FUNCTIONAL FINISHING OF TEXTILES USING BIOACTIVE AGENTS BASED ON NATURAL PRODUCTS

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There is nowadays an increasing vogue for so-called cosmetic textiles which are essentially garments that are designed to come into contact with the skin, which then transfer some active substances that may be used for cosmetic purposes. In order to obtain the textile materials with potential for use in the development of cosmetic textiles, this study approached the experimentation of deposition by padding and exhaustion of commercial rose microcapsules on knitted textile structures with different fiber compositions. The finished textile materials were characterized in terms of functionalization treatments durability, release of bioactive compounds in sweat solution and antibacterial activity, respectively. In order to identify the functionalization method which ensures the deposition of a high content of microcapsules on the knitted fabric surface, which implicitly leads to a high functional effect, solid-liquid and liquid-liquid extraction method for sample preparation and a gas chromatography with mass spectrometry detection (GC-MS) for identification of active compounds have been involved. SEM analysis was used to investigate the distribution of microcapsules on the textile materials surface, before and after 1 washing cycle and 9000 abrasion cycles. Antibacterial activity of treated samples has been evaluated before and after 1 washing cycle against the *Staphylococcus aureus* test strains.

Keywords: knitted textile materials, rose essential oil, GC-MS, SEM

INTRODUCTION

Health and beauty are an increasing vogue for everybody in this entire world. In recent years, cosmetic textiles take over as a rapidly growing and developing segment of functional textiles used in health and hygiene sectors, means new target groups and new markets for textile industry (Azizi *et al.*, 2014). Microencapsulation technology is gaining wide acceptance in a broad range of industrial applications in markets such as textiles, drug delivery systems, pesticides and agrochemicals, consumer care and adhesives. Some active substances which are used for cosmetic purpose are then introduced to the skin through friction or other trigger mechanisms, in particular to combat ageing effects in order to promote the younger look (Alonso *et al.*, 2013).

Rose essential oil is especially great for dry or aging skin. Researchers have found that it contains several therapeutic compounds known to promote healing, especially antimicrobial and anti-inflammatory compounds. As a result, rose essential oil helps refine skin texture and tone, and can be helpful with managing skin conditions such as psoriasis and dermatitis.

In order to obtain the textile materials with potential for use in the development of cosmetic textiles, this study approached the experimentation of deposition by padding and exhaustion of commercial microcapsules with rose essential oil content on knitted textile structures with different fiber compositions.

EXPERIMENTAL PART

Materials

Deposition of microcapsules dispersions was performed on 100% cotton pique knitted fabric with the weight of 196 g/m² (Code V1) and 50% cotton / 50% polyester interlock knitted fabric with the weight of 178 g/m² (Code V2). For functionalization treatments, highly concentrated rose essential oil microcapsules slurry from Rudolf Duraner (Turkey) with 80% active content has been used. Binder ST from Rudolf Duraner (Turkey) has been used for the fixation of microcapsules on textile materials.

Functionalization Treatments

Prior to functionalization treatments the textile fabrics were subjected to preliminary preparation by hot alkaline treatment and bleaching.

Padding Method (P)

The deposition of microcapsules on the textile materials was performed in a single bath, on the laboratory padder (Roaches, UK) with 9.6 g/L microcapsule slurry and 2.4 g/L Binder ST, with a wet pick-up rate of 100 %. Drying of the impregnated textile materials was carried out at a temperature of 100°C for 2 minutes on the drying/ curing/ heat-setting/ vaporization unit, model TFO/S 500 mm (ROACHES, UK).

Exhaustion Method (E)

In the exhaustion method, the textile materials were treated with 1,6% microcapsule slurry (w/v) and 0,4% Binder ST (w/v) at MLR (material to liquor ratio) 1:20 for 20 minutes at 50°C. The pH=5 of the process bath was adjusted with CH₃COOH (60%). The treated textile materials were then squeezed and dried at 100°C for 2 minutes. For the functionalization treatments, the laboratory equipment Ugolini (Italy) has been used.

Methods

Durability to Washing

The samples treated with microcapsules have subjected to 1 wash cycle under the following conditions: 2g/L detergent containing no phosphate and bleaching agent, at a temperature of 40°C for 30 minutes. Samples were subsequently rinsed with cold water, freely dried horizontally and then analyzed by GC-MS method and SEM.

Durability to Abrasion

According to the SR EN ISO 12947-2/2017 standard, the treated fabrics were tested by the Martindale Abrasion Tester for 9000 abrasion cycles. The presence of the microcapsules after an abrasion test was observed by SEM.

Release of Active Compounds to Sweat (Acidic and Alkaline)

For the release assessment of bioactive compounds to sweat, the samples were immersed during 30 minutes in alkaline and acidic solutions. The identification of the main active compounds remained on the treated samples was performed by GC-MS.

Antibacterial Tests

The antibacterial activity of the treated samples was qualitatively assessed through agar diffusion method according with the SR EN ISO 20645:2004 standard, by using of cultures in liquid medium of ATCC 6538 *Staphylococcus aureus* test strains (Gram-positive). The samples were disposed on the culture medium in the middle of Petri plates, and after were incubated at 37°C for 24 h. In order to asses the durability of applied treatment the samples were also tested after 1 washing cycle.

Gas-Chromatography (GC-MS)

For GC-MS analysis, 0,3 g textile material were cut in small pieces and then extracted in 30 mL distilled water by agitation at 500 rpm, during for 10 minutes, after that the extracted solution was filtered. 30 mL ethyl acetate was added in the filtered solution and the solution was agitated for 3 minutes, while the organic phase was separated. Gas-Chromatography was carried out with an Agilent 6890N Gas-Chromatograph coupled with 5973N Mass Spectrometer (USA).

RESULTS AND DISCUSSION**Gas-Chromatography (GC-MS)**

Retention time (Rt), CAS number and peaks area for the representative compounds identified on the functionalized samples are shown in Table 1.

Table 1. Main compounds indentified by GC-MS

Rt	CAS	Component	Area	Rt	CAS	Component	Area
V1E				V1P			
6.46	1117-61-9	(R)-(+)- β -Citronellol	128655	6.46	1117-61-9	(R)-(+)- β -Citronellol	184020
6.98	624-15-7	Geraniol	51633	6.98	624-15-7	Geraniol	78645
11.71	90-17-5	Rose acetate	45069	11.71	90-17-5	Rose acetate	31432
13.21	110-27-0	Isopropyl myristate	95213	13.21	110-27-0	Isopropyl myristate	49388
-	-	-	-	9.09	97-53-0	Eugenol	21786
V2E				V2P			
6.46	1117-61-9	(R)-(+)- β -Citronellol	117487	6.46	1117-61-9	(R)-(+)- β -Citronellol	162723
6.98	624-15-7	Geraniol	43716	6.98	624-15-7	Geraniol	68934
11.71	90-17-5	Rose acetate	32200	11.71	90-17-5	Rose acetate	37565
13.21	110-27-0	Isopropyl myristate	61312	13.21	110-27-0	Isopropyl myristate	52364

GS-MS analysis identified the main components of rose essential oil, such us: rose acetate, R)-(+)- β -Citronellol, geraniol, isopropyl myristate, and eugenol.

From the GC-MS data, a clear delimitation between the experimental variants and the most efficient solution in terms of immobilization of the microcapsules with rose essential oil on the surface of the textile materials cannot be made.

Functionalization Treatments Durability to Washing

Retention time (Rt), CAS number and peaks area for the representative compounds identified on the functionalized samples after 1 washing cycle and treatment with sweat (alkaline and acidic) solution are depicted in Table 2.

Table 2. Main compounds indentified on the samples after 1 washing cycle / treatment to sweat solution

Rt	CAS	Component	Area	Rt	CAS	Component	Area
1 washing cycle							
V1E				V1P			
11.71	90-17-5	Rose acetate	14069	11.71	90-17-5	Rose acetate	17202
13.21	110-27-0	Isopropyl myristate	17648	13.21	110-27-0	Isopropyl myristate	20947
V2E				V2P			
11.71	90-17-5	Rose acetate	17653	11.71	90-17-5	Rose acetate	18370
13.21	110-27-0	Isopropyl myristate	33495	13.21	110-27-0	Isopropyl myristate	42800
Release to sweat							
Acidic perspiration				Alkaline perspiration			
V1E							
-	-	-	-	6.45	1117-61-9	(R)-(+)- β -Citronellol	16223
11.71	90-17-5	Rose acetate	7125	11.71	90-17-5	Rose acetate	6195
-	-	-	-	13.21	110-27-0	Isopropyl myristate	3334
V1P							
6.45	1117-61-9	(R)-(+)- β -Citronellol	19129	11.71	90-17-5	Rose acetate	5121
11.70	90-17-5	Rose acetate	5148	13.21	110-27-0	Isopropyl myristate	3558
V2E							
6.46	1117-61-9	(R)-(+)- β -Citronellol	18581	11.71	90-17-5	Rose acetate	5812
11.71	90-17-5	Rose acetate	6020	-	-	-	-
V2P							
11.70	90-17-5	Rose acetate	3580	11.70	90-17-5	Rose acetate	3483

Analyzing the data obtained after 1 washing cycle, it can be observed that (R)-(+)- β -citronellol and geraniol components were not been highlighted for all the samples, meaning that durability of the treatment is decreased. However, some of the main compounds have been identified, confirming that the microcapsules remained loaded after 1 washing cycle. The amount of rose acetate component decreased when the impregnated textile knitted fabrics were subjected to 1 washing cycle, the loss of this component ranging from 45% (V1P, V2E) to 69 % (V1E). Also, the isopropyl myristate component decrease after 1 washing cycle, the small decrease being around of 18 %, observed for the 50 % cotton / 50 polyester knitted fabric treated by padding method (V2P).

Regarding the release of active compounds to sweat solution, it can be noticed that the peak areas for compounds remained on the samples after treatment are substantially diminished, indicated the release tendency that occurs after treatment to sweat.

Electron Microscopy

SEM images obtained at a magnification of x 2000 for analyzed samples before and after 1 washing cycle and 9000 abrasion cycles are shown in Figure 1.

Figure 1. SEM images for the analyzed samples

SEM micrographs registered for the functionalized fabrics highlights that the microcapsules were successfully embedded in the textile materials structure. 1 washing cycle reveal that part of the microcapsules still adhered to the fibers surface and the washing operation did not cause any significant damages to the microcapsules. The amount of the microcapsules is significantly lower after 9000 abrasion cycles, only few microcapsules were still found adhering to the fiber surface.

Antibacterial Tests

Images of Petri plates after 48 h incubation are shown in Figure 2.

Figure 2. Images of Petri plates after 24 h incubation

It can be observed that the knitted fabrics before washing shows a good antibacterial effect against *S. aureus* (Figure 1), but after 1 washing cycle, the effect is reduced being evaluated at the efficacy limit (0 mm inhibition area and slight growth of bacteria).

CONCLUSIONS

The commercial microcapsules with rose essential oil content were embedded in the cotton and cotton/polyester knitted fabrics by using padding and exhaustion techniques. From the GC-MS data, a clear delimitation between the experimental variants and the most efficient solution in terms of immobilization of the microcapsules on the surface of the textile materials cannot be made. The peak areas for compounds remained on the samples after treatment to sweat solution are substantially diminished indicated the release tendency that occurs. The overall results confirmed that the functionalized treatments withstand 1 washing cycle, but the amount of the microcapsules is significantly lower after 9000 abrasion cycles. An antibacterial effect against the *Staphylococcus aureus* test strain is observed for all experimental variants, antibacterial activity decrease after 1 washing cycle.

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