Indian Journal of Fibre & Textile Research
Vol. 40, June 2015, pp. 140-143

Embedding aromatherapy essential oils into textile fabric using β-Cyclodextrin inclusion compound

Subhas Ghosh[a] & Natalie Chipot
College of Technology, Eastern Michigan University, Ypsilanti, Michigan, USA

Received 31 October 2013; revised received and accepted 15 April 2014

β- Cyclodextrin inclusion compounds have been attached to the 100% cotton and 50:50 cotton/polyester fabrics using sol-gel, prepared from 3-glycidoxypropyltrimethoxysilane and tetraethoxy orthosilicate. Aromatherapy essential oils, having eucalyptus, lavender and lemon fragrances, have been applied on to the treated fabrics by a selected application method. The durability of these fragrances on fabrics has been rated by four judges for six weeks, and up to six washings. The judges rated the fragrance intensity once a week during this period. The scent ratings are found to decrease over six weeks and six washing; however, the fabrics continued to have a decreasing fragrance after six weeks. Fabric abrasion resistance, thermal resistivity and moisture vapor resistivity do not alter significantly; however, an increase in the tensile strength of the polyester/cotton blend fabric is observed after the application of the finish.

Keywords: Aromatherapy, Cotton, Cotton/polyester fabric, β – Cyclodextrin, Essential oils, Sol-gel

1 Introduction

Aromatherapy is the use of concentrated essential oils extracted from herbs, flowers and other plants to treat various types of ailments. Brown[1] reported the effects of individual fragrances on human health. Essential oils, like all organic compounds, are made up of complex mixtures of several hydrocarbons[2] such as terpenes, alcohols, esters, aldehydes, ketones, oxides and phenols. Even a simple essential oil may contain 80 - 300 different chemical constituents. Hu *et al*.[3] described the creation of a perfume release fabric using rose fragrance nano-capsules directly on finished cotton fabrics. The authors claimed an excellent sustained fragrance release property. Wang and Chen[4] used aromatherapy oil on textile fabric after treating the fabric with β-Cyclodextrin, where the inclusion compound was applied on to the fabric using the traditional pad method. Normand *et al*.[5] studied the effect of surfactant micelles on the deposition of perfume molecules on fabric using mass transfer mechanisms. This method was used to measure the performance of key raw perfume materials. Srivastava *et al*.[6] applied perfume by finishing fabric with fragrance oil, with propylene glycol using a spray application method and also by using a padding method where silicon softener was used with fragrance solution. Our investigation is based on the study of the chemical agent release from textile fabric, where aromatherapy essential oils were used as the releasing agent. The effects of aromatherapy on the human body are not within the scope of this study.

Cyclodextrins are polysaccharides having 6-8 D–glucose units and are formed during the enzymatic breakdown of starch. The D–glucose units are covalently linked at the carbon atoms C-1 and C-4[7, 8]. Wang and Cai[9] described the Cyclodextrins as toroidal-shaped cyclic oligosaccharides with a hydrophilic outer surface, and an internal hydrophobic hollow interior that has an affinity for lipid compounds into their oleophilic cavity.

β-Cyclodextrin was chosen for this investigation as a releasing agent, because of the feasibility of its use in textile finishes, and its ability to hold the essential oils in its oleophilic cavity. It's acceptability for use in textile finishes is based on detailed studies by Brewster[10] on the toxicity, mutagenicity, teratogenicity, and carcinogenicity of Cyclodextrins and their derivatives. Cyclodextrins are considered to be very important environment-friendly auxiliaries because they are biodegradable and show no toxic symptoms[10]. It is suggested by Buschmann[11] that while using Cyclodextrins for textile applications, some derivatives may be chemically bonded to or permanently fixed to fabrics such as cotton and

[a]Corresponding author.
E-mail: sghosh@emich.edu

polyester. Sols are the dispersion of colloidal particles in liquid having a diameter ranging from 1 nm to 100 nm. Gels are interconnected showing rigid networks with pores of sub-nanometer dimensions and polymeric chains, whose average length is greater than a nanometer[12]. The sol-gels used in this investigation were prepared from two precursors, 3-glycidyloxypropyltrimethodoxysilane (GPTMS) and tetraethoxy orthosilicate (TEOS). TEOS makes a sol and forms a gel when cross-linked with GPTMS.

In this study, cotton and polyester/cotton fabrics were treated with sol-gel, which attaches with cotton cellulose through covalent bonding[13,14]. The durable attachment of sol-gel on polyester is attributed to the secondary bonding of sol-gel on polyester, and cross linking during curing that forms a film on the fabric surface. β- Cyclodextrin molecules are held in place in the micro- porous structure of a sol-gel- network. Essential oils are introduced into the cavities of β- Cyclodextrin inclusion complex. This article describes a unique synthesis method of attaching aromatherapy essential oil to the textile fabric using sol-gel and β- Cyclodextrin.

2 Materials and Methods

Wang and Chen's[13] procedure was modified to create a new synthesis procedure in this study. GPTMS, TEOS, HCL, dimethyl sulfoxide, dibutyl amine, β- Cyclodextrin and methanol were obtained from Sigma-Aldrich and used without further purification. Aromatherapy essential oils were purchased from Mountain Rose Herbs Company. Cotton (100%) and polyester/cotton (40:60) blend fabrics were used. The cotton fabric weight was 3.5 oz./ yard^2, which was plain weave with a 87 × 62 (ends × picks per inch) construction, while the polyester/cotton (40:60) blend fabric weighed 5 oz/ yard^2 with 100 × 47 (ends × picks per inch) construction. The specimens from both fabrics were prepared by washing them with 2g of disodium carbonate in 1 L water for 1 h at 75°C. The fabric was then washed and dried.

2.1 Preparation of TEOS Solution

One gram of HCl was mixed well in a beaker with 99 g of DI water. Sixty five gram (0.3 mole) of TEOS was poured into a plastic beaker and the HCl solution was added while stirring at a high speed and maintaining the average *p*H at 3.5, that varied within the range of 3.0 and 4.0. The solution was stirred for 8 h.

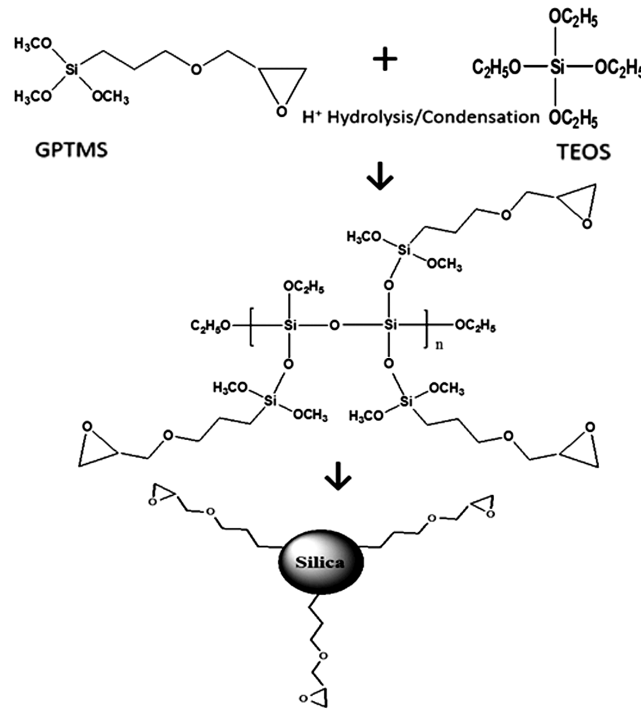

Fig.1—Schematic structure of sol-gel formation

2.2 Preparation of β- Cyclodextrin Solution

β- Cyclodextrin (36g or 0.3 moles) was mixed with 130g of dimethyl sulfoxide anhydrous and stirred until the solution became clear. The stirring continued for one hour after the solution became clear.

2.3 Preparation of Sol-gel with GPTMS and TEOS followed by Addition of β- Cyclodextrin

In order to open GPTMS' glycedal ring, 0.1 g of dibutyl amine was mixed with 70.8 g (0.3 moles) of GPTMS, and stirred for 10 min. The TEOS solution was then added and the solute was reduced to 60% with methanol and distilled water, followed by stirring for 15 min. The sol-gel is formed by a hydrolysis reaction, followed by a condensation reaction. The schematic structure of a sol-gel formation is shown in Fig. 1, where some free Si-O-Et or Si-O-Me will eventually hydrolyze/condense to form epoxy-functional silicate particles. β- Cyclodextrin solution was then added to the mixture and stirred for 1h.

2.4 Application on Textile Fabric

Both cotton and polyester/cotton fabric specimens were dipped into the solution while stirring and the specimen processed through the padder to evenly distribute the solution under pressure (0.18 lb/ inch^2). Three specimens were prepared for each sample and each specimen went through the application process twice. The wet pick- up for cotton fabric varied from

123% to 125% and it varied in the range of 127% - 134% for polyester/cotton fabric. The cotton fabric weight was 3.5 oz. /yard^2, while the polyester/cotton fabric weighed 5 oz. /yard^2. The specimens were dried in an oven at 160^o C for an hour. The dry add-on was measured for both specimens, which revealed an average of 24.7% and 31% (w/w) dry add-ons for the cotton and the poly/cotton fabric respectively. A higher add-on for the polyester/cotton fabric can be attributed to 42% higher fabric weight.

2.5 Essential Aromatherapy Oil Application

The dried specimen fabrics were force sprayed with undiluted aromatherapy essential oils until the specimens were completely soaked. It was observed that spray application yielded better results in the scent intensity than the dip and padding application process. The spraying force helped the essential oil to penetrate the cavities of the β- Cyclodextrins. All of the specimens were dried overnight at ambient conditions.

2.6 Scent Intensity and Durability Evaluations

Four healthy judges were selected complying with the human subject criteria of Eastern Michigan University, for this evaluation. Over a period of six weeks, the judges smelled the samples every five days. After each wash the specimen's strength of fragrance was recorded. The rating scale used was an ordinal scale, 5 being a very strong scent and 0 being no scent. During this period, samples were washed 6 times at 50^o C with tide commercial detergent (containing sodium per carbonate, sodium sulfate, sodium carbonate, sodium alumininuosilicate, sodium polyacrylate, sodium alkyl benzene sulfonate, DPA, polyethylene glycol, sodium palmitate, amylase, modified starch and FD&C blue) and air dried at ambient conditions.

The treated fabrics were also tested for tensile strength, abrasion resistance, thermal resistivity and moisture vapor resistivity to determine any significant changes in the test fabrics due to the applied agents' release finish. The fabric breaking strength was measured according to the ASTM D 5034 method using MTS Test Works, model 200/L system. Each sample was tested 5 times and their average as well as standard deviation was recorded. The abrasion resistance of the fabric was determined following the ASTM D 4966 method on a Martindale abrasion tester. Thermal and evaporative resistance measurements were conducted using the ASTM method F 1869 by a sweating hot plate from Measurement Technology NW Inc.

3 Results and Discussion

The fragrance durability of cotton and polyester/cotton fabrics are shown in Table 1. The average rating was based on the 4 individual specimens for each fabric type scent and the 4 judges. A 0.25 standard error of estimate in ratings was determined at a 95% level of confidence.

In most cases the judges rated the fragrance intensity very similarly. Fragrance release from the fabrics is influenced by the presence of β- Cyclodextrin and the solvent used in the aromatherapy oils. In general, the rating points are higher for the polyester/cotton fabric than that for the cotton fabric for all scents. This phenomenon can be attributed to a higher add-on of β- Cyclodextrin onto the polyester/cotton fabric, which causes it to hold more aromatherapy essential oils. The first rating has been given to the fabrics the day after the scents are applied, and the final rating is given after a period of 6 weeks and 6 washes. As expected, with time, a general decreasing trend of ratings is observed for all fragrances on both fabrics. The rating values do not show a significant difference in the scents' intensity of the different types of scents used. The presence of the scents is greatly reduced after 6 weeks and 6 washes; however, they do not disappear completely. For polyester/cotton fabric, the fragrances of lemon and lavender are clearly evident after 6 weeks. After

Table 1—Average scent intensity rating by judges

Evaluation day	Eucalyptus		Lemon		Lavender	
	Polyester/cotton	Cotton	Polyester/cotton	Cotton	Polyester/cotton	Cotton
1 (No wash)	4.25	3.25	5.00	4.25	4.25	3.25
5 (1wash)	3.50	3.38	4.38	3.13	4.75	3.13
10 (2 wash)	3.75	2.25	4.50	2.50	4.25	2.75
15 (3 wash)	3.25	2.00	3.75	2.50	4.00	2.25
20 (4 wash)	2.75	2.00	3.50	2.00	3.25	2.00
25(5 wash)	2.00	1.63	2.25	1.75	2.63	1.63
30 (6 wash)	0.50	1.00	1.88	0.50	1.25	0.75

Table 2—Treated and untreated fabric properties

Fabric	Breaking strength lbf		Weight loss due to abrasion %		Thermal resistivity Rct m^2 $^oC/W$		Water vapor resistivity Rct m^2 $^oC/W$	
	C	P/C	C	P/C	C	P/C	C	P/C
Treated	46.3	129.2	0.67	0.081	0.0423	0.0317	3.2419	5.3621
Untreated	46.4	47.5	0.76	0.021	0.0312	0.0154	4.7384	4.7476

C— Cotton and P/C— Polyester/cotton.

treating the fabric with sol-gel and β-Cyclodextrin the treated samples are tested for tensile strength, abrasion resistance, thermal resistivity and moisture vapor resistivity. These evaluations are performed to examine the fabrics for any significant changes in these important textile properties. Both the cotton and the polyester/cotton fabrics properties are illustrated in Table 2.

The strength of the cotton fabric does not change significantly due to the application of sol-gel and β-Cyclodextrin. The strength values are 46.4 lbf for untreated and 46.3 lbf after treatment having standard deviations of measurements 1.93 and 2.50 respectively. The strength of the polyester/cotton fabric exhibits a significant increase from 47.5 lbf to 129.2 lbf with standard deviations of 3.75 and 7.70 respectively, due to the application of the finish. The fabrics are dried at an elevated temperature (160^o C), but at this temperature the cotton fabric does not show any significant loss in strength. This may be attributed to the compensating effects of sol-gel film on the fabric providing further integrity to the fabric structure. The increase in strength for polyester/cotton fabric is probably caused by the higher level of finish add-on (31% w/w). This may arise because sol-gel forms a stronger film on the fabric that is bonded to the polyester/ cotton fabric. This provides a greater integrity to the fabric structure. The differences in the polyester/cotton fabrics strength are much higher than the standard deviation of measurements, indicating a true change in strength. Abrasion data reveal no significant changes in the fabrics abrasion resistance. The small amount of fibre loss detected does not have any significant effect on the fabric performance. The differences in thermal resistivity and moisture vapor resistivity between the treated and untreated fabrics appear to be within normal experimental variations. In this study, as a result of the application of the agent releasing finish, no significant detrimental effects on these two fabrics are detected.

4 Conclusion

β- Cyclodextrin was used as an inclusion complex for 3 different scents of essential oils. Sol-gel chemistry was used to anchor β- Cyclodextrin onto the fabrics. The aromatherapy essential oils were sprayed into the oleophilic cavities of β- Cyclodextrin. The scents lasted through at least six washes and 6 weeks after the application; however, they continued to provide fragrance even after the last evaluation. The polyester/cotton fabric was found to have a higher scent intensity, which was thought to be due to a loading of a greater amount of the β- Cyclodextrin that held more aromatherapy oil into the cavities. Higher amounts of β- Cyclodextrin, in the pores of sol-gel may be used to increase the scent's intensity and durability.

References

1 Brown D, *Aromatherapy*, Lincolnwood (NTC Publishing Group, Chicago) 1996, 51.
2 www.therapeuticgradecom/refs/chemistry.html (accessed on 29.9.2013.)
3 Hu J, Xiao Z, Ma S, Wang M & Li Z, *Chinese Chem Eng,* 19 (3) (2011) 523.
4 Wang C & Chen Sh, *Industrial Text,* 40 (2010) 12
5 www.researchgate.net/publication/230272112- Modeling-per (accessed on 15.9.2013).
6 www.indiantextilejournal.com/articles/FAdetails.asp?id=5298 (accessed on 20.8.2013).
7 http://en.wikipedia.org/wiki/Cyclodextrin (accessed on 16.4.2014).
8 http://www.sigmaaldrich.com/catalog/product/sigma/c4805?lang=en&region=US (accessed on 28.1.2009).
9 Wang J & Cai Z, *Carbohydrate Polym, 72* (2008) 695.
10 Brewster M E, in *New Trends in Cyclodextrins and Derivatives*, edited by D Duchêne (Editions de Santé, Paris), 1991, 313.
11 Buschmann H J, Knittel D & Schollmeyer E, *J Phys Macromol Chem,* 72 (2001) 169.
12 Mohlitig B & Textor T, *J Sol-Gel Sci Technol*, 39 (2006) 111.
13 Ghosh S, Mannari V & Yadav S, in *Recent Developments in Applied Polymer Science*, edited by S G Pandalai (Research Signpost, Kerala, India), 2009, 331.
14 Wang C X & Chen S L, *Fibers Text Eastern Eur,* 13 (2006).