension of the yarn during which the fabric becomes stiffer; the stiffness depends mainly on the character of the yarn. The more is the crimp in the yarn, the more extensible is the fabric. The values of breaking extension of terry towels against the concentration of  $\beta$ -cyclodextrin were depicted in figure 6. The results of ANOVA shown in table 7 showed that the cyclodextrin concentration has no significant impact on the terry towels breaking extension. As shown from figure 6 a decreasing trend was detected assuring that increasing the cyclodextrin concentration decreased the breaking extension of towel samples slightly. The insignificant influence of cyclodextrin concentration on the breaking extension of terry towels agrees with the findings of wang et al [16].

The statistical analysis proved that increasing the  $\beta$ -cyclodextrin concentration from 10% to 40% leads to a reduction of towels breaking extension by approximately 4%. This slight decrease in breaking extension may be ascribed to the action of alkali on cellulose at higher temperature during its functionalization with  $\beta$ -cyclodextrin.

The regression relationship between breaking extension of terry towels and the concentration of  $\beta$ -cyclodextrin is linear of the following form:

Breaking extension, % = -0.038 X + 25

The correlation coefficient between the two variables equals -0.82 which is signifies that the relationship is very negative and weak.

Table 7: Analysis of variance results for the effect of cyclodextrin concentration on breaking extension of

terry towels.

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	3.96	3	1.320	0.152	0.926	3.490
Within Groups	104	12	8.667			
Total	107.96	15				

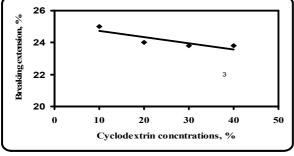


Figure 6: Effect of cyclodextrin concentration on breaking extension of terry towels

# 3.5. Quality assessment of the experimental results

In order to evaluate the overall effect of terry towel properties, the radar chart statistical method was used. For each level cyclodextrin concentration, the area of the radar chart was evaluated and then the final quality assessment of the results was conduced. Figure 7 represents the quality area of the terry towel properties under study at the different levels of cyclodextrin concentrations. The quality index according to cyclodextrin concentrations were listed in table8.

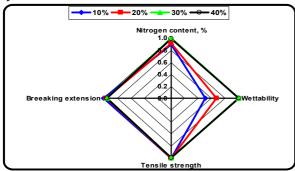


Figure 7. Radar charts for the experimental results at different cyclodextrin concentrations. Table 8. Quality index for the experimental results

at different cyclodextrin concentrations.

Cyclodextrin concentrations, %	Quality index
10	1.42
20	1.55
30	1.94
40	1.93

From this figure and results in table 7, it can be Table 9: Analysis of variance results for the seen that the terry towel sample treated with cyclodextrin of concentration 30% has the high quality area followed by the samples treated with cyclodextrin of concentrations, 40% and 20 and 10% respectively.

# **3.7.** Effect of curring temperature on Nitrogen content

The samples treated with cyclodextrin of concentration 30% cured at different temperatures for 2 minutes. The values of Nitrogen content versus the temperature of curring process were depicted in figure 8, and the results of the analysis of variance were presented in table 9. From this table it is evident that the curring temperature has a significant impact on the Nitrogen content at 0.01 significant level. From figure 9 an increasing trend was detected confirming that as the curing temperature increases, the Nitrogen content has the same trend. The statistical analysis proved that increasing the curing temperature from 120 °C to 140 °C lead to an augmented of the Nitrogen content by about 20%.

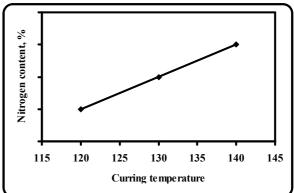


Figure 8: Effect of curring temperature on Nitrogen content

The regression relationship which correlates the curring time to Nitrogen content has the following linear form:

Nitrogen content, % = 0.2 X - 8

The coefficient of determination for this model was found to have one, which means that this model fits the data very well.

Table 9: Analysis of variance results for the effect of curring temperature on Nitrogen co	ontent.
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Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	24.667	2	12.333	5.286	0.030	4.256
Within Groups	21	9	2.333	24	÷ 75	
Total	45.667	11				

# **3.6.** Effect of washing cycles on percent loss in weight gained and fragrance

Percent loss in weight gained and fragrance against the number of washing cycles was plotted in Figure 9. The analysis of variance (ANOVA) results for the effects of washing cycles on the percent loss of weight gained and fragrances were listed in tables 10 and 11 respectively. From this figure and tables it is appeared that washing cycles have a significant influence on the percent loss of weight gained and fragrance. An increasing trend was detected assuring that as the number of washing cycles increases the percent loss also increases. Increasing the number of washing cycles from 5 to 20 cycles leads to an increase of percent loss of weight gained from 2% to 14%; while the percent loss of fragrance was increased from 3 % to 18%.

The regression relationship which correlates the number of washing cycles to both the percent loss of weight gained and fragrance have the following linear forms:

Percent loss in weight gained, % = 0.98 X - 2

Percent loss in fragrance, % = 0.8 X - 2.5where X is number of washing cycles.

The  $R^2$  values were found to have 0.99 and 0.98 for percent loss in weight gained and fragrance

respectively.

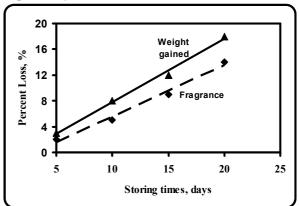


Figure 9: Effect of number of washing cycles on percent loss of weight gained and fragrance.

Table 10: Analysis of variance results for the effect of washing cycles on the percent loss of weight gained.

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	329.546	3	109.849	46.765	0.000	3.4903
Within Groups	28.1875	12	2.349			
Total	357.734	15	Se 2			

Table 11: Analysis of variance results for the effect of washing cycles on the percent loss of fragrance

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	483	3	161	96.6	0.000	3.4903
Within Groups	20	12	1.667			5
Total	503	15				

Table 12: Analysis of variance results for the effect of storing times on the percent loss of weight gained.

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	3252	3	1084	215.008	0.000	3.490
Within Groups	60.5	12	5.042	1		
Total	3312.5	15		_		

Table 13: Analysis of	variance results for	the effect of storing times	on the percent	loss of fragrance

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	5972.51	3	1990.83	393.446	0.000	3.490
Within Groups	60.72	12	5.06			1
Total	6033.23	15				

# **3.5.** Effect of storing times on percent loss in weight gained and fragrance

According to the quality assessment of the experimental results shown in the above section, it is apparent that the cyclodextrin of concentration 30% yield the best results with respect to the overall quality. After perfume finishing, the woven terry towel samples which were treated using  $\beta$ -cyclodextrin of concentration 30% at curring temperature 140 °C for 2 minutes were

stored for different durations, i.e. 7, 21, 42 and 90 days respectively. After each storing time the percent loss in the weight gained and the percent loss in fragrance were evaluated and the final results were plotted against the storing times as shown in figure 9. The results of Analysis of Variance for the effects of storing times on the percent loss of weight gained and fragrance were listed in tables 12 and 13 respectively.

The statistical analysis proved that the storing



times were found to have a huge influence on the percent loss in the weight gained and fragrance at significance level 0.01. From this figure, increasing trends were detected assuring that as the storing times increase the loss in both weight gained and fragrance swiftly increases. Increasing the storing times from 7 to 90 days increased the percent loss in weight gained from 5% to 40% and increased the percent loss in fragrance from 7% to 55%.

The regression analysis revealed that the relation between storing time and both the percent loss in gained weight and fragrance has the following non-linear models:

Percent loss in weight gained,  $\% = 0.007 \text{ X}^2 - 0.09 \text{ X} + 7.9$ 

Percent loss in fragrance,  $\% = 0.005 \text{ X}^2 - 0.13 \text{ X}$ +6

Where, X is the storing time with days. The statistical analysis proved that the correlation coefficients were 0.99 for both models, which means that the relation between storing time and percent loss in weight gained and fragrance is strong and positive.

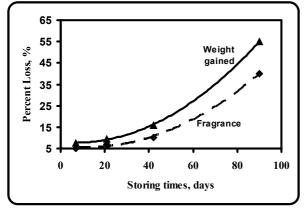


Figure 9: Effect of storing time on percent loss of weight gained and fragrance.

### Conclusion

objective this the The of study was microencapsulation of fragrances and perfumes to be applied in textile industry, in particular in the terry towels. The of production effect concentration of Reactive  $\beta$ -cyclodextrin on nitrogen content, terry towels wettability and their tensile properties were accomplished. Also the effect of storing time on the percent loss of the weight gained and percent loss of fragrance were also examined. The findings of this work revealed that the increased Reactive  $\beta$ -cyclodextrin leads to an increase of the nitrogen content and enhanced the towels wettability. It is also noticed that the towels breaking strength and extension slightly reduced with the cyclodextrin concentration. Storing times were found to have a huge impact on the percent loss in in weight gained and fragrance.

The overall quality of the experimental results revealed that the best quality for the terry towels associated with Reactive  $\beta$ -cyclodextrin of 30% concentration followed by samples treated with cyclodextrin of 40% and 20% and 10% concentrations respectively. The obtained results showed that the best curring temperature was 140 °C. Increasing the number of washing cycles from 5 to 20 cycles leads to an increase of the percent loss of fragrance from 3 % to 18%.

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