ENCAPSULATION LAVENDER OIL BY SPRAY DRYING FOR SPORTSWEAR

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Abstract: Microencapsulation technology has been increasingly used in textile industry due to give some peculiarities such as controlled released etc. In this study we aim to make aromatherapy sportswear using lavender oil. Lavender oil is encapsulated by different wall materials. Microcapsules were obtained by spray drying method. Differential scanning calorimetry, thermal gravimetric analysis, Fourier Transform infrared spectroscopy, morphological analyses were applied to microcapsules. Optimum microcapsules were applied to different fabrics. Fastness to washing and rubbing, SEM and antibacterial tests were carried out.

Keywords: aromatherapy, lavender oil, microencapsulation, spray drying, textile finishing

1. INTRODUCTION

Microencapsulation is a technique used to protect active substances from external influences by coating them in small droplets. This technology has preferably been used in the pharmaceutical, chemical and food industries to preserve and prolong fragrance, flavour or therapeutic effect [1,2]. Microencapsulation applications have succeeded in becoming one of the products that add value in the textile industry, create a competitive environment and difference, and increase its market share. In this study, it is aimed to obtain essential oil from *Lavandula angustifolia* plant, which has a pleasant smell and known antibacterial effect [3], and to use it in textile products. In this way, while the effect of this product, which has a limited lifespan, will be increased, and alternative sports and leisure clothes will be developed.

2. MATERIALS AND METHODS

In this study, knitted fabric from different natural and synthetic fibres was used. Gelatin (Gel), gum arabic (GA) and ethyl cellulose (EC) was used as shell material. Gel

was kindly donated from Sel Gel. Sigma-Aldrich branded GA and EC was employed. Lavender oil used as core material was obtained from by Doğal Destek A.Ş. (Tabia). Tween 80 was used as a surface-active agent and obtained from Merck. Tanatex Nano PU was employed as a binder. All other auxiliary chemicals used in the study are technical grade.

Plants grown in the Southern and Central Aegean were harvested and lavender essential oil was obtained by using the water vapour distillation method. The composition of the essential oil was determined by gas chromatography-mass spectrometry (GC-MS). Microcapsules were obtained in different shells and core ratios using selected polymers by spray-drying method. The solutions prepared at the different concentrations were sprayed from the 0.5 mm nozzle into the spray dryer cabinet. The formulation of lavender oil capsules are given in table 1.

Table 1. Formulation of Lavender oil microcapsules

Formulation	Polymer	Lavender oil (%)	Pump speed (ml/min)	Inlet temperature (°C)	Outlet temperature (°C)	Yield (%)
L1	5% Gel	4	10	140	90	34.2
L2		6	5	140	90	31.9
L3		8	5	140	90	32.8
L4	3% Gel	5	2.5	125	85	37.7
L5		10	2.5	125	85	26.6
L6	2.1% Gel	0.5	2.5	125	85	39.1
L7*	1.25% Gel 0.85% GA	0.5	2	125	85	61.1
L8	3% EC	3	2.5	115	85	63.0
L9		6	2.5	115	85	72.4
L10		9	2.5	115	85	78.1

The compressor and air circulation speed were operated at maximum. The experiments were carried out in a Lab Plant brand SD-Basic spray drying device with a main cabinet size of 380 mm x 110 mm. The production yield of the lavender microcapsules was calculated according to the following equation:

$$(\%)Yield = \frac{Actual\ capsule\ amount\ (g)}{Theoretical\ capsule\ amount\ (g)} \times 100 \quad (1)$$

Morphology of the microcapsules is a good indicator for ideal capsule formation. Therefore, scanning electron microscopy (SEM) micrographs were taken. The chemical properties of microcapsules were examined by Fourier transformed infrared (FT-IR) spectrometry. The thermal resistance of microcapsules and behaviour of the essential oil were investigated through thermogravimetric analysis and differential thermal analysis system (DT/TGA). The optimum microcapsule formulation was applied to the different knitted fabrics used in sports and leisure garments according to the exhaustion method. The fabrics were examined using SEM micrographs for the presence of capsules on the fabrics and the determination of the effect of domestic washing and rubbing. The effect of microcapsule finishing on colour was determined. The antibacterial effect of the microcapsule treated fabrics was studied according to AATCC 147 standard. Permeability properties were evaluated before and after microcapsule application regarding air permeability and water vapour permeability tests using ISO 9237 and BS 7209 standards respectively.

3. RESULTS AND DISCUSSIONS

The composition of the lavender oil was revealed as 39.2% linalool and 34.7% linalyl acetate. The other active agents found in the essential oil were camphor, 1,8-Cineole, β -ocimene, β -caryophyllene and borneol. Photomicrographss of gelatine, gelatine-gum arabic and ethyl cellulose micro particles formed using lavender oil are given in Figure 1. When the images of micro particles with 5% gelatine concentration were examined (L1-L3), collapses were observed in the capsule structure. It was thought that this could be due to the relatively high temperature required to dry solutions with high polymer concentrations. The drying effect at lower temperature, provided by using a low polymer concentration, resulted in an improvement in the capsule morphology (L4-L6). A lower aggregation tendency was observed in L7 coded micro particles prepared using a combination of gelatine and gum arabic polymer. When the images of the L8, L9 and L10 samples, which were created using ethyl cellulose, were examined, it was determined that the micro particles obtained were small, spherical and had a smooth surface compared to other formulations. Microcapsules with ethyl cellulose walls were less likely to agglomerate, and the capsules were found to be more rounded. The production yield of the optimum formulation was calculated as 72.4%, which has one of the highest yield.

Figure 1. SEM Micrograph of the optimum microcapsule formulation with lavender oil

When the spectrum of lavender oil was examined, the broad band of the OH groups was seen in the range of $3600\text{--}3200\text{ cm}^{-1}$, and the peak of the CH_3 groups was around 2900 cm^{-1} . The characteristic molecular groups of COOR and C=O and the peak of C=O stretching at 1735 cm^{-1} were determined. Both lavender and ethyl cellulose peaks were found in lavender microcapsules (figure 2).

Figure 2. FT-IR Spectra of EC-Lavender capsules

TGA thermograms and DSC diagrams of ethyl cellulose, lavender oil and microcapsules formed with the oil are given in figure 3.

Figure 3. TGA thermograms and DSC diagrams of lavender oil and EC micro particles

When the TGA graph of ethyl cellulose is examined, a weight loss was observed at 100°C due to the moisture in the material. Pyrolysis starts around 225°C and accelerates at 300°C. It is known that the degradation temperature of ethyl cellulose is around 440°C [4]. When the graphs of lavender oil are examined, it is seen that the weight loss starts around 35°C and degrades around 125°C. A wide endothermic peak was observed in the range of 30-120°C in the DSC curve of lavender oil. TGA curves similar to ethyl cellulose were obtained when this oil, which evaporated at low temperatures, was confined to the ethyl cellulose shell. The mass loss at 150°C, where lavender oil is completely decomposed, is around 5% in L8, L9 and L10 formulations.

SEM images of knitted fabrics with L9 formulation before washing, after 1 and 10 washings are shown in figure 4. It was determined that the micro particles were successfully transferred to all the fabrics. It has been observed that the micro particles on the fabric and in the fibre spaces are especially concentrated in the areas covered with binder. A low rate of removal of micro particles from the fabric surface after washing indicates that permanent bonding based on 10 washings is achieved.

Figure 4. Washing resistance of fabrics containing lavender oil microcapsules

Another of the highest mechanical effects that leisure clothing faces is rubbing. It is important that the micro particles are permanently attached to the fabric exposed to friction. The images of the samples taken after the rub test from the lavender microcapsules applied fabrics are presented in figure 5. It was determined that the micro particles remained on the fabric after rubbing.

Antibacterial assay results on fabrics containing lavender oil capsules are given in table 2. Gram-negative and gram-positive bacterial types were heavily grown for the control samples. Zone of inhibition was not observed against both *E. coli* and *S. aureus* bacteria in lavender-containing fabrics and lower bacterial growth was observed on the contact surface.

Figure 5. Rubbing resistance of fabrics containing lavender oil microcapsules

Table 2. Antibacterial analysis results of fabrics containing lavender microcapsules

Sample	Gram Negative Bacteria		Gram Positive Bacteria	
	<i>E.coli</i>		<i>S.aureus</i>	
	Inhibition Zone	Growth on the Surface	Inhibition Zone	Growth on the Surface
Control	Not observed	High bacterial growth	Not observed	High bacterial growth
Microcapsule Treated Fabric	Not observed	Medium bacterial growth	Not observed	Medium bacterial growth

Permeability properties of the cotton fabrics were determined before and after microcapsule application. Accordingly, capsule application was decreased the permeability of the fabrics due to blocking the spaces. Theoretically, binder and microcapsule applications are expected to reduce the permeability of fabrics as they block the pores in the fabric. While pristine cotton fabrics have 298.3 mm/s air permeability and 904.7 g/m²/day water vapour permeability, lavender capsule applied fabrics reveal 292.7 mm/s and 813.5 g/m²/day, respectively. However, there was no statistically significant decrease in both air and water permeability results (figure 6).

Figure 6. Permeability properties of fabrics containing lavender oil microcapsules

4. CONCLUSION

The aim of this study is to develop sports and leisure clothes containing nano and micro sized capsules with lavender oil. In the manufacture of the capsules, polymers that skin-safe are used as shell polymers. An industrially applicable method, the spray drying, was chosen as the capsule production technique. Lavender oil was selected to provide both aromatherapy effect and odour control that may occur during workout. The developed products have potential for commercialization and suitable for industrial production.

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