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ENCAPSULATION OF *HYPERICUM PERFORATUM* L., JOJOBA OIL AND JASMINE OIL BY SPRAY DRYING AND THEIR APPLICATIONS IN TEXTILES

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Abstract: *Microencapsulation is a technique that allows liquid or solid agents, such as pharmaceutical agents, pesticides, insect repellent agents, dyes, cosmetics and fragrances, to be encapsulated by a suitable barrier wall. Liquid or solid agents that are encapsulated are called core material. The importance of functional finishes have been increasing rapidly in the World. Microencapsulation is an alternative way to achieve the functional finishes because of their unique properties, such as controlled release, protection against to hazardous and destructive media, and providing higher surface area. In this study, jojoba, jasmine and St. John's Wort oils, were encapsulated according to spray drying method. St. John's Wort and jojoba oils were used at 1:1 ratio as fixed oil. Jasmine essential oil was added to fixed oil mixture at two different ratios. After preparing the core mixture, encapsulation studies were performed three different core : wall ratios. Morphological analyses of microcapsules were carried out using SEM (Scanning Electron Microscope). FTIR spectroscopy spectrums of microcapsules were determined (Fourier Transform Infrared). Particle size distribution microcapsules were analyzed by laser scattering measurement method. DSC (Differential Scanning Calorimetry) thermographs of microcapsules were obtained. All microcapsules were applied to 100% cotton knitted fabrics. Strength to washing of fabrics were observed by SEM micrographs.*

Key words: *encapsulation, aromatherapy, Hypericum Perforatum L., jojoba oil, jasmine oil*

1. INTRODUCTION

Capsulation technology seems unbeatable in conferring functional properties to textile products, especially prompted controlled release. Active substances that have different properties can be encapsulated and applied to the fabric so that many functions are imparted to the fabric. “Cosmetic textiles”, has now opened up new opportunities and markets in the textile industry, especially in sport and leisure wears [1].

Essential oils assist in the protection of plants from disease, parasites and changes in climatic conditions, and are extracted from various parts of many different plants to be utilised in the food and perfumery industries and in aromatherapy [2]. Aromatherapy is a science and an art in which essential oils, derived from herbs, flowers, and other plants, are used for health, well-being, and medical treatment [3].

Hypericum perforatum L. (St. John's Wort) is a perennial herb that is commonly known as St. John’s Wort, which contains phloroglucinol derivatives (hyperforin, adhiperforin),

naftodiantrones (hypericin, pseudohypericin, protohypericin, protopseudohypericin), phenolic acids and a large number of flavonoids such as rutin and quercetin [4], [5].

Jojoba oil, which is obtained from seeds of jojoba plant, is a non-irritating and non-comedogenic product that is used as a moisturizer in many skin care products [6], [7].

Jasminum grandiflorum L. (=*Jasminum officinale* L. *var. grandiflorum* (L.) Kobuski) is widely consumed as infusion, due to its pleasant taste, and has therapeutic properties against psychiatric disorders and other illnesses [8].

In this study, we aimed to production of microcapsules with three different effects including skin care, pleasant fragrances and antibacterial effect for sports and leisure clothing and then application of that microcapsules to the textile product. For this purpose, jojoba oil was used for skin care, jasmine essential oil was used for pleasant fragrance peculiarities and St. John's Wort oil was used for antibacterial properties.

2. EXPERIMENTAL

2.1 Materials

St. John's Wort oil was kindly supplied by Mecitefendi (Turkey). Ethyl cellulose (100 cp), ethyl acetate, essential oils and other materials were purchased from Sigma-Aldrich. Scoured, bleached 100% cotton knitted fabric was kindly supplied by UNITEKS (unit weight 150 g/m^2, 21 courses/cm and 16 wales/cm). N-methylol dihydroxy ethylene urea was used as crosslinking agent (Rucon Fas, Rudolf-Duraner, Turkey)

2.2 Method

2.2.1 Preparation of Microcapsules

St. John's Wort oil and jojoba oil was used 1:1 ratio as fixed oil and core wall : essential oil ratios were given at Table 1. Microcapsules were produced by using spray drying method at the LabPlant spray-drying apparatus and the inlet temperature was 100 ^oC, exhaust temperature was 90 ^oC. Wall material : solvent ratio was selected 2%. Active ingredients were weighed at specified ratios and 250 ml solvent added. Ethyl cellulose was slowly added while the solution was mixed with a magnetic stirrer. After the ethyl cellulose had completely dissolved, the solution was fed to the spray dryer apparatus.

Table 1: *Microcapsule ratios*

Microcapsules	Oil:Wall Material Ratio			Jasmine Essential Oil:Fixed Oil Ratio	
	1:2	1:3	1:4	1:3	1:5
Y1			√		√
Y2			√	√	
Y3		√			√
Y4		√		√	
Y5	√				√
Y6	√			√	

2.2.2 Application To Textiles

Microcapsules (20 g/L) were applied to 100% cotton knitted fabrics using a 60 g/L suitable crosslinking agent (Rucon Fas-Rudolf Duraner) according to impregnating method (90% pickup). Drying and curing (3 min.) was carried out at 110 and 150 ^oC respectively.

2.2.3 Washing of Fabrics

Washing of microcapsule impregnated fabrics were performed by using Atlas Linitest apparatus according to TS EN ISO 105- C06 A1S test standart.

2.2.4 Analyses

SEM micrographs were done at FEI QUANTA 250 FEG. Before SEM analysis, the samples were covered with gold. FTIR analyses were performed at Elmer Spectrum BX device in the range of 650-4000 cm^{-1}. Resolutions were selected 4 cm^{-1}, while the number of scanning is 20. DSC results were obtained using Pyris Diamond device (Perkin Elmer). Particle size analysis studies were performed at Haribo Partica LA-950 V2 device with by laser scattering measurement method. 3% Tween 20 was used for dispersed particles uniformly in water.

3. RESULTS

3.1 Morphology of Microcapsules

SEM micrographs of morphology of microcapsules are shown in Figure 1. As can be seen in figure, some of the microcapsules have spherical shape with smooth surfaces, however some of them in the shrinked form. This result is compitable with the literature and can be attrubitued to rapid evaporation of ethyl acetate from core of the microcapsules [9].

3.2 SEM Analysis of Microcapsule Impregnated Fabrics

Figure 2 depicts microcapsules on cotton fabric. Microcapsules successfully applied to cotton fabrics. Figure 3 shows the cotton fabrics after 1 washing. Microcapsules were onto cotton fibers after 1 washing.

3.3 FTIR Analysis

Figure 4 depicts the FTIR results of jasmine oil, jojoba oil, St. John's Wort oil and microcapsules. When the FTIR analysis results are examined, the peaks were seen at 2853-2858 cm^{-1} due to asymmetrical and symmetrical stretching vibration of methylene (—CH2) group. Peaks of ester carbonyl functional group of the triglycerides were observed around 1740 cm^{-1}. A peak of bending vibrations of the CH_2 and CH_3 aliphatic groups around 1465 cm^{-1} was only observed in the case of pure oils. After encapsulation of them, that peak was disappeared. A board peak between 1000-1150 cm^{-1} was observed due to nature of cellulose (C-O-C asymmetrical stretching, C-C, C-OH, C-H ring vibrations) [10]. All characteristic peaks of oils were observed at spectrum of all microcapsules. This result indicates that all microcapsules contain active ingredients.

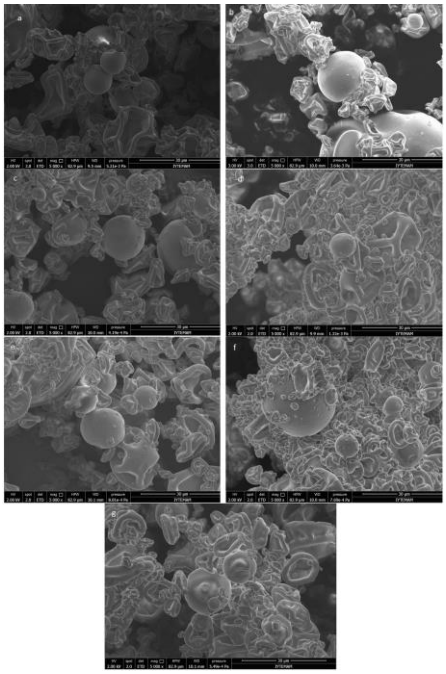

Fig. 1*: SEM Micrographs of Microcapsules* ***a.*** *Blank Microcapsules* ***b****. Y1* ***c.****Y2* ***d.*** *Y3* ***e.*** *Y4* ***f****. Y5* ***g****.Y6*

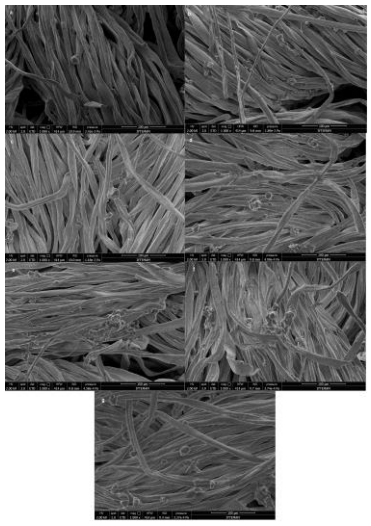

Fig. 2*: SEM Micrographs of Microcapsule Impregnated Fabrics Before Washing* ***a.*** *Blank Microcapsules* ***b****. Y1* ***c.*** *Y2* ***d.*** *Y3* ***e.*** *Y4* ***f****. Y5* ***g****. Y6*

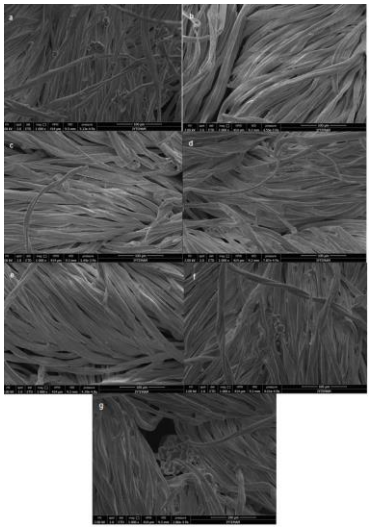

Fig. 3*: SEM Micrographs of Microcapsule Impregnated Fabrics After One Washing* ***a.*** *Blank Microcapsules* ***b****. Y1* ***c.*** *Y2* ***d.*** *Y3* ***e.*** *Y4* ***f****. Y5* ***g.*** *Y6*

3.4 DSC Analysis

Figure 5 shows DSC thermographs of microcapsules. When the DSC analysis results are examined, there is no peak of enthalpy change. It can be inferred that the capsules have completely confined the active ingredients, thus concealing the thermal behavior.

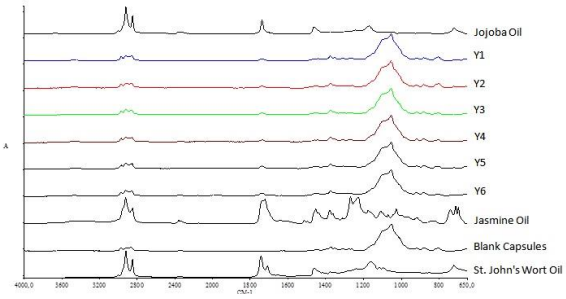

Fig. 4: *FTIR spectrums of jasmine oil, jojoba oil, St. John's Wort oil and microcapsules*

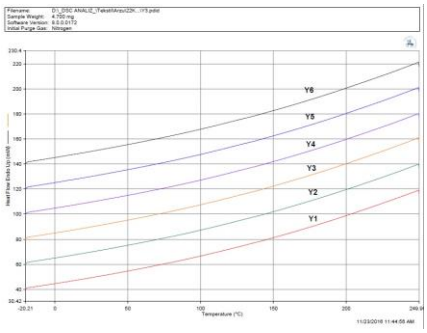

Fig. 5: *The graph results of the DSC analysis*

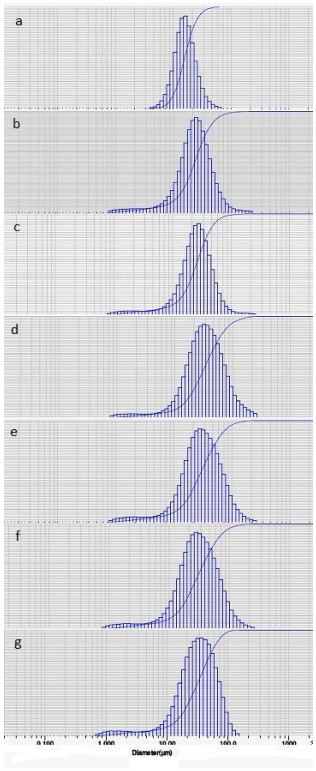

Fig. 6*: Particle Size Distribution Graphics of Microcapsules*
a. *Blank Microcapsules* ***b.*** *Y1* ***c.*** *Y2* ***d.*** *Y3* ***e.*** *Y4* ***f.*** *Y5* ***g.*** *Y6*

3.5 Particle Size Analysis

According to the particle size analysis, the mean size of blank capsules was approximatley 17 µm and the mean size of capsules containing oil mixture were between 27-43 µm (Figure 6). Thus, it can be concluded that the particle size distrubitions of microcapsules were enough for textile applications.

3. CONCLUSIONS

In this work, mixture of Jojoba oil, St. John's Wort oil and jasmine oil were microencapsulated by spray drying apparatus using ethyl cellulose. Three different core : wall ratios were applied. The results show that oil mixtures were encapsulated by ethyl cellulose and obtained microcapsules had appropirate particle sizes to apply the textiles. Obtained microcapsules were applied to the 100% cotton knitted fabrics. Further studies are antimicrobial testing, GC and HPLC analyses of applied fabrics. Washing resistance also will be investigated against to 5 and 10 washing cycles.

4. ACKNOWLEDGMENTS

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REFERENCES

[1] M. Sarıışık, S. Okur and Ş. Asma, "*Odor Adsorption Kinetics on Modified Textile Materials Using Quartz Microbalance Technique"*, Acta Physica Polonica A, vol. 121, no: 1, pp. 243-246, 2012.

[2] D. Tiran, "*Aromatherapy in midwifery: benefits and risks"*, Complementary Therapies in Nursing & Midwifery, vol. 2, issue 4, pp. 88-92, 1996.

[3] M. S. Lee, J. Choi, P. Posadzki and E. Ernst, "*Aromatherapy for health care: An overview of systematic reviews"*, Maturitas, vol. 71, issue 3, pp. 257-260, 2012.

[4] Z. Saddiqe, I. Naeem and A. Maimoona, "*A review of the antibacterial activity of Hypericum perforatum L.*", Journal of Ethnopharmacology, vol. 131, issue 3, pp. 511-521, 2010.

[5] N. Kalogeropoulos, K. Yannakopoulou, A. Gioxari, A. Chiou and D. P. Makris, "*Polyphenol characterization and encapsulation in beta-cyclodextrin of a flavonoid-rich Hypericum perforatum (St John's wort) extract.*" , LWT - Food Science and Technology, vol. 43, issue 6, pp. 882-889, 2010.

[6] Y. L. Dréau, N. Dupuy, V. Gaydou, J. Joachim, and J. Kister, "*Study Of Jojoba Oil Aging By Ftır"*, Analytica Chimica Acta, vol. issues 1-2, 642, pp. 163–170, 2009.

[7] R. R. Habashy, A. B. Abdel-Naim, A. E. Khalifa and M. M. Al-Azizi, "*Anti-inflammatory effects of jojoba liquid wax in experimental models"*, Pharmacological Research, vol 51, issue 2, pp. 95–105, 2005.

[8] F. Ferreres, C. Grosso, A. Gil-Izquierdo, P. Valentão and P. B. Andrade, "*Assessing Jasminum grandiflorum L. authenticity by HPLC-DAD-ESI/MSn and effects on physiological enzymes and oxidative species"*, Journal of Pharmaceutical and Biomedical Analysis, vol. 88, pp. 157–161, 2014.

[9] A. López, S. Castro, M. J. Andina, X. Ures, B. Munguía, J. M. Llabot, H. Elder, E. Dellacassa, S. Palma and L. Domínguez, "*Insecticidal Activity of Microencapsulated Schinus Molle Essential Oil*", Industrial Crops and Products, vol. 53, pp. 209–216, 2014.

[10] A. Rohman and Y. B. Che Man, "*Fourier transform infrared (FTIR) spectroscopy for analysis of extra virgin olive oil adulterated with palm oil*", Food Research International, vol. 43, issue 3, pp. 886-892, April 2010.