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Synthesis of Fragranced Dyes and Their application to Cotton Textiles

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ABSTRACT

The aim of the study was to synthesize fragranced dyes and application of these dyes to cotton fabric. β-cyclodextrin was grafted to heterobifunctional reactive dyes and the cavity of the cyclodextrine was utilized for loading various fragrances in it. The modified dyes were characterized by FTIR and UV/VIS spectroscopy. The fragranced dyes were used for dyeing of cotton fabrics. The treated samples were subjected to number of washing cycles as well as exposure to sunlight for prolonged time to study the fragrance retention on the fabric. The extent of attachment of the modified dye and colour strength developed on cotton fabric were determined. Olfactometrical analysis of the dyed fabric was conducted to have an idea of perfume retention onto the fabric.

Keywords: fragranced dye, modified reactive dye, olfactometry, perfumed fabric

1. Introduction

Textiles with advanced technology could be much more than the conventional textiles, with new wrinkle free and stain resistant function, to give pleasant aroma, antibacterial properties and many more. Such textiles are called smart textiles *(Azoulay, 2005)*. In Germany five million people suffer from neurodermitis, two million from psoriasis, and further three million from dermal diseases of occupational origin (like hairdresser's allergic reactions on the hands, athlete's foot, etc.). Numerous studies, conducted around the globe, indicate that odours have an impressive and measurable effect on people's behaviour and mood state *(Cline, 2008).* Perfumes can evoke emotional reactions *(Wrzesniewski et al, 1999).* Insertion of Fragrance to the textiles is the process where we enhance the value of the product by adding some incentives to it. *(Bolenwar et al, 2011).* The use of herbs in medicine spread from ancient Indian civilization to other parts of the world. This ancient system of herbal medicinal therapy with modern scientific understanding and experimentation forms the basis of "Aromatherapy". Aromatherapy as the name suggests is a holistic therapy using fragrant essential oils to treat body and mind *(Dixit, 2004).* Insertion of fragrance to textiles is one such immaculate magnanimous entry into any textile culture. Fragranced textiles can have their medicinal values depending on the fragrances used. Such as Jasmine oil has therapeutic properties like anti-depressant, antiseptic, sedative and uterine. Likewise Citronella oil has the properties that it relieves painful muscles and joints, helps in nervous fatigue, etc.

To prepare aroma therapeutic textiles, β-cyclodextrin was the first choice as the host molecule, because β-cyclodextrin molecules are capable of forming inclusion compounds with organic compounds that fit into their own cone-shaped hydrophobic cavities. *(Bolenwar et al, 2011, Singh et al, 2010 and Wang & Chen, 2005).* Cyclodextrins (α-, β- and γ-CD) are formed during the enzymatic degradation of starch. These are non-reducing, crystalline, water soluble, cyclic, oligosaccharides. β -Cyclodextrin is the most accessible, the lowest-priced and generally the most useful. *(Martindel valle, 2004).* Wang and Chen (2004) reported the dyeing of cotton with reactive dye from a bath containing both bifunctional reactive dye and β-cyclodextrine. The samples were then subjected to perfume spraying.

The present work was aimed at attaching β-cyclodextrine to heterobifunctional reactive dyes and loading the modified dyes with different fragrance extracts. The synthesized fragranced dyes were then used for the dyeing of cotton to incorporate fragrance into the textiles along with dyeing. The modified as well as the unmodified dyes were characterized by analyzing their spectra obtained using UV / VIS Spectrophotometer and FTIR spectroscopy. The treated fabric samples were subjected to the measurement of graft %, colour strength, tensile properties and olfactometrical analysis.

2. Materials

Plain RFD (Ready for Dyeing) cotton fabric was kindly provided by the Arvind Mills Ltd., a composite textile unit, Ahmedabad, India. β-cyclodextrin was gifted by the National Chemicals Ltd., Vadodara, India. Dimethyl formamide, Isobutyl alcohol, sodium chloride, sodium carbonate, and Sodium hydroxide were purchased from Sulab Chemicals, Vadodara, India. Essential oils and perfumes such as citronella oil, jasmine oil, Lonkom and Gambit perfumes were purchased from various perfumery industries.

3. Apparatus & equipments

- UV – 2450 spectrometer, Shimadzu scientific instruments, North America
- Nicolet iS10FT-IR spectrometer, Thermo scientific Ltd., U.S.A.
- Spectrascan 1500A, Premier colorscan Pvt. Ltd., India .
- Lloyd Tensile Tester, U.K.
- High Temperature High Pressure Beaker dyeing machine, R. B. Electronics Pvt. Ltd, India.
- Launderometer with microprocessor, R. B. Electronics Pvt. Ltd**,** India

4. Experimental methods

4.1 Modification of heterobifunctional reactive dyes

The procedure consisted of the addition of alkaline β-cyclodextrin solution into the dye solution at $4-5^{0}$C. The mixture kept in a closed container was slowly agitated for 20 minutes on a shaker. The modified dyes were then solvent extracted and filtered. The resulted modified dyes in the form of lumps were then dried in an oven till complete drying.

4.2 Encapsulating fragrances into the modified dyes

The modified dyes were weighed according to the shade requirements. The fragrances were then encapsulated into the dye (by spraying technique) in various amounts like 10%, 20% and 60% (on weight of the dye). The fragrance encapsulated dyes were then kept in open air to ensure evaporation of the solvent present in the perfumes. Thus perfume encapsulated dyes (hereafter known as fragranced dyes) were then further used for dyeing cotton fabric samples.

4.3 Application of fragranced dyes to cotton Fabric

The cotton fabric was dyed using 1 % and 3% modified dyes (with and without fragrance loading) at 60°C using 100 g/l sodium chloride as exhausting agent and 30 g/l sodium carbonate as dye fixing agent. The total time for dyeing was 60 minutes. The dyed samples were then rinsed, washed and soaped followed by complete drying. The cotton fabric dyed with unmodified dyes (hereafter known as control dyes) in the same manner to compare the results. After the reaction, the treated fabric was thoroughly washed and dried to constant weight. All the samples dyed with modified and control dyes were then analyzed for colour strength as well as tensile strength.

4.4 Washing of the treated textiles

The perfumes are volatile in nature. Thus, the intensity of fragrances gets reduced day by day due to wear and wash. All the dyed samples were, therefore, examined for fragrance retention after number of washings for the evaluation of fragrance retention on clothing usage in day to day routine life. The washings were done according to the ISO 105-C10, 2006 standards using 5 g/l soap solution and 2 g/l soda ash solution at 60ºC for 30 minutes. This procedure was repeated 5 times to examine the fragrance retention after the washing procedure. After each washing the samples were sent for olfactory analysis to panelists for fragrance intensity ratings.

4.5 Characterization of modified dyes

The prepared coloured reactive entities i.e. modified heterobifunctional dyes were characterized using UV / VIS Spectrophotometer (UV – 2450 spectrometer, Shimadzu Scientific Instruments, North America) and FTIR spectroscopy (Nicolet IS10FT-IR Spectrometer, Thermo scientific, USA) for examining the attachment of β-cyclodextrin to the dyes qualitatively. The spectra of the modified dyes were compared against the control dyes.

4.6 Characterization of treated textiles

Determination of graft % on the textiles: After the dyeing, the treated fabric was thoroughly washed and dried to constant weight. The graft per cent was expressed in terms of % gain in weight of the fabric after dyeing compared to the undyed one. For each sample five readings were recorded and an average of these five readings was expressed as graft%.

Determination of colour strength: The colour strength in terms of K/S values was measured on 1500 Premium colorscan spectrophotometer (Thermo scientific, USA).

Determination of tensile strength: The tensile strength of all the samples was measured on Lloyd Tensile Tester, U.K.

Analysis by Olfactometry: It is one kind of sensory analysis to establish the odour concentration which employs a group of panelists. The odour concentration or the intensity was divided into the following categories according to the intensity:

0 → No odor 1 → Very weak 2→ Weak
3 → Medium strong 4→ Strong
5 → Very strong

This method was applied in the laboratory and was done by a series of suitably trained panelists observers.

5. Results & discussion

5.1 Characterization of modified dyes

5.1.1 Properties of modified dyes

The modified dyes had slight variation in colour than that of the control dyes and they were found to be completely soluble in water. The other properties of the modified dyes viz. dyeing strength and graft %, tensile properties on cotton fabric were also characterized as discussed in the part 5.2.

5.1.2 FTIR spectroscopy

Figure 1 & 2 shows the FTIR spectra of control and modified Corafix Red GD3R dye respectively. The medium peaks at 1290 cm^{-1}, 1593 cm^{-1} and 792 cm^{-1} & 747 cm^{-1} indicating C-N, C=N and C-Cl stretching were observed in the control dye. All these peaks are also observed in the FTIR spectra of Cyanuric chloride. Thus, it shows the presence of chlorotriazine group in the heterobifunctional dye. Amongst these peaks, first two peaks were intact while third peak got diminished after modifications of the dyes. Such reduction in peak intensity signifies the replacement of chlorine atom of the chlorotriazine with β-Cyclodextrin. Further, the peak at 2921 cm^{-1} was due to the C-H and O-H stretching of β-Cyclodextrin. The absorption bands at 1290 and 1180 cm^{-1} appeared from the asymmetric and symmetric O=S=O stretching respectively of the sulphone group. Figure 3 to 6 show the FTIR spectra of control and modified Corafix Yellow GDR dye and Corafix Yellow F3R dye respectively. All the control dyes also showed same peaks as that of Corafix Red GD3R. Dyes are showing the presence of chlorotriazine and vinyl sulfone groups with other peaks as per their chemical structure. The identical peaks for vinyl sulfone as well as the diminished chlorine peak were also found in all the modified dyes.

5.1.3 UV/VIS spectroscopy

Figure 7, 9 & 11 depict UV/VIS spectra of Control Corafix Red GD3R, Corafix Yellow GDR and Corafix Yellow F3R dyes whereas Figure 8, 10 & 12 depict the UV/VIS spectra of the dyes after modification. The Red dye (Figure 7 & 8) showed shifts of peaks from 238 nm to 234.5 nm, 505.5 nm to 509 nm after modification. Quite high change in shifts from 422 nm to 406.5 nm, 337 nm to 332 nm, 333 nm to 336.5 nm were observed in case of Yellow GDR dye (Figure 9 & 10). From 198 nm to 204.5 nm, 264 nm to 256 nm shifts were found in Yellow F3R dye (Figure 11 & 12). These shifts denote the modification of the dye.

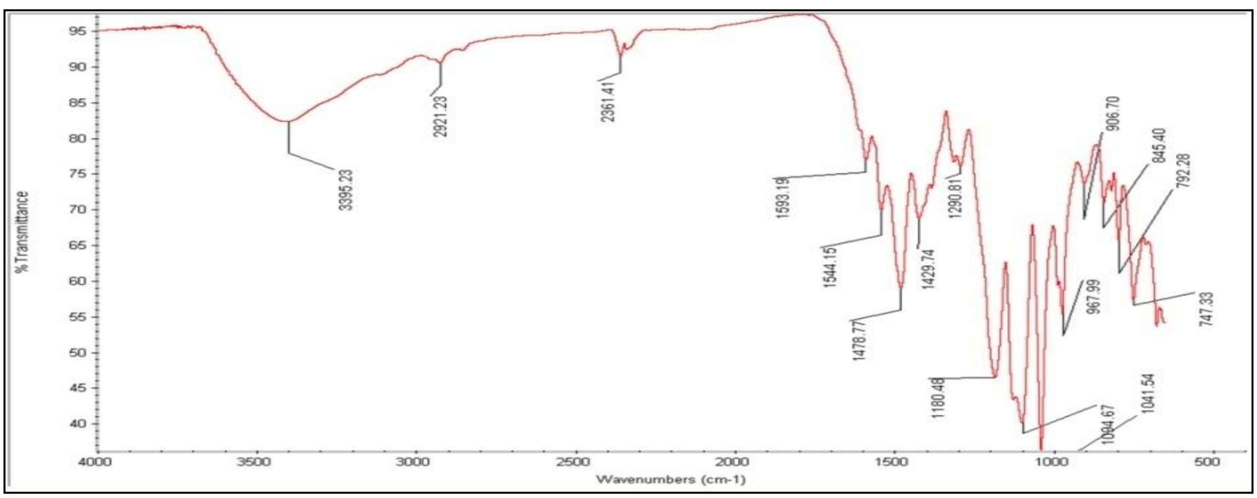

Figure 1. FTIR spectra of Corafix red GD3R dye

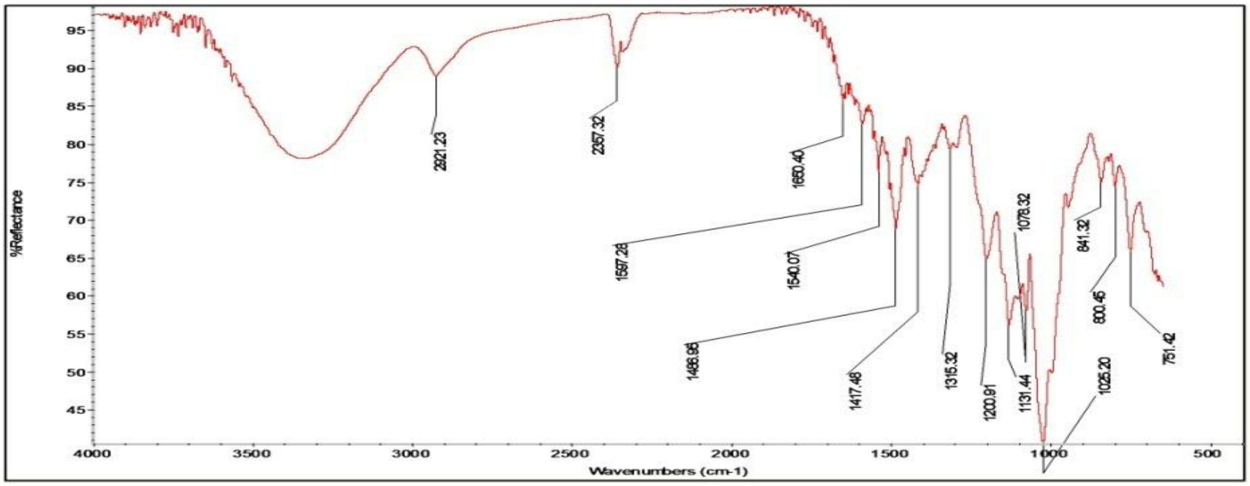

Figure 2. FTIR spectra of modified Corafix red GD3R dye

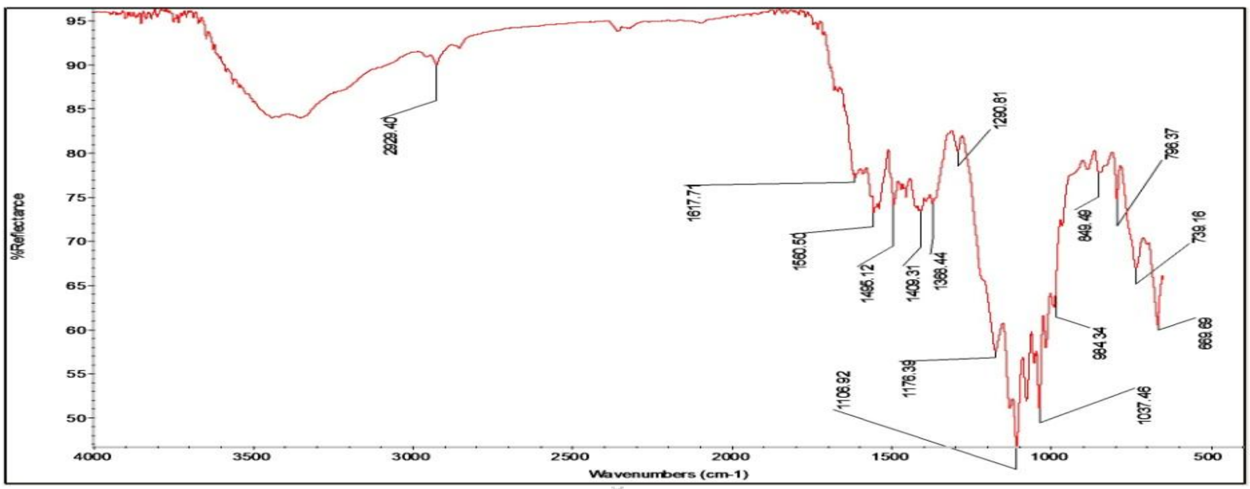

Figure 3. FTIR spectra of Corafix yellow GDR dye

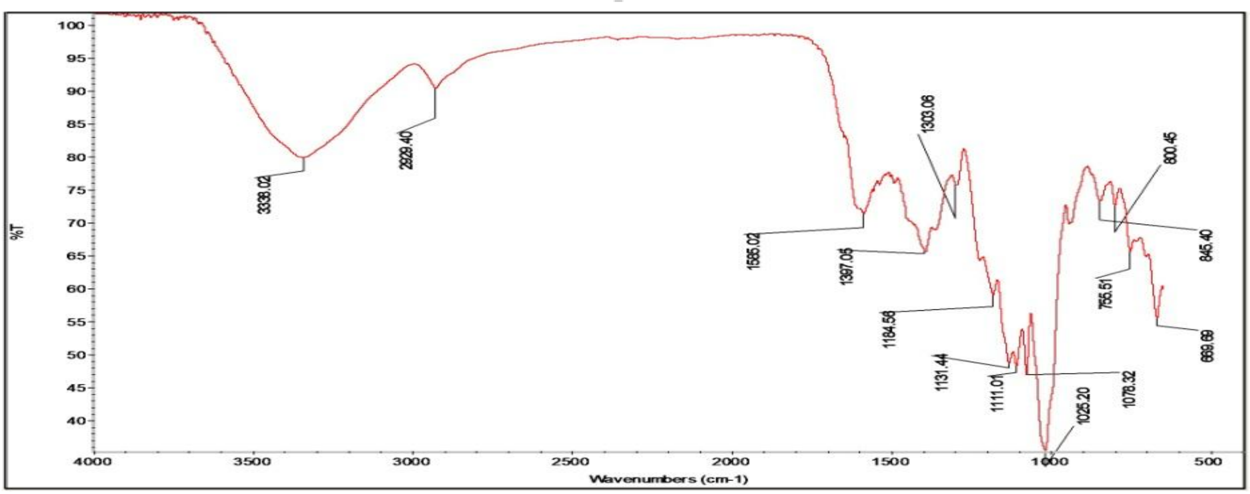

Figure 4. FTIR spectra of modified Corafix yellow GDR dye

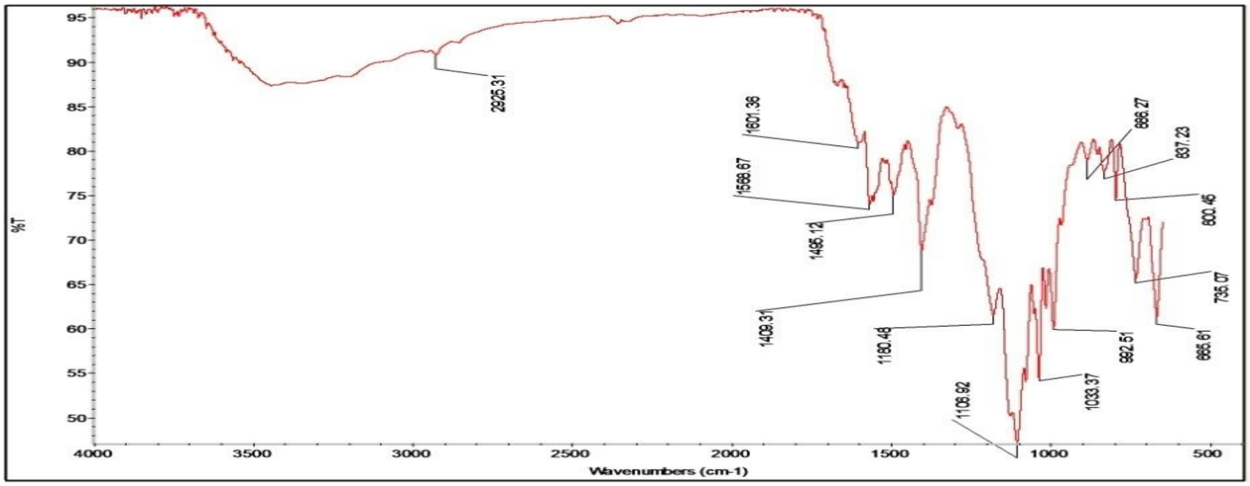

Figure 5. FTIR spectra of Corafix yellow F3R dye

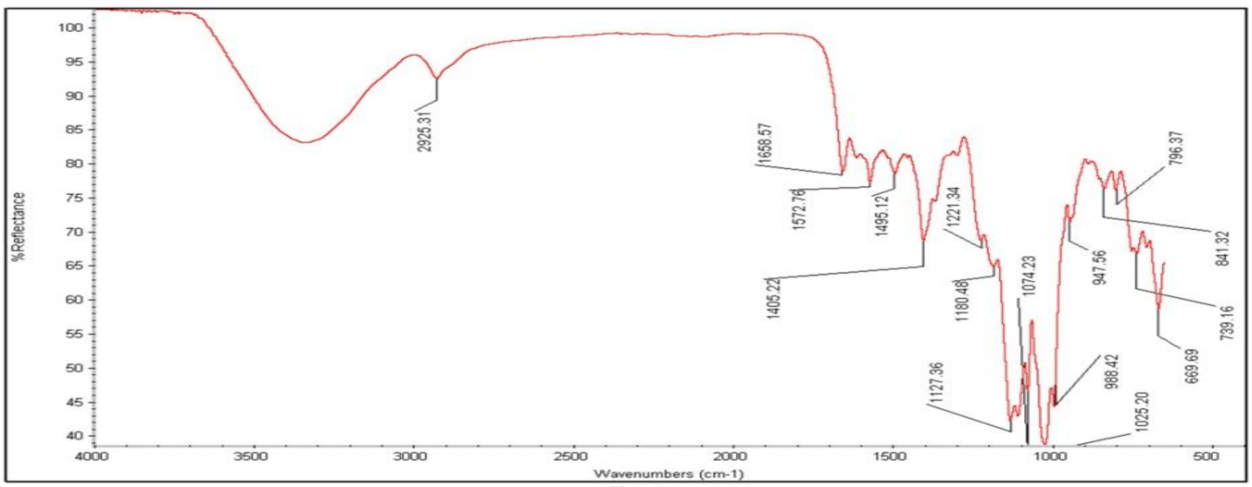

Figure 6. FTIR spectra of modified Corafix yellow F3R dye

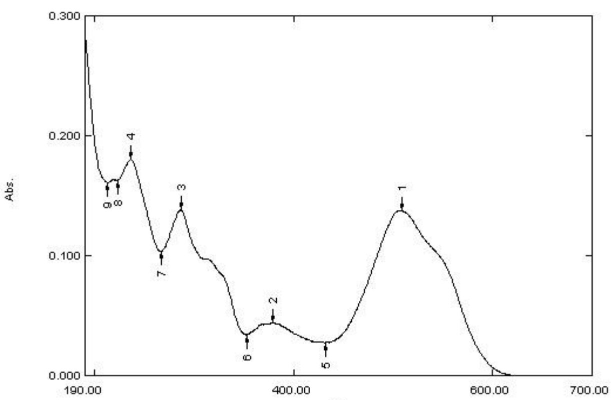

Figure 7. UV/VIS spectra of Corafix red GD3R dye

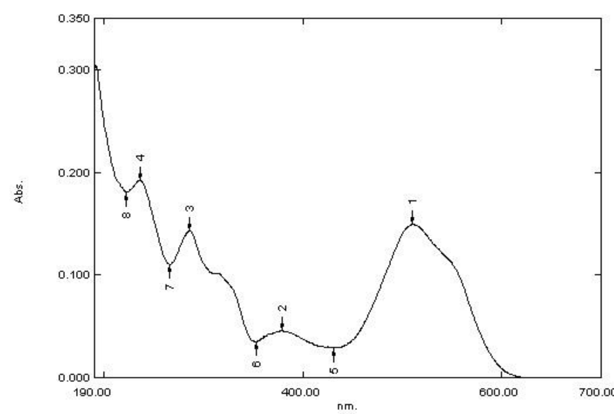

Figure 8. UV/VIS spectra of modified Corafix red GD3R dye

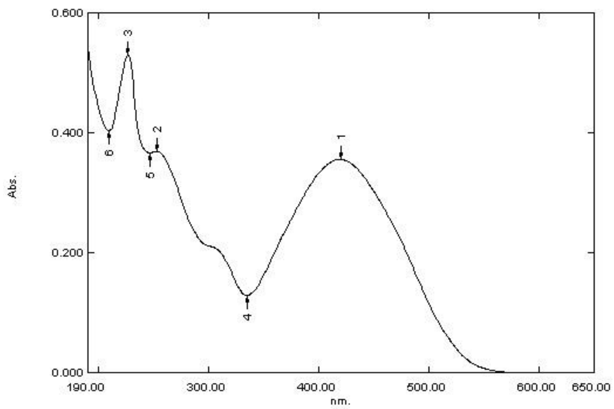

Figure 9. UV/VIS spectra of Corafix yellow GDR dye

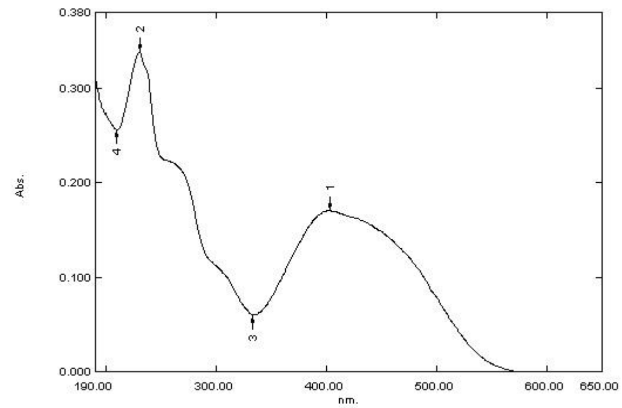

Figure 10. UV/VIS spectra of modified Corafix yellow GDR dye

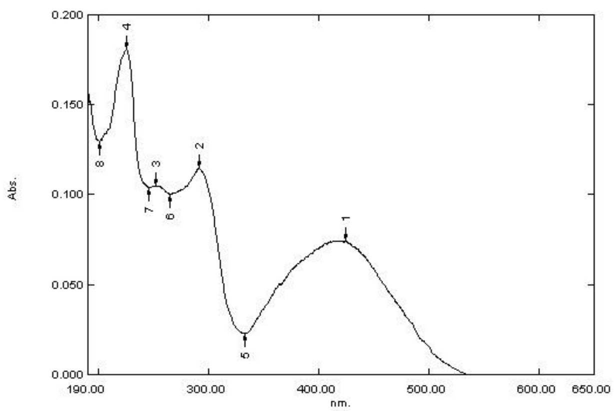

Figure 11. UV/VIS spectra of Corafix yellow F3R dye

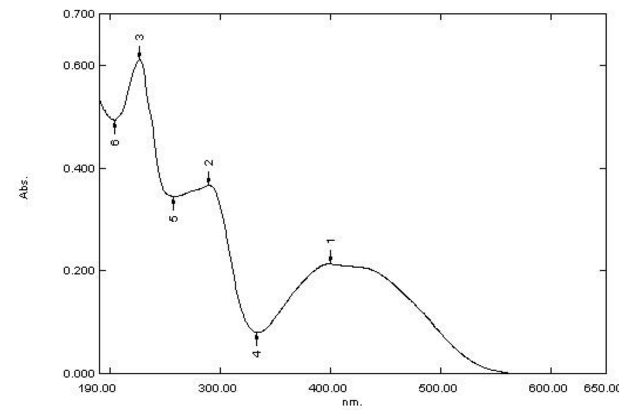

Figure 12. UV/VIS spectra of modified Corafix yellow F3R dye

5.1.4 Effect of β-cyclodextrin grafting on colour value of dyes

Table I represents the effect of β-cyclodextrin grafting on colour value (in terms of absorbance at λ_{max}.) of the dyes used. Colour values of the dyes got reduced due to β-cyclodextrin grafting with the actual dye moiety. The reduction was almost same for both the yellow dyes *i.e.* approximate 17%. The reduction in case of Red dye was less compared with the yellow dyes.

5.2 Characterization of treated textiles

5.2.1 Effect of β-cyclodextrin grafting on graft % of the fabric dyed with different heterobifunctional reactive dyes used

Table II represents the effect of grafting β-cyclodextrin to the heterobifunctional reactive dyes viz., Corafix Red GD3R, Corafix Yellow GDR and Corafix Yellow F3R on their graft %. There was a rise in graft % (per cent gain in weight) of the cotton fabric after modification of the dyes irrespective of the type and shade of the dyes studied, which is due to higher molecular weight of β-cyclodextrin (1138 gm/mole). There was a marginal increase in case of Yellow GDR i.e. 16% for 1 % shade and 32% for 3 % shade compared to that of the other dyes studied. On the contrary, red dye showed little increase in the graft %.

Table I. Effect of β-cyclodextrin grafting on colour value of dyes

Name of dye	Colour value (in terms of absorbance at λ_{max})	
	Control dye	Modified dye
Corafix Red GD3R	0.632	0.581 (8.07)
Corafix Yellow GDR	0.738	0.610 (17.34)
Corafix Yellow F3R	0.523	0.434 (17.02)

Note: Control dyes were the dyes used without modification. Values in the parenthesis show % Loss in Colour Value of Modified Dyes compared to that of the control dyes.

5.2.2 Effect of β-cyclodextrin grafting on colour strength of modified dyes

Table III represents the effect of modification *i.e.* effect of grafting β-cyclodextrin to heterobifunctional reactive dyes viz., Corafix Red GD3R, Corafix Yellow GDR and Corafix Yellow F3R on their Colour strength value measured in terms of K/S. The table III reveals that there was a loss in colour strength measured in terms of K/S values of all the dyes. The loss was not in a particular order. But average 25% loss was observed in case of all the dyes. The loss was highest in case of Yellow GDR dye. The loss in colour strength may be attributed to the loss in colour value of the individual dye on account of β-cyclodextrin grafting. The extent of loss was found to be dependent both on the structure and the amount of the dye used. The Red and Yellow F3R dyes showed less reduction compared to Yellow GDR dye.

Table II. Effect of β-cyclodextrin grafting on graft % of the fabric dyed with different heterobifunctional reactive dyes used

Name of dye	Shade (%)	Graft %	
		Control dye	Modified dye
Corafix Red GD3R	1%	2.22	2.48
	3%	2.29	2.81
Corafix Yellow GDR	1%	1.79	2.13
	3%	2.05	3.02
Corafix Yellow F3R	1%	2.06	2.43
	3%	2.15	3.14

Note: Control dyes were the dyes without any modification.

5.2.3 Effect of β-cyclodextrin grafting on tensile properties of fabric treated with modified dyes

Table IV represents the effect of grafting of dyes on the tensile properties of the fabric after dyeing with modified dyes (without encapsulating perfume). The β-Cyclodextrin works as a plasticizer. As the table describes the tensile properties of the fabrics treated with control and modified heterobifunctional dyes viz., Corafix Red GD3R, Corafix Yellow GDR and Corafix Yellow F3R, it can be seen that there was an increase in % elongation of all the dyes irrespective of the type and amount of the dye used but tensile strength decreased. The gain in elongation was higher for 1% dye shade than that of the 3% dye shade. The loss in tensile strength was due to uneven distribution in segmental mobility of the chains. The gain in elongation was highest for Yellow GDR dye for both 1% as well as for 3% whereas least for Red dye for both the shades of the dye.

Table III. Effect of β-cyclodextrin grafting on colour strength of modified dyes

Name of dye	Shade (%)	Colour strength value (in terms of K/S)	
		Control dye	Modified dye
Corafix Red GD3R	1%	4.8	3.9 (17.19)
	3%	8.0	6.8 (14.73)
Corafix Yellow GDR	1%	8.4	5.1 (38.48)
	3%	12.8	8.3 (35.57)
Corafix Yellow F3R	1%	7.2	4.7 (34.90)
	3%	13.2	9.1 (31.53)

Note: Control dyes were the dyes used without modification. Values in the parenthesis show % Loss in Colour Strength of Modified Dyes compared to the control dyes.

Table IV. Effect of β-cyclodextrin grafting on tensile properties of fabric

Name of dye	Shade (%)	Samples dyed with control dye		Samples dyed with modified dye		Loss in tensile strength of samples dyed with modified dye compared to the samples dyed with control dye (%)	Gain in elongation at break of samples dyed with modified dye compared to the samples dyed with control dye (%)
		Tensile strength (kgf)	Elongation at break (%)	Tensile strength (kgf)	Elongation at break (%)		
Corafix Red GD3R	1%	21.24	4.46	20.22	5.57	4.82	24.9
	3%	23.74	4.39	23.22	5.26	2.19	19.76
Corafix Yellow GDR	1%	22.62	4.36	19.34	5.91	14.48	35.57
	3%	24.40	4.36	21.84	5.66	10.49	29.79
Corafix Yellow F3R	1%	21.74	4.24	19.23	5.71	11.53	34.58
	3%	24.19	3.90	22.11	4.98	8.61	27.58

Note: Control dyes were the dyes used without modification.

5.2.4 Effect of perfumes and their concentrations on graft % of modified dyes

Table V represents the effect of perfumes and their concentrations on the graft % of the fabric treated with modified dyes (loaded with perfume) with reference to the fabric treated with modified dyes (without encapsulating perfumes). The solvents present in the perfumes affected the properties and the dyeing behavior of the dyes used. The table V reveals this characteristic of the solvents affecting graft % of the fabrics. The solvents had adverse effect on all the dyes decreasing graft % as could be seen from table V. The reduction was higher for 1% dye shade than that of the 3% irrespective of the dye and perfume used. The loss was in the range of 15% - 30%, 10% - 20% and 2% - 17% for 1% dye shade for Red, Yellow GDR and Yellow F3R dyes respectively, while in the range of 6% - 20%, 4% -19% and 0% -12% for 3% dye shade. Thus, it can be said that effect of solvents completely depended on their structures but unfortunately the structures of perfumes were not available.

Table V. Effect of perfumes and their concentrations on graft % of fabric dyed with perfume loaded modified dyes

Type of perfume	**Perfume concentration**	**Shade (%)**	**Graft (%)**			**Loss in graft compared to modified dye (%)**		
			Corafix Red GD3R	**Corafix Yellow GDR**	**Corafix Yellow F3R**	**Corafix Red GD3R**	**Corafix Yellow GDR**	**Corafix Yellow F3R**
Citronella Extract Oil	10%	1%	1.90	1.82	2.32	23.4	14.5	4.4
		3%	2.44	2.74	3.10	13.1	9.0	1.0
	20%	1%	1.84	1.77	2.29	25.8	16.5	5.4
		3%	2.37	2.71	3.07	15.6	10.1	2.2
	60%	1%	1.73	1.62	2.15	30.2	23.6	11.3
		3%	2.23	2.56	2.96	20.6	15.1	5.4
Jasmine Extract Oil	10%	1%	2.20	1.91	2.37	11.3	10.2	2.3
		3%	2.61	2.87	3.11	7.1	4.7	0.74
	20%	1%	2.15	1.86	2.35	13.3	12.5	3.2
		3%	2.58	2.84	3.10	8.2	5.7	1.2
	60%	1%	1.98	1.75	2.21	20.1	17.4	8.9
		3%	2.48	2.71	3.02	11.7	10.2	3.5
Lonkom Perfume	10%	1%	2.03	1.80	2.19	18.1	15.4	9.8
		3%	2.48	2.68	2.93	11.7	11.0	6.4
	20%	1%	1.99	1.75	2.15	19.7	17.5	11.3
		3%	2.43	2.60	2.90	13.5	13.8	7.5
	60%	1%	1.84	1.58	1.99	25.8	25.6	17.9
		3%	2.31	2.43	2.75	17.8	19.3	12.3
Gambit Perfume	10%	1%	2.08	1.89	2.26	16.1	11.2	6.8
		3%	2.58	2.85	3.03	8.2	5.6	3.3
	20%	1%	2.01	1.84	2.22	18.9	13.5	8.5
		3%	2.52	2.78	2.99	10.3	7.8	4.7
	60%	1%	1.89	1.70	2.06	23.8	20.0	14.9
		3%	2.38	2.62	2.83	15.3	13.2	9.7

Note: Values of graft % for samples dyed with (i) Modified Corafix Red GD3R Dye (without perfume encapsulation) were 2.48 and 2.81 for 1% and 3% dye shades respectively (ii) Modified Corafix Yellow GDR Dye (without perfume encapsulation) were 2.13 and 3.02 for 1% and 3% dye shade respectively (iii) Modified Corafix Yellow F3R Dye (without perfume encapsulation) were 2.43 and 3.14 for 1% and 3% dye shade respectively.

5.2.5 Effect of perfumes and their concentrations on colour strength of modified dyed fabric

Table VI represents the effect of perfumes and their concentration on the Colour strength of the modified heterobifunctional dyed fabric The tables clearly elucidate that perfumes affect the colour strength of all the dyes showing reduction in it. This is attributed to the characteristics and amount of the perfume used and as well on the characteristics and amount of dyes used. Different perfumes presented different effects on the colour strength of the dyes. In all cases, the colour strength reduced with increase in its concentration irrespective of the type of the dye and the perfume. In the case of all the dyes (Table VI), Jasmine oil affected their colour strength to the least extent, while citronella oil affected to the highest. The reduction was lesser in case of 3% dye shade than that of 1% dye shade for the same amount of the perfume loaded. All the perfumes behaved almost in a similar way to both Yellow GDR and Yellow F3R dyes. Thus it was concluded that the perfume has been loaded into the cavity of β-Cyclodextrin grafted to the dye.

Table VI. Effect of perfumes and their concentrations on colour strength of fabric dyed with modified dyes

Type of perfume	Perfume concentration	Shade (%)	Colour strength (in terms of K/S Value)			Loss in colour strength value compared to sample dyed with control dye (%)		
			Corafix Red GD3R	Corafix Yellow GDR	Corafix Yellow F3R	Corafix Red GD3R	Corafix Yellow GDR	Corafix Yellow F3R
Citronella Extract Oil	10%	1%	2.7	3.4	3.6	32.31	12.54	9.12
		3%	4.9	6.2	6.6	27.65	9.41	3.06
	20%	1%	2.6	3.4	3.5	34.69	14.68	10.23
		3%	4.9	6.0	6.5	28.45	12.21	4.35
	60%	1%	2.4	3.2	3.4	39.40	19.47	13.44
		3%	4.5	5.6	6.3	33.68	17.30	7.58
Jasmine Extract Oil	10%	1%	2.8	3.7	3.7	28.98	6.64	5.22
		3%	5.2	6.5	6.7	22.69	4.51	2.02
	20%	1%	2.7	3.6	3.7	30.02	8.55	6.87
		3%	5.1	6.4	6.5	24.52	5.84	3.65
	60%	1%	2.6	3.5	3.5	34.83	12.20	10.03
		3%	4.8	6.1	6.4	29.55	9.68	6.24
Lonkom Perfume	10%	1%	2.6	3.3	3.5	33.25	15.89	9.96
		3%	5.0	5.9	6.3	26.56	13.77	7.40
	20%	1%	2.5	3.3	3.5	34.98	17.20	11.32
		3%	4.8	5.7	6.2	28.84	15.48	8.57
	60%	1%	2.5	3.1	3.3	37.02	22.14	16.54
		3%	4.6	5.4	5.9	32.33	20.54	12.69
Gambit Perfume	10%	1%	2.8	3.6	3.6	29.37	9.56	8.87
		3%	5.2	6.3	6.5	23.84	7.88	4.30
	20%	1%	2.7	3.5	3.5	31.66	11.33	10.54
		3%	5.1	6.1	6.4	25.49	9.54	5.64
	60%	1%	2.5	3.3	3.3	35.23	15.56	14.87
		3%	4.7	5.9	6.1	30.50	13.09	9.95

Note: Values of Colour strength (in terms of K/S) for (i) Modified Corafix Red GD3R dye (without encapsulating perfume) were 3.98 and 6.84 for 1% and 3% dye shade respectively (ii) Modified Corafix Yellow GDR dye (without encapsulating perfume) were 5.19 and 8.30 for 1% and 3% dye shade respectively (iii) Modified Corafix Yellow F3R dye (without encapsulating perfume) were 4.73 and 9.19 for 1% and 3% dye shade respectively

5.2.6 Effect of perfumes and their concentrations on tensile properties of fabric

Tables VII to IX show the effect of perfumes and their concentrations on the tensile properties of the fabrics treated with modified dyes with reference to modified dyes without encapsulating perfume. The β-Cyclodextrin works as a plasticizer. As the table describes the tensile properties of the fabrics treated with perfume loaded modified dyes, it can be seen that there was gain in elongation (%) of all the dyes irrespective of the type and amount of the dye used but loss in the tensile strength in all cases. The loss was higher for 1% dyes shade than that of the 3% dye shade. The gain in elongation was highest for Yellow GDR dye for both 1% as well as for 3% and that the tensile strength. In case of the Red dye, Jasmine oil affected its tensile properties to the least extent. On the other hand, citronella oil affected it to highest. In case of Yellow GDR and Yellow F3R also, Jasmine oil affected to the least extent while Lonkom perfume affected to the highest.

Table VII. Effect of perfumes and their concentrations on tensile properties of fabric dyed with modified Corafix red GD3R dye

Type of perfume	Perfume concentration	Shade (%)	Tensile strength (kgf)	Loss in tensile strength (%)	Elongation at break (%)	Loss in elongation at break (%)
Citronella Extract Oil	10%	1%	17.32	14.32	6.10	7.1
		3%	20.54	11.54	5.63	9.54
	20%	1%	15.80	21.84	6.45	11.02
		3%	19.35	16.65	5.84	15.87
	60%	1%	14.83	26.65	6.81	17.56
		3%	18.47	20.45	6.18	22.32
Jasmine Extract Oil	10%	1%	18.95	6.26	6.06	4.2
		3%	22.09	4.87	5.48	8.86
	20%	1%	17.99	11.03	6.30	6.51
		3%	21.31	8.21	5.60	13.05
	60%	1%	16.30	19.35	6.60	10.31
		3%	19.86	14.45	5.80	18.46
Lonkom Perfume	10%	1%	18.08	10.56	6.13	6.88
		3%	21.77	6.25	5.62	10.09
	20%	1%	16.56	18.07	6.48	9.87
		3%	20.15	13.23	5.78	16.3
	60%	1%	15.32	24.21	6.94	14.48
		3%	18.48	20.41	6.02	24.54
Gambit Perfume	10%	1%	18.53	8.32	6.10	5.4
		3%	22.05	5.06	5.54	9.51
	20%	1%	17.10	15.40	6.41	8.41
		3%	20.55	11.52	5.70	15.09
	60%	1%	15.64	22.65	6.77	12.54
		3%	19.22	17.22	5.92	21.6

Note: Values of Tensile strength (in kgf) and elongation at break (%) for modified Corafix Red GD3R dye (without encapsulating perfume) were 20.22, 23.22 and 5.57%, 5.26% for 1% and 3% dye shade respectively.

Table VIII. Effect of perfumes and their concentrations on tensile properties of fabric dyed with modified Corafix yellow GDR dye

Type of perfume	Perfume concentration	Shade (%)	Tensile strength (kgf)	Loss in tensile strength (%)	Elongation at break (%)	Loss in elongation at break (%)
Citronella Extract Oil	10%	1%	18.23	5.74	6.29	3.97
		3%	21.22	2.86	5.88	6.45
	20%	1%	16.91	12.59	6.57	7.54
		3%	20.31	7.02	6.09	11.2
	60%	1%	15.22	21.3	6.96	13.65
		3%	19.11	12.48	6.43	17.78
Jasmine Extract Oil	10%	1%	18.77	2.99	6.12	2.54
		3%	21.62	1.03	5.80	3.5
	20%	1%	17.90	7.49	6.28	4.87
		3%	21.38	2.12	5.93	6.2
	60%	1%	16.81	13.1	6.54	8.57
		3%	20.34	6.87	6.14	10.61
Lonkom Perfume	10%	1%	18.07	6.57	6.34	5.23
		3%	20.75	4.98	5.95	7.27
	20%	1%	16.74	13.48	6.65	9.74
		3%	19.60	10.24	6.21	12.56
	60%	1%	14.62	24.42	7.12	15.21
		3%	18.06	17.31	6.52	20.49
Gambit Perfume	10%	1%	18.38	4.99	6.19	3.3
		3%	21.50	1.56	5.85	4.72
	20%	1%	17.31	10.54	6.42	6.58
		3%	20.56	5.87	6.05	8.65
	60%	1%	15.97	17.47	6.75	10.24
		3%	19.60	10.24	6.24	14.25

Note: Values of Tensile strength (in kgf) and elongation at break (%) for modified Corafix Yellow GDR dye (without encapsulating perfume) were 19.34,21.84 and 5.91%, 5.66% for 1% and 3% dye shade respectivel.

Table IX. Effect of perfumes and their concentrations on tensile properties of fabric dyed with modified Corafix yellow F3R dye

Type of perfume	Perfume concentration	Shade (%)	Tensile strength (kgf)	Loss in tensile strength (%)	Elongation at break (%)	Loss in elongation at break (%)
Citronella Extract Oil	10%	1%	18.50	3.83	6.04	3.09
		3%	21.84	1.23	5.13	5.87
	20%	1%	17.81	7.4	6.27	6.48
		3%	21.10	4.56	5.30	9.89
	60%	1%	15.81	17.79	6.66	10.98
		3%	19.78	10.54	5.52	16.74
Jasmine Extract Oil	10%	1%	18.73	2.6	5.92	2.1
		3%	21.72	1.76	5.08	3.78
	20%	1%	17.91	6.87	6.10	4.58
		3%	21.33	3.5	5.20	6.89
	60%	1%	16.61	13.65	6.35	7.45
		3%	20.40	7.74	5.35	11.21
Lonkom Perfume	10%	1%	18.18	5.48	6.12	4.99
		3%	21.25	3.89	5.22	7.23
	20%	1%	16.98	11.7	6.37	9.25
		3%	20.28	8.26	5.44	11.63
	60%	1%	14.94	22.31	6.82	15.02
		3%	18.74	15.21	5.72	19.54
Gambit Perfume	10%	1%	18.29	4.89	6.06	4.2
		3%	21.76	1.58	5.18	6.19
	20%	1%	17.21	10.52	6.28	8.4
		3%	20.78	5.99	5.39	10.11
	60%	1%	15.37	20.09	6.73	12.54
		3%	19.35	12.45	5.60	17.87

Note: Values of Tensile strength (in kgf) and elongation at break (%) for modified Corafix Yellow F3R dye (without encapsulating perfume) were 19.23, 22.11 and 5.71% , 4.98% for 1% and 3% dye shade respectively.

5.2.7 Olfactometry analysis of perfume intensity with respect to time (days)

Tables X to XII represent the olfactory analysis of the fragrance present on to the fabric dyed with Corafix Red GD3R, Corafix Yellow GDR and Corafix Yellow F3R respectively after number of days of evaluation. It can be easily seen that the fragrance intensity decreased day by day irrespective of the quantity and type of the perfume loaded as well as the type and shade (%) of the dye studied. This was because of the volatile nature of the perfumes. It can also be concluded that the perfume concentrations as well as the shade (%) of the dye also affected the perfume intensity. It can be observed from all tables X to XII that the perfumes withstood longer with the increase in the % dye shade as well with increase in perfume concentration. All the tables reveal that the perfumes withstood more than 30 days on the fabric in all cases. In all the individual case, the intensity of perfumes was almost same in the initial days of evaluation which gradually decreased with increase in number of days of evaluation. It can also be viewed that in the case of the oil based perfumes; citronella extract oil gave good results compared to Jasmine extract oil. As same that in the case of the solvent based perfumes; lonkom perfume gave very good results compared to that of the gambit perfume. The perfume intensity after number of days completely depended on the type of the perfume used and the structure of the dyes. This was because the β-cyclodextrin cannot form complex with all the moieties of any size. The Jasmine extract oil could not withstood more than 20 days when its concentration was 10% for any shade of Red dye (Table X). Yellow GDR (Table XI) showed overall very good results for all the perfume concentrations for all shade (%). Yellow F3R (Table XII) showed very poor results almost for all the perfumes except for Lonkom perfume.

Table X. Olfactometry analysis of cotton treated with modified Corafix red GD3R dye loaded with various perfumes with respect to time

Type of perfume	**Perfume concentration**	**Number of days →** **Shade (%) ↓**	**5th Day**	**10th Day**	**20th Day**	**30th Day**
Citronella Extract Oil	10%	1%	4	3	1	1
		3%	4	4	2	1
	20%	1%	4	3	1	1
		3%	5	4	4	2
	60%	1%	5	4	3	2
		3%	5	4	3	2
Jasmine Oil	10%	1%	3	2	1	0
		3%	3	3	1	0
	20%	1%	3	2	1	1
		3%	4	3	3	1
	60%	1%	4	4	3	2
		3%	5	4	4	2
Lonkom Perfume	10%	1%	5	4	4	2
		3%	5	4	2	1
	20%	1%	5	4	4	2
		3%	5	4	3	1
	60%	1%	5	4	4	2
		3%	5	4	3	2
Gambit Perfume	10%	1%	4	2	2	0
		3%	4	3	2	1
	20%	1%	4	3	1	0
		3%	5	4	2	1
	60%	1%	4	3	2	1
		3%	5	4	2	2

Table XI. Olfactometry analysis of cotton treated with modified Corafix yellow GDR dye loaded with various perfumes with respect to time

Type of perfume	Perfume concentration	Number of days → / Shade (%) ↓	5th Day	10th Day	20th Day	30th Day
Citronella Extarct Oil	10%	1%	4	4	3	1
		3%	4	4	3	1
	20%	1%	4	3	2	1
		3%	4	4	3	2
	60%	1%	5	5	4	2
		3%	5	5	4	3
Jasmine Extract Oil	10%	1%	4	3	2	1
		3%	3	1	1	1
	20%	1%	4	3	2	1
		3%	3	2	1	1
	60%	1%	4	4	3	1
		3%	3	3	2	1
Lonkom Perfume	10%	1%	4	3	2	1
		3%	4	4	3	1
	20%	1%	4	4	3	1
		3%	4	4	3	1
	60%	1%	5	4	3	1
		3%	5	4	4	2
Gambit Perfume	10%	1%	4	2	2	1
		3%	4	3	2	1
	20%	1%	4	2	3	1
		3%	4	3	3	1
	60%	1%	4	2	1	1
		3%	5	4	3	1

Table XII. Olfactometry analysis of cotton treated with modified Corafix yellow F3R dye loaded with various perfumes with respect to time

Type of perfume	Perfume concentration	Number of days → / Shade (%) ↓	5th Day	10th Day	20th Day	30th Day
Citronella Extarct Oil	10%	1%	3	2	1	0
		3%	4	3	2	0
	20%	1%	5	4	3	1
		3%	4	3	3	1
	60%	1%	5	4	3	1
		3%	5	4	4	1
Jasmine Extract Oil	10%	1%	3	1	1	0
		3%	3	1	1	0
	20%	1%	3	1	1	0
		3%	3	2	1	0
	60%	1%	3	2	1	0
		3%	4	2	1	1
Lonkom Perfume	10%	1%	3	2	1	0
		3%	4	3	1	0
	20%	1%	3	2	1	1
		3%	5	4	3	1
	60%	1%	4	3	2	1
		3%	5	4	3	1
Gambit Perfume	10%	1%	3	2	1	0
		3%	4	3	2	0
	20%	1%	3	2	1	0
		3%	4	4	3	1
	60%	1%	4	3	2	1
		3%	5	4	3	1

5.2.8 Olfactometry analysis of perfume intensity with respect to washing cycles

Tables XIII to XV represent the effect of number of washings onto the intensity of the perfume present onto the cotton fabric dyed with modified heterobifunctional reactive dyes viz., Corafix Red GD3R, Corafix Yellow GDR and Corafix Yellow F3R. This assessment gave an idea about the life of perfume on number of washing cycles given to the fabric in the day to day routine life. It can be clearly seen that the fragrance intensity became weaker on every wash given irrespective of the quantity and type of the perfume loaded as well as the type and shade of the dye studied. This was because of the volatile nature of the perfumes. The table shows the olfactory analysis of the fabrics done before washings as well. The ratings revealed that all the samples gave very strong sensation of the perfume before washing irrespective of the quantity and type of the perfume loaded as well as the type and shade of the dye studied which remains almost same even on first wash. It was observed from the tables that the Yellow GDR (Table XIV) and Yellow F3R (Table XV) showed better smell before washing compared to the red dye. This then gradually decreased with number of wash cycles. In case of the oil based perfumes, Jasmine extract oil could not withstood number of wash cycles as compared to that of the Citronella extract oil. In case of the solvent based perfumes, Lonkom perfume withstood number of washing cycles as compared to Gambit perfume. This was basically due to the properties of the perfumes and dyes used. If the dyes were compared then, it can be clearly visible from the tables that Yellow GDR and Yellow F3R showed overall good results compared to red dye. From all this discussions, it can be clearly said that the number of washes influenced the perfume intensity to very large extent.

Table XIII. Olfactometry analysis of cotton treated with modified Corafix red GD3R dye loaded with various perfumes with respect to washing cycles

Type of perfume	Perfume concentration	Number of washings → / Shade (%) ↓	Before washing	1st	2nd	3rd	4th	5th
Citronella Extract Oil	10%	1%	4	4	3	3	1	0
		3%	4	4	3	3	2	1
	20%	1%	4	5	4	3	3	1
		3%	4	4	4	3	2	2
	60%	1%	4	5	4	4	3	1
		3%	5	5	4	3	3	2
Jasmine Extract Oil	10%	1%	4	3	2	1	0	0
		3%	4	3	2	1	0	0
	20%	1%	4	3	2	1	0	0
		3%	4	4	3	1	1	0
	60%	1%	4	4	3	1	1	0
		3%	4	4	3	2	2	1
Lonkom Perfume	10%	1%	4	4	3	2	1	0
		3%	4	4	4	3	3	1
	20%	1%	4	4	3	2	1	0
		3%	5	4	4	4	3	1
	60%	1%	5	5	4	2	1	1
		3%	5	5	5	4	3	3
Gambit Perfume	10%	1%	4	4	2	1	0	0
		3%	4	4	3	2	1	0
	20%	1%	4	4	2	1	1	0
		3%	5	4	4	3	2	1
	60%	1%	5	5	3	2	1	0
		3%	5	5	5	4	2	1

Table XIV. Olfactometry analysis of cotton treated with modified Corafix yellow GDR dye loaded with various perfumes with respect to washing cycles

Type of perfume	**Perfume concentration**	**Number of washings →** **Shade (%) ↓**	**Before washing**	**1st**	**2nd**	**3rd**	**4th**	**5th**
Citronella Extract Oil	10%	1%	4	4	3	1	1	0
		3%	5	4	3	2	1	1
	20%	1%	4	4	3	3	2	1
		3%	5	4	3	3	1	1
	60%	1%	4	4	4	3	2	1
		3%	5	5	4	3	2	2
Jasmine Extract Oil	10%	1%	4	3	2	1	0	0
		3%	4	3	2	1	1	0
	20%	1%	4	4	3	3	1	0
		3%	4	3	2	1	1	1
	60%	1%	4	4	3	3	2	1
		3%	4	4	3	2	1	1
Lonkom Perfume	10%	1%	4	4	3	3	2	1
		3%	5	4	3	1	1	1
	20%	1%	4	4	3	3	2	1
		3%	5	4	4	3	1	1
	60%	1%	5	5	4	4	3	2
		3%	5	5	4	3	1	1
Gambit Perfume	10%	1%	4	3	2	1	1	0
		3%	4	3	2	1	1	1
	20%	1%	4	4	3	1	1	1
		3%	5	4	3	2	1	1
	60%	1%	5	4	4	2	1	1
		3%	5	5	4	4	2	1

Table XV. Olfactometry analysis of cotton treated with modified Corafix yellow F3R dye loaded with various perfumes with respect to washing cycles

Type of perfume	Perfume concentration	Number of washings → / Shade (%) ↓	Before washing	1^{st}	2^{nd}	3^{rd}	4^{th}	5^{th}
Citronella Extract Oil	10%	1%	4	4	3	1	1	0
		3%	5	4	3	1	1	1
	20%	1%	4	4	3	3	2	1
		3%	5	4	3	2	1	1
	60%	1%	4	5	3	3	3	2
		3%	5	5	3	3	2	2
Jasmine Extract Oil	10%	1%	4	3	2	1	1	0
		3%	4	3	2	1	1	1
	20%	1%	4	3	2	2	1	1
		3%	4	4	3	1	1	1
	60%	1%	4	4	4	2	1	1
		3%	4	4	4	2	2	2
Lonkom Perfume	10%	1%	4	3	3	1	1	0
		3%	5	4	3	3	1	1
	20%	1%	4	4	4	3	1	1
		3%	5	5	4	4	3	2
	60%	1%	5	4	4	4	2	2
		3%	5	5	5	4	3	2
Gambit Perfume	10%	1%	4	3	3	1	1	0
		3%	4	3	2	1	1	1
	20%	1%	4	4	3	2	1	1
		3%	5	4	3	2	1	1
	60%	1%	5	4	3	3	1	1
		3%	5	5	4	4	3	2

6. Conclusions

As a host molecule β-Cyclodextrin could be satisfactorily grafted to heterobifunctional reactive dyes and subsequently loaded with perfume to form fragranced dyes. The grafting was characterized by FTIR analysis. The grafted β-Cyclodextrin was successfully attached to cotton fabric via modified dyes. Using the inclusion complex forming property of β-Cyclodextrin, various perfumes were successfully encapsulated in modified dyes. As depicted from various studies on different dyes used showed different results. The modified dyes showed slight reduction in colour value due to β-Cyclodextrin grafting. All the treated samples were examined for graft %, colour strength and tensile properties. The characterization of the treated textiles revealed that β-Cyclodextrin grafting influenced all the properties assessed for the dyes studied. The extent of effects varied from dye to dye. The extent of perfume intensity found to be different for different perfumes. Olfactometry Analysis of the treated samples showed that perfume intensity became weaker with respect to time and washing cycles. The intensity varied depending on the type and concentration of the perfumes used. The study generated a new avenue of fragranced dye synthesis and their application to textiles for better feel.

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