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FUNCTIONALIZATION OF TEXTILE FABRICS WITH MICROENCAPSULATED VITAMIN E

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Abstract: *In this study the experimental deposition of vitamin E microcapsules by padding technique on the textile support made of 50% cotton and 50% polyamide high tenacity Nm 50/1 were performed. The preliminary preparation of textile materials has been made in four consecutive sequences: hot alkaline treatment in absence of NaOH, bleaching, drying and curing. In the pretreatment of textile materials the crosslinking agent Itobinder AG is used, which is an anionic emulsion based on the acrylic copolimer, being followed by the application of a dispersion with content of vitamin E microcapsules. In the present raport the evaluation of obtained performances was made through SEM, GC and FTIR-ATR analysis. By SEM has been determined the wash durability of deposition of vitamin E microcapsules before and after one washing cycle. Following qualitative analysis by Gas-Chromatography coupled with Mass Spectrometry and Fourier Transform Infrared Spectroscopy with Attenuated Total Reflection absorption the related compoundes presents on the surface of the textile materials were identified. After retention time the vitamin E acetate is found with preponderance in all chromatograms at 15, 70 min with aproximation. Also by FTIR-ATR the presence of vitamin E acetate is confirmed by the apparition of a new peak at 1731 cm^{-1} and changes of intensity of various peaks, especially in the fingerprint region of the spectra of the functionalized fabrics.*

Key words: *textiles, microcapsules, vitamin E acetate, FTIR-ATR, SEM, GC-MS*

1. INTRODUCTION

The study of vitamin E is relatively complex because it can be found in many forms. These forms are jointly called tocols and are classified in tocopherols and tocotrienols [1]. An alternative of using vitamin E is the vitamin E acetate which hydrolyze when meets the H_2O molecules on the human skin being converted to free vitamin E. Also, vitamin E can be absorbed through all the skin layers to the cell membrane offering healthy glow to the skin and speeding up regeneration [2], [3]. The vitamin E acetate is less prone to oxidative stress and more stable during the light and heat exposure, than free tocols [1]. It is known that the vitamin E is the most common lipophilic antioxidant meet into the structure of the human skin layers, reacting with the reactive oxygen

species to protect the skin [4], [5], [6]. Once with the degradation of the epidermal structure by the UV exposure, the concentration of the vitamin E decreases [2], [7], [8]. So one of the most met way to protect the skin from UV exposure is to wear protective clothes, clothes functionalised with vitamin E. In this study the deposition of vitamin E microcapsules by padding technique on the textile materials made of 50% cotton and 50% polyamide high tenacity Nm 50/1 were performed.

2. EXPERIMENTAL PART

2.1 Materials

Deposition of suspension with vitamin E microcapsules content (LJ Specialities, UK) was performed on fabrics with fiber composition of 50% cotton/50% polyamide high tenacity, Nm 50/1. Textile fabrics were pretreated with Itobinder AG, a crosslinking agent supplied from LJ Specialities (UK). Itosilicone LJ88 has been used as non-ionic agent (LJ Specialities, UK).

2.2. Preliminary preparation of textile materials

For ensuring a good hydrophilicity of the textile materials, these were preliminary prepared by hot alkaline treatment at 95ºC temperature, for 90 minutes, in absence of NaOH to avoid the unwanted deffects on the polyamide component from the mixture. After hot alkaline treatment, the fabrics were washed repeatedly at 80ºC, 60ºC, 40ºC and at room temperature for 10 minutes.

2.3. Pretreatment of textile materials with crosslinking agent

For fabrics pretreatment Itobinder AG has been used as a crosslinking agent, applied by padding technique. Itobinder AG is an anionic emulsion based on the acrylic copolimer with good properties of adhesion and lasting effect in the treatments of washing and chemical cleaning. The textile materials were padded with different concentration of Itobinder AG, dried at 120 ºC for 2 minutes, followed by curing at a temperature of 150 ºC for 1 minute.

2.4. Treatment of textile materials with vitamin E

For deposition and for fixation of vitamin E microcapsules on the surface of the pretreated textiles the next technological steps has been followed: impregnation, drying and curing. The pretreated textile materials were impregnated with the suspension which contains vitamin E microcapsules under the following conditions: 2 passes and 2 bar squeezing pressure, followed by drying of the impregnated material at a temperature of 120^{0}C for 2 minutes and curing of of 150 ºC for 1 minute. In the drying and curing proceses the drying/curing/heat-setting/vaporization**,** model TFO/S 500 mm (ROACHES, UK) was used.

Table 1*: Codification of experimental variants in order to deposit and fix Vitamin E microcapsules*

Variant Code	Bath composition of treatment by padding method	Drying		Curing	
		Time (min)	Temperature (ºC)	Time (min)	Temperature (ºC)
V_{3a}	Itofinish Vitamin E = 30 g/L Itobinder AG = 50 g/L	2	120	1	150
V_{3b}	Itofinish Vitamin E = 30 g/L Itobinder AG = 60 g/L	2	120	1	150
V_{3c}	Itofinish Vitamin E = 30 g/L Itobinder AG = 80 g/L	2	120	1	150
V_{3d}	Itofinish Vitamin E = 30 g/L Itobinder AG = 80 g/L Itosilicone LJ88 = 20 g/L	2	120	1	150

The codification of experimental variants carried out in order to deposit and to fix the vitamin E microcapsules on the textile materials is shown in the Table 1.

2.5. Methods

2.5.1. FT-IR Spectroscopy – Attenuated Total Reflection

FT-IR measurements were performed using Bruker Vertex 70 spectrophotometer and OPUS software. The ATR spectra were measured in the wave number interval 400 – 4000 cm^{-1}, using 64 scans and 4 cm^{-1} resolution.

2.5.2. Gas-Chromatography (GC)

For the identification of vitamin E derivative compound, GC was carried out with an Agilent 6890N Gas-Chromatograph coupled with 5973N Mass Spectrometer. The column was a DB-35MS J&W Scientific (35m x 0.25 mm i.d., 0.25 µm film thickness). As carrier helium gas has been used with a flow rate of 1.2 mL / min constant volume and a column headpressure of 17.83 psi.

The sample of 2 g of textile material was immersed in 100 mL hexane, stirred using VWR Mini-Shaker device. After stirring, the solution was filtered using PTFE 0.45 µm and injected using the autosampler. The injection volume was 1.0 µL. Injection temperature was 290 °C, the oven temperature was started from 180 °C with an increase of 15°C/min to 280°C ending with a 10 min of isothermal at 280°C and the auxiliar temperature was 300°C.

The identification was made using NIST RESEARCH LIBRARY. The detector was a Mass Spectrometer. MS parameters were: 70 eV with a scan interval in the 30-500 mass units. The temperature for MS Source was 230 °C and for the MS Quadrupole was 150°C.

2.5.3. Scanning Electron Microscopy

For study the distribution of Vitamin E microcapsules on the fabrics surface the Quanta 200 (FEI, Netherlands) electron microscope with X-EDS module integrated was used.

3. RESULTS AND DISCUSSIONS

3.1. FT-IR Spectroscopy – Attenuated Total Reflection

ATR spectra of the fabrics before and after functionalization treatment are shown in the Figure 1. Typical cellulose and amide bands are easily identified at 1029, 1106 and 1632 cm^{-1} respectively [9, 10]. A new band at 1731 cm^{-1} and several changes of peaks intensity in the fingerprint region are observed after functionalisation, confirming the presence of vitamin E microcapsules.

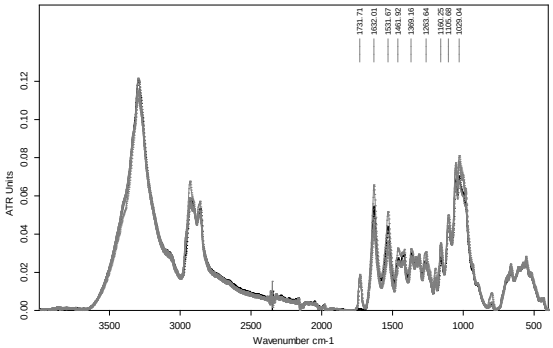

Fig. 1: *ATR spectra of reference (with black) and functionalized (with gray) fabrics*

3.2. Gas-Chromatography coupled with Mass Spectrometry

GC analysis of textile hexane extract of vitamin E is shown in the Figure 2 and Figure 3.

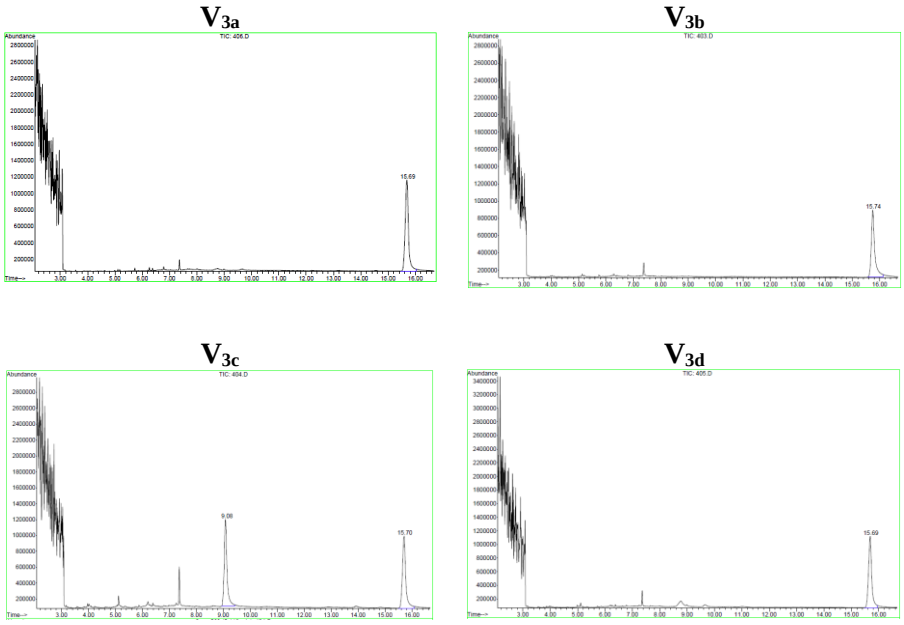

***Fig. 2**: GC-MS chromatogram for hexane extract of vitamin E*

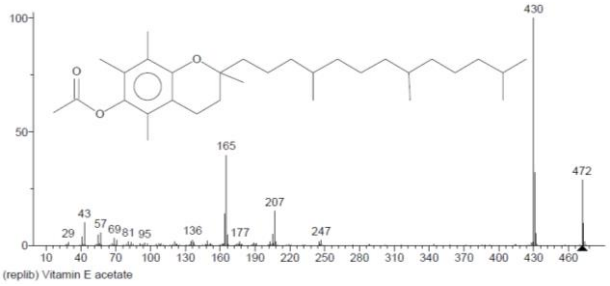

***Fig. 3**: Characteristic mass spectrum of vitamin E acetate*

In all chromatograms, at the beginning peak is attributed to the hexane solvent. Peak allure is given by the detector saturation with solvent. The appereance of several peaks is attributed to isomers existence of vitamin E like vitamin E acetate. According to the method used, vitamin E acetate has been identified at an approximate retention time of 15,70 min.

***Table 2**: Result based on MS and NIST identification*

Sample no.	Chemical constituents	RT	Peak area	MW	MF
V_{3a}	Vitamin E acetate	15,69 min	9374335	472,74	$C_{31}H_{52}O_3$
V_{3b}	Vitamin E acetate	15,74 min	6848088	472,74	$C_{31}H_{52}O_3$
V_{3c}	Vitamin E acetate	9,08 min	7844820	472,74	$C_{31}H_{52}O_3$
	Vitamin E acetate	15,70 min	7819407	472,74	$C_{31}H_{52}O_3$
V_{3d}	Vitamin E acetate	15,69 min	8427270	472,74	$C_{31}H_{52}O_3$

3.3. Scanning Electron Microscopy

In the Figure 4 is presented the distribution of vitamin E microcapsules on the surface of textile materials before and after washing process, the images were obtained at a magnification of x 4000. Electronic images recorded for textile materials treated with vitamin E reveal a predominant interfibrillar distribution of microcapsules. These are presenting spherical shape of different sizes and the majority are found in unbroken forme after the curing step, which demonstrate that the shell of the microcapsule is resistent at high temperature values. In the case of treatment variants with a higher concentration of binder (80 g/L) it is observed a deposition of some smaller microcapsules. After one washing cycle the number of vitamin E microcapsules from textiles surface is diminished.

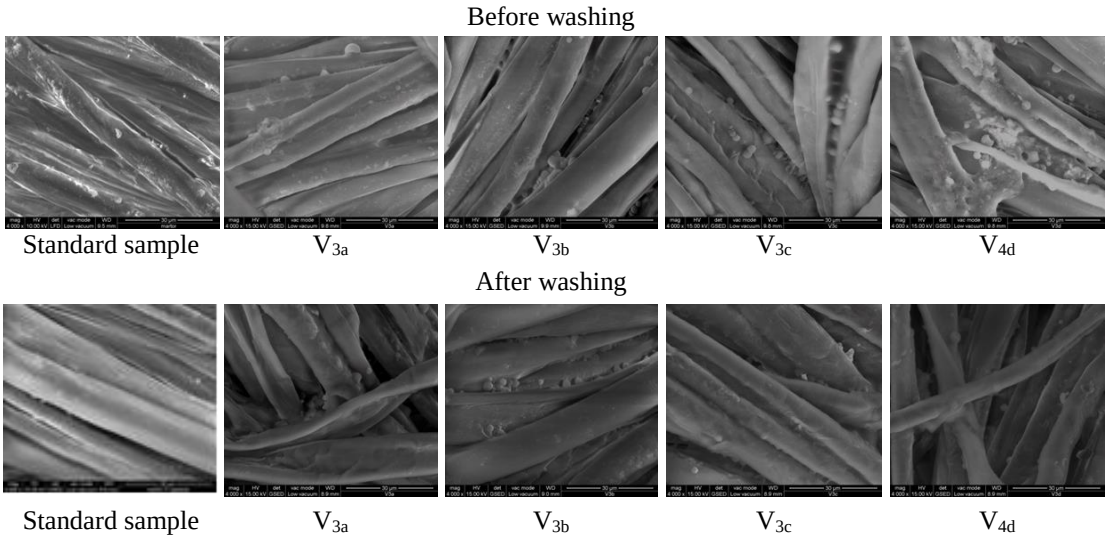

Fig. 4*: SEM images before and after one washing cycle of the textile materials*

4. CONCLUSIONS

Using GC-MS analysis has been evidencied the presence of vitamin E acetate, confirming the presence of vitamin E deposited on the textile materials for each experimental variant. A new band at 1731 cm^{-1} and several changes of peaks intensity in the fingerprint region were observed after functionalization treatment of textile materials, confirming the presence of vitamin E microcapsules. Electronic images recorded for textile materials treated with vitamin E microcapsules reveal a preponderant interfibrillar distribution of vitamin E microcapsules that were predominantly intact on the textile support, having smaller or larger sizes with spherical shapes. After one washing cycle, the number of vitamin E microcapsules from the surface has been reduced.

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