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Encapsulation of Pomegranate Seed Oil in Beta-Cyclodextrin

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Abstract— Cyclodextrins (CD)s play an important role in innovative textile processing and the functionalization of textiles. In the textile industry CDs may have many applications such as: absorption of unpleasant odours; they can complex and release fragrances, using as skin-care-agents, bioactive substances and filters for adsorption of small pollutants from waste water. The uses of beta-CDs provide different opportunities for developing new innovative products and eco friendly textile processes which are of specific interest to the textile industry. Due to the anti-oxidant effect of pomegranate seed oil (PSO), in this study encapsulation of PSO by beta-CD was carried out and inclusion complexes were formed successfully. The formed compound was applied onto the textile substrate and both formulation and surface morphology of the substrates were discussed in this study.

Index Terms— Cyclodextrin, complexes, encapsulation, formulation, inclusion, morphology, pomegranate

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1 Introduction

Pomegranate (Punicagranatum L.) is one of the fruit that has the highest concentration of total polyphenols when compared to other fruits. Pomegranate seed oil consists of 65% to 80% conjugated fatty acids [1,2]. They are fruits rich in aril, the percentage of which ranges from 50 to 70% of total fruit and comprises of 78% juice and 22% seeds. Oil content of pomegranate seed varies from 12 to 20% of the seed on a dry weight basis [2]. It was reported to present biological properties such as antioxidant and eicosanoid, enzyme inhibition properties, immune function and lipid metabolism (Yamasaki and others 2006), estrogen content, skin photoaging inhibition effect, lipoperoxidation and activity of antioxidant enzymes, toxicological evaluation, and protective effect against gentamicin-induced nephrotoxicity [1]. It has been also reported that it is anti□inflammatory, antioxidant, antiangiogenic, immunomodulator, and chemopreventor [3].

Pomegranate seed oil has high levels of tocopherols, mainly γ-tocopherol followed by α-tocopherol, β-tocotrienol, α-tocotrienoland δ-tocopherol. The abundance of γ-tocopherol could be one of the reasons why pomegranate seed oil is highly resistant to oxidation, as this tocopherol has high antioxidant activity.Pomegranate oil has various health-promoting traits due to its higher concentration of unique antioxidants and anticancer agents [4].

Conjugated fatty acid is the general term of position land geometric isomers of polyunsaturated fatty acids with double bonds in conjugation. It has attracted considerable attention because of its potentially beneficial biological effects such as anti-oxidant property. These natural anti-oxidants which occur in vegetable oils are always preferred over the synthetic anti-oxidants from health point of view. Unsaturated fatty acids oxidize rapidly. Differences in chain length, degree of unsaturation, position and stereo isomeric configuration of the double bonds make the unsaturated fatty acids vulnerable to oxidation and polymerization. Microencapsulation would serve to protect these fatty acids [5].

Microencapsulation is used to encapsulate solid, liquid, and gaseous materials. These capsules can release their contents at controlled rates over prolonged periods. The simplest microcapsule consists of a core or active agent surrounded by a wall or carrier material. Some of the carrier materials used in food microencapsulation are proteins (e.g., sodium caseinate, whey proteins, gelatin, and soy proteins), hydrocolloids (e.g., modified starches and gums), hydrolyzed starches (glucose, lactose, corn syrup, and maltodextrin), lipids (mono□, di□, and triglycerides), and cellulosic materials (methyl□ and ethyl□cellulose and carboxy methyl cellulose). Many techniques are available to encapsulate food ingredients [6]. Microencapsulation is one of the methods that incorporates materials for release under controlled conditions mechanically, electrically, chemically or by- leaching action in a liquid environment [7-9].

Cyclodextrins (CD)s are non-toxic macrocyclic oligosaccharides, consisting of (a-1,4)-linked a-D-glucopyranose units, with a hydrophilic outer surface and hollow hydrophobic interior. Number of glucose units determines the diameter of inner cavity of cyclodextrins. Since alpha cyclodextrin compose of 6 glucose units, it has the smallest cavity with a diameter of 0,49 nm. Beta cyclodextrin and gamma cyclodextrins compose of 7 and 8 glucose units and their cavity sizes are 0.62 and 0.79 nm, respectively [10].

Cyclodextrins (CDs) are relevant molecules used in different applications and industries from pharmacology,

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cosmetics, textiles, filtration to pesticide formulations [11]. Cyclodextrins are cyclic oligosaccharides composed of glucose units linked by α-1,4-glycosidic bonds. There are three types; α-cyclodextrin, β-cyclodextrin, γ-cyclodextrin, which are composed of 6, 7, and 8 α-1,4-glycosidic bonds as shown in Figure 1 [12]. The radii of the rigid cavities vary from 0.50 to 0.85 nm. In these cavities guest molecules can be enclosed [13].

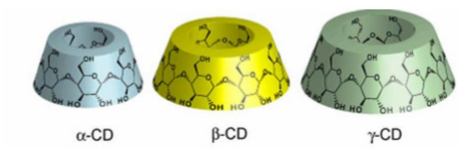

Fig.1. Types of CDs [11]

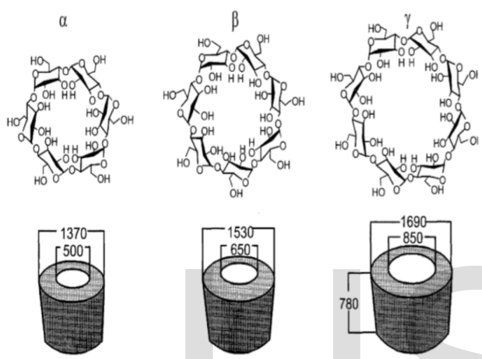

Fig.2. Structure of CDs (in dimensions)[13]

The peculiar shape, and the presence of a hydrophobic cavity in cyclodextrins produce the extraordinary capability of these hosting species to include a large variety of different molecules, and form stable inclusion complexes (IC) and supramolecular adducts [14]. It has been reported that the inclusion complexes of guest compounds with CDs can enhance guest stability, improve the aqueous solubility, protect against oxidation, light-induced decomposition, and heat-induced changes, and mask or reduce unwanted physiological effects, and reduce volatility. Amongst the CDs, beta-CD is widely used since its cavity size is suitable for common guests with molecular weights between 200 and 800 g/mol and also due to its availability and reasonable price [15]. Hence, in this study pomegranate seed oil was encapsulated in beta-cyclodextrin and after encapsulation process, formed inclusion complexes were applied on polypropylene textile fabric. Morphology characteristics of capsules were investigated.

2 Experimental

2.1 Materials

Pomegranate seed oil was purchased from Zade Vital (Turkey) while beta-CD, sodium sulphate and formaldehyde were taken from Sigma Aldrich. Polypropylene nonwoven fabric was supplied from Mogul Gaziantep, Turkey.

2.2 Experimental: Co-precipitation method

Co-precipitation method comprised of dissolving beta-CD in distilled water at a ratio of 1:10 and subsequent addition of 3.5 g PSO to this solution drop by drop at 20 ^{0}C. After addition of PSO to the CD solution, they were mixed together for 30 minutes. The mixture was stirred at 1500 rpm prior to the addition of 1 ml sodium sulphate solution (25%) to separate the shell and core materials. 1.8 g formaldehyde was then added under continuous stirring for solidifying the shell material. Finally, cooling was carried out at -18^{0}C for 12 hours.

2.3 Application to Textile

Hydrophilic polypropylene nonwoven fabric was conditioned at 20^{0}C±2^{0}C and 65 % ±2 humidity for 24 hours. After formation of inclusion compound, complexes were applied to fabrics by pad-dry method with 80 % wet pick-up ratio. The treated fabrics were dried at room temperature for 12 hours.

2.4 Washing Resistivity

In order to determine the effect of washing resistivity, the treated fabrics were washed five times after the pad-dry process in Whirlpool home type washing machine with a short washing program at 40 ^{0}C for 60 min with 4 g/L of domestic detergent.

2.5 Scanning Electron Microscope (SEM) Analysis

Surface morphology of polypropylene fabric samples were scanned by an electron microscope by Hitachi S-3200N 20 kV under a high vacuum at 1000 and 6000 magnification after being coated with gold-palladium.

2.6 Color Spectrum Test

CIELAB color spectrum results including L*,a*,b*, ΔE values, yellowness indexes and brightness indexes of untreated and treated samples were indicated by Spectraflash SF 600X Datacolor Reflectance Spectrophotometer.

3 Results and Discussion

3.1 Scanning Electron Microscope (SEM) Analysis Results

SEM images of untreated fabric and treated with PSO-beta-CD inclusion compounds were shown in Figure 3. While untreated fabric showed a smooth surface with no superficial particle/droplet, droplets deposited on the textile fibers were observed on the fabrics treated PSO-beta-CD inclusion compounds. When 5 times washed treated fabrics were examined, it could be clearly seen at 1000 magnification that deposited particles were decreased compared to the unwashed treated fabrics.

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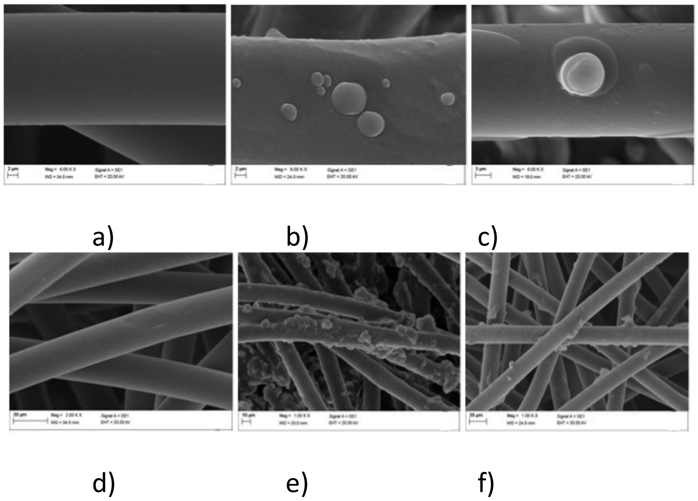

Fig.3.SEM images of a)Untreated sample, b) PSO-beta-CD deposited fabric, c) 5 times washed PSO-beta-CD deposited fabric at 6000 magnification, d) untreated sample, b) PSO-beta-CD deposited fabric, c) 5 times washed PSO-beta-CD deposited fabric at 1000 magnification

3.2 Color Spectrum Test Results

According to the color spectrum test results (Table 1), there was not a significant difference in ΔE values when untreated and treated samples were compared to each other. It was considered that formulation of inclusion complexes of PSO-beta-CDs could be applied on to polypropylene fabrics with padding application without changing the color values dramatically. Treatment of inclusion compounds on to textile fabrics caused a slight increase in yellowness indexes while washing procedure caused an increase in brightness index.

TABLE 1
COLOR SPECTRUMS FOR UNTREATED AND TREATED SAMPLES

	CIE LAB					
Samples	L*	a*	b*	ΔE	Yellowness index (ASTM D1925)	Brightness index (Stensby)
Untreated	93.098	0.325	1.649	-	4.124	84.365
PSO-CD-treated	91.617	0.379	2.598	1.8	6.198	80.576
W-PSO-CDtreated	92.953	0.018	0.886	2.5	0.634	93.649

4 Conclusion

In this study encapsulation of PSO by beta-CD was carried out and inclusion complexes were formed successfully. The formed inclusion compound was applied onto the textile substrate by pad-dry method and both formulation and surface morphology of the substrates were discussed in detail. According to the SEM results, untreated fabric showed a smooth surface with no superficial particle/droplet whereas droplets deposited on the textile fibers were observed on the fabrics treated PSO-beta-CD inclusion compounds. According to multiple washing tests, it could be mentioned that this procedure was not durable enough, hence usage of crosslinking agent was considered to be used in further encapsulation studies. Color spectrum test showed that no significant difference in ΔE values was indicated between untreated and treated fabrics. There was a decrease in yellowness indexes of 5 times washed sample when compared to untreated sample.

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