ANNALS OF THE UNIVERSITY OF ORADEA
FASCICLE OF TEXTILES, LEATHERWORK

MICROENCAPSULATION OF BIO-DEGRADABLE PCM USING COCONUT OIL AND ETHYL CELULOSE

DIRLIK UYSAL Çagla Dilara, BOU-BELDA Eva, BONET-ARACIL Maria Ángeles, GISBERT-PAYÁ Jaime, DÍAZ-GARCÍA Pablo

[1] Universitat Politècnica de València, Textile and Paper Department, Ferrándiz y Carbonell s/n, 03801, Alcoy, Spain.

Corresponding author: Bonet, MA, maboar@txp.upv.es

Abstract: *Over the few past decades, preparation of uniform microparticles has received a great interest in the textile area to add new functionalities to the textile fabric. Amongst them, thermal insulation textile and more specially microencapsulation of phase change materials (PCMs) have attracted more and more attention in recent years. In an latent heat thermal energy storage system, energy is stored during the melting, and recovered during the freezing, of phase change material (PCM). It plays an important role in solving energy imbalance, by improving thermal efficiency and protecting the environment. The aim of this study is obtaining bio-degradable PCM which shell is Ethly cellulose and active agent is coconut oil by using solvent evaporation as a microencansulation method. In order to microencapsulation with solvent evaporation method: we have prepared 4 different combinations of emulsions by changing the amount of ethyl cellulose and coconut oil. We examined the samples obtained by scanning electron microscope (SEM) to see how the ratio of materials used as active agent and shell play a role in microcapsule formation. SEM analysis showed that we obtained significative results when we use ethyl cellulose and coconut oil in a ratio of 1: 1, we obtain the spherical microcapsules.*

Key words: *Microencapsulation; Phase Change Material; Functional Textiles; Coconut oil; SEM.*

1. INTRODUCTION

Over the few past decades, preparation of uniform microparticles has received a great interest in the textile area to add new functionalities to the textile fabric. Amongst them, thermal insulation textile and more specially microencapsulation of phase change materials (PCMs) have attracted more and more attention in recent years[1]. Beside textile, latent heat thermal energy storage systems have been applied in many fields, such as environmental building materials, solar energy systems, central air conditioning systems and industrial waste heat recovery, due to their high energy storage density and small temperature variation from storage to retrieval. In an latent heat thermal energy storage system, energy is stored during the melting, and recovered during the freezing, of phase change material (PCM). It plays an important role in solving energy imbalance, by improving thermal efficiency and protecting the environment [2]. PCMs can be categorized into two major groups: inorganic compounds and organic compounds. Also, organic PCMs are classified paraffinic PCMs and non-paraffinic. Compared to paraffinic PCMs, non-paraffinic PCMs are significantly less flammable[2]. For this reason and also ecological reasons, we decided to use non-paraffinic PCMs, namely bio-based PCMs which contained various fatty acids.

Bio-originated PCMs such as soybean oils, coconut oils, palm oils, and beef tallow [2] have high latent heat of fusion, good thermal stability, and no toxicity, similarly to paraffin, and are also suitable for microencapsulation. However, most paraffins are flammable, while bio-originated PCMs have considerably higher ignition resistance. Since bio-based PCMs are fully hydrogenated, they are not sensitive to oxidation [3]. Their melting point can be adjusted in a wide temperature range, from −23 °C up to 78 °C, hence they can be suited to various application fields in various climatic condition. They possess low vapor pressure, self-nucleating behavior, safety, and commercial availability at low cost [4].

Microcapsules production can be achieved by means of physical or chemical techniques. One of them was based on solvent extraction/evaporation allowing the preparation of a wide range of microspherical and microcapsular products [5]. Cellulose derivatives and more specially cellulose acetate buty-rate are ideally suitable for the preparation of microparticles by solvent evaporation[1]. Solvent extraction/evaporation neither requires elevated temperatures nor phase separation-inducing agents. Controlled particle sizes in the nano to micrometer range can be achieved, but careful selection of encapsulation conditions and materials is needed to yield high encapsulation efficiencies and a low residual solvent content [6].

2. EXPERIMENTAL

The preparation of the microcapsules was carried out by using following materials and method:

Coconut oil used as active agent of Mcs, ETYL CELLULOSE (Viscosity of a 5%solution in toluene: ethanol (4:1) at 25C Approx. 45cps. Ethoxy content 49.0%) used as shell of microcapsules and ethyl acetate used as solvent. 200mg/100mg liquid coconut oil, 200ml/400ml ethyl cellulose, 10mg ethyl acetate, 20ml 1% polyvinyl alcohol in total 4 different combination of solution have been used which are shown in the table 1. Each solution combinations was emulsified at 900 rpm with a homogenizer at room temperature during 150 minutes. A drom of sample has taken during mixing in each 15 minutes till 150th minute.

***Table 1**: Formulations of coconut microcapsules.*

REFERENCE	POLYMER	SOLVENT	OIL	WATER	CONDUTIONS		
	ETHYL CELULOSE (mg)	**ETHYL ACETATE (mg)**	**COCONUT (mg)**	**1% PVA**	**TIME**	**T**	***rpm**
CO-EC 01	200	10	200	20 mL	150’	ROOM	900
CO-EC 02	400	10	200	20 mL	150’	ROOM	900
CO-EC 03	200	10	100	20 mL	150’	ROOM	900
CO-EC 04	400	10	100	20 mL	150’	ROOM	900

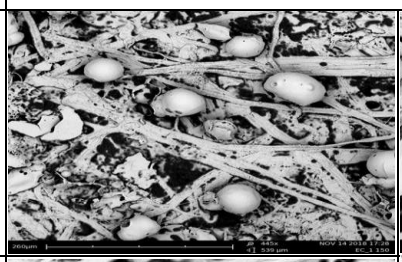
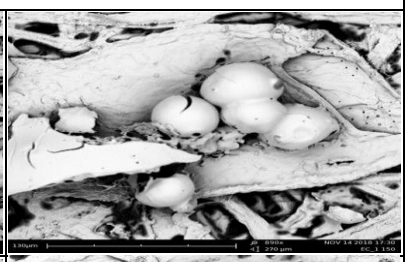
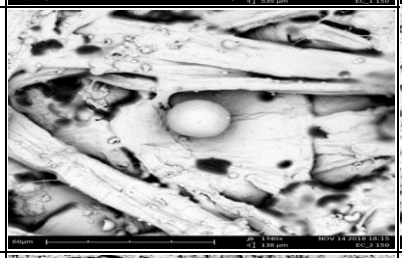
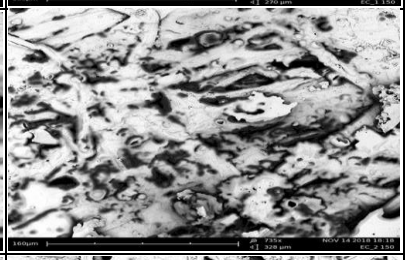
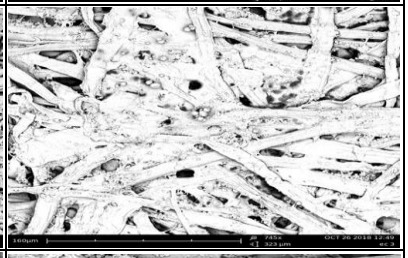
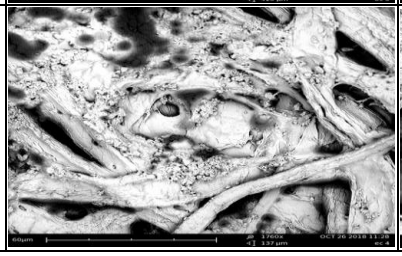
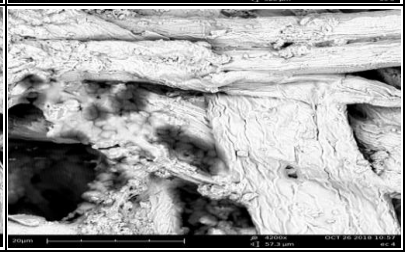
In order to characterize the microcapsules samples the Scanning electronic microscope (SEM), FEI model Phenom (Fei, Oregon, USA).

To obtain biodegradable PCM, solvent evaporation method was used in microencapsulation. Microsphere preparation by solvent extraction/evaporation basically consists of four major steps: (i) dissolution or dispersion of the bioactive compound often in an organic solvent containing the matrix forming material; (ii) emulsification of this organic phase in a second continuous (frequently aqueous) phase immiscible with the first one; (iii) extraction of the solvent from the dispersed phase by the continuous phase, which is optionally accompanied by solvent evaporation, either one transforming the droplets into solid microspheres; (iv) harvesting and drying of the microspheres [6].

3. RESULTS

A drope of sample of each formulation was analyzed by SEM to see if microcapsules were obtained or not. In below table 2 each 4 samples SEM images have been shown.

Table. 2*: SEM images of microcapsules.*

REFERENCE	SEM IMAGES	
CO-EC 01		
CO-EC 02		
CO-EC 03		
CO-EC 04		

According to SEM images, we obtained the best result in the sample CO-EC 01 which we used 200mg Ethyl cellulose and 200 mg coconut oil. In CO-EC 02 we increased Ethyl cellulose amount, which is 400mg, although we obtained some microcapsules as it is shown in the image on left, in the image on right the photo is taken increasing the magnification and it seems that ethyl cellulose rate is high due to the excess of polymer. In CO-EC 03 we decreased coconut oil amount which is 100mg and it seems that this quantity is not enough to obtain microcapsules. In CO-EC 04 it was increased ethyl cellulose amount using 100mg and decreased coconut oil amount as 100mg and in SEM images we can see clearly that the ethyl cellulose amount is too high and we have not got enough coconut oil to establish regular microcapsules.

4. CONCLUSIONS

In order to microencapsulation with solvent evaporation method: we have prepared 4 different combinations of emulsions by changing the amount of ethyl cellulose and coconut oil. We examined the samples obtained by SEM to see how the ratio of materials used for active agent and shell play a role in microcapsule formation.

SEM analysis shown that we have obtained significative results that when we use ethyl cellulose and coconut oil in a ratio of 1: 1, we obtain spherical microcapsules.

REFERENCES

[1] Fabien Salaün, Eric Devauxa, Serge Bourbigot, Pascal Rumeau, *"Influence of the solvent on the microencapsulation of an hydrated salt"* Carbohydrate Polymers, vol. 79(4), 2010, pp. 964-974.

[2] Su-Gwang Jeong Okyoung Chung Seulgi Yu Sughwan Kim Sumin Kim, *"Improvement of the thermal properties of Bio-based PCM using exfoliated graphite nanoplatelets"*. Solar Energy Materials and Solar Cells, vol. 117, 2013, pp. 87-92.

[3] Bence Németha,b, Ágnes S. Németha, *"Fully bio-originated latent heat storing calcium alginate microcapsules with T high coconut oil loading"* Solar Energy 170, 2018, pp. 314–322.

[4] Yujin Kang, Su-Gwang Jeong, SeunghwanWi, SuminKim, *"Energy efficient Bio-based PCM with silica fume composites to apply in concrete for energy saving in buildings"* Solar Energy Materials and Solar Cells, vol.143, 2015, pp. 430-434.

[5] R. Arshady. *"Microspheres and microcapsules, a survey of manufacturin techniques: Part III: Solvent evaporation"* Polymer Engineering and Science, 30 (15), 1990, pp. 915-924.

[6] Sergio Freitas Hans, P. Merkle Bruno Gander, *"Microencapsulation by solvent extraction/evaporation: reviewing the state of the art of microsphere preparation process technology"* Journal of Controlled Release, 102 (2), 2005, pp. 313-332.