STRUCTURED BIOEMULSIONS BASED ON MIXTURE OF MEMBRANE CONCENTRATED PLANT EXTRACTS WITH APPLICATIONS IN FUNCTIONALIZATION OF TEXTILE MATERIALS

Demetra SIMION[1*], Alina POPESCU[1], Laura CHIRILĂ[1], Doina TOMA[1], Laurențiu DINCĂ[1], Daniela BERECHET[2]

[1]The National Research and Development Institute for Textiles and Leather (INCDTP), 16 Lucretiu Patrascanu st., 030508 Bucharest, Romania, email: demetra.simion@yahoo.com

[2]INCDTP – Division: Leather and Footwear Research Institute (ICPI), 93 Ion Minulescu st., 031215, Bucharest, Romania

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STRUCTURED BIOEMULSIONS BASED ON MIXTURE OF MEMBRANE CONCENTRATED PLANT EXTRACTS WITH APPLICATIONS IN FUNCTIONALIZATION OF TEXTILE MATERIALS

ABSTRACT. The work refers to the process of obtaining structured bioemulsions based on mixtures of plant extracts (sage, thyme, mint and lemongrass) concentrated by membrane technology, formulated with surfactants (Lauryl glucoside, Tween 20 and Tween 80) and collagen hydrolysate, used for the functionalization of textile materials, giving them antibacterial properties with durability when washed. The bioemulsions are diluted with water in different concentrations and are applied to textile supports made of 100% cotton fibers, by the padding method, in three successive passes, with a take-up degree 85% and pressure of 2.7 bar, followed by a drying step at temperatures of 60°C and 100°C, for approximately 2-5 minutes. A method for validating durability is FTIR-ATR spectroscopy to identify the markers, for calculation of retention rates and analysis of decomposition curves. SEM electron microscopy is used for morphological confirmation of fixation of structured bioemulsions on textile materials even after 5 repeated washing cycles.
KEYWORDS: structured bioemulsions, membrane technology, functionalization of textile materials, antibacterial properties and durability when washed

BIOEMULSII STRUCTURATE PE BAZĂ DE AMESTEC DE EXTRACTE VEGETALE CONCENTRATE PRIN TEHNOLOGIE MEMBRANARĂ, CU APLICAȚII ÎN FUNCȚIONALIZAREA MATERIALELOR TEXTILE

REZUMAT. Lucrarea se referă la obținerea de bioemulsii structurate pe bază de amestecuri de extracte din plante (salvie, cimbru, mentă și lemongrass) concentrate prin tehnologie membranară, formulate cu surfactanți (Lauril glucozid, Tween 20 și Tween 80) și hidrolizat de colagen, utilizate pentru funcționalizarea materialelor textile, conferindu-le proprietăți antibacteriene rezistente la spălări repetate. Bioemulsiile sunt diluate cu apă în diferite concentrații și aplicate pe suporturi textile din fibre de bumbac 100%, prin metoda impregnării în trei treceri succesive, cu grad de absorbție de 85% și presiune de 2,7 bari, urmate de o etapă de uscare la temperaturi de 60 °C și 100 °C, timp de aproximativ 2-5 minute. O metodă de validare a durabilității la spălări repetate este spectroscopia FTIR-ATR pentru identificarea markerilor, pentru calcularea ratelor de retenție și analiza curbelor de descompunere. S-a utilizat microscopia electronică SEM pentru confirmarea morfologică a fixării bioemulsiilor structurate pe materialele textile chiar și după 5 cicluri repetate de spălare.
CUVINTE CHEIE: bioemulsii structurate, tehnologie membranară, funcționalizarea materialelor textile, proprietăți antibacteriene și durabilitate la spălare

BIOÉMULSIONS STRUCTURÉES À BASE D'UN MÉLANGE D'EXTRAITS VÉGÉTAUX CONCENTRÉS PAR TECHNOLOGIE MEMBRANAIRE ET LEURS APPLICATIONS DANS LA FONCTIONNALISATION DES MATÉRIAUX TEXTILES

RÉSUMÉ. Ce travail porte sur l'obtention de bioémulsions structurées à base de mélanges d'extraits végétaux (sauge, thym, menthe et citronnelle) concentrés par technologie membranaire, formulées avec des tensioactifs (lauryl glucoside, Tween 20 et Tween 80) et de l'hydrolysat de collagène, utilisées pour la fonctionnalisation de matériaux textiles, leur conférant des propriétés antibactériennes qui résistent aux lavages. Les bioémulsions sont diluées dans de l'eau à différentes concentrations et appliquées sur des supports textiles composés de fibres 100 % coton, par la méthode d'encollage, en trois passes successives, avec un taux d'absorption de 85 % et une pression de 2,7 bars, suivies d'une étape de séchage à des températures de 60 °C et 100 °C, pendant environ 2 à 5 minutes. Une méthode de validation de la durabilité consiste à utiliser la spectroscopie FTIR-ATR pour identifier les marqueurs, pour le calcul des taux de rétention et l'analyse des courbes de dégradation. La microscopie électronique à balayage (MEB) est utilisée pour confirmer, d'un point de vue morphologique, la fixation des bioémulsions structurées sur les matériaux textiles, même après cinq cycles de lavage répétés.
MOTS-CLÉS : bioémulsions structurées, technologie membranaire, fonctionnalisation des matériaux textiles, propriétés antibactériennes et durabilité au lavage

* *Correspondence to: Demetra SIMION, The National Research and Development Institute for Textiles and Leather (INCDTP), 16 Lucretiu Patrascanu st., 030508 Bucharest, Romania, email: demetra.simion@yahoo.com*

INTRODUCTION

The paper presents the obtaining of structured bioemulsions based on a mixture of surfactants: Lauryl glucoside, Tween 20, Tween 80, with collagen hydrolysate and a mixture of plant extracts, membrane concentrates from: sage, thyme, lemongrass, mint, in equal ratios. The structured bioemulsions created can be used as new additives, auxiliaries in the textile industry, for the functionalization of surfaces. There is an interest in the textile industry to obtain new auxiliaries and replace the classic, toxic and polluting ones [1-10]. The aim is to create auxiliaries that improve the properties of surfaces, be "friendly" with the environment and comply with European pollution standards. The paper also presents a "bio" fixing system on textile surfaces of the obtained bioemulsions, which is innovative, based on biosurfactants and collagen hydrolysate, eliminating the need for synthetic, toxic additives.

The functionalization of textile materials with bioactive compounds of plant origin is a major area of interest, driven by current requirements regarding sustainability, biocompatibility and reducing environmental impact. In this context, numerous technological solutions have been developed aimed at obtaining plant extracts, formulating them in transport systems and applying them to textile supports [1]. Due to the high content of active compounds (polyphenols, flavonoids, volatile oils), plant extracts are used to confer antibacterial, antifungal and antioxidant properties to textile materials [2]. To obtain plant extracts, classical methods of extraction in aqueous or hydroalcoholic medium are known, as well as assisted methods, such as ultrasound or microwave extraction [3]. With the evolution of processing technologies, membrane separation and concentration methods (microfiltration, ultrafiltration, nanofiltration, reverse osmosis) have been developed, which allow the obtaining of concentrated extracts, with a high content of active compounds and improved stability. In this regard, patents RO128610 and RO132969 describe the use of membrane technologies to obtain concentrated plant extracts, rich in active compounds (polyphenols, flavonoids, saponins, polysaccharides), highlighting the advantages of these methods in terms of purity and separation efficiency [2]. However, these solutions do not address the formulation of extracts in complex dispersed systems nor their use in textile applications.

For the stabilization and application of active compounds on textile supports, the literature documents the use of emulsified systems, microemulsions or bioemulsions, as well as of encapsulation systems, such as microcapsules or coacervates [1-3].

The direct application of plant extracts to textile materials is described in the literature, but, in the absence of appropriate structuring systems, this leads to reduced stability and limited persistence of the functional effect after use and washing cycles. Emulsified systems or bioemulsions are described in which plant extracts are stabilized with ionic or nonionic surfactants and used for the treatment of textile surfaces, these systems being, in some cases, combined with biopolymers or crosslinking agents, in order to increase adhesion to the textile support and resistance to washing [1, 4-6]. However, existing solutions present a series of technological and functional limitations that restrict the effective use of plant extracts in the functionalization of textile materials. Thus, systems based on microcapsules or dispersed particles are generally characterized by relatively large particle sizes and by an uneven distribution on the surface of the textile support, which leads to a reduced treatment efficiency and an uncontrolled release of active compounds [3]. In the case of emulsified systems or classic bioemulsions, their stability is often limited in time, and the internal structure does not always allow for the effective protection of bioactive compounds, especially under conditions of repeated use and washing [2, 3]. In addition, the use of biosurfactants and natural compounds as stabilizing agents for emulsion systems is an emerging direction, highlighting their advantages in terms of biocompatibility and the ecological character of the developed systems [4-6]. Direct application of plant extracts or poorly structured systems leads to

insufficient fixation of the active compounds on the textile support, causing a rapid loss of the functional effect after use and washing cycles [5]. Membrane concentration technologies are mainly used to obtain concentrated plant extracts with a high content of active compounds, but the integration of these extracts into stable and structurally controlled dispersed systems for textile applications is limited or insufficiently developed [7-10]. In most existing solutions, plant extracts are used individually or in simple combinations, without capitalizing on the synergy of complex mixtures and without controlling the architecture of the dispersed systems, which leads to reduced functional efficiency and increased variability of results [10].

Consequently, there is a great need to develop new technological solutions that allow the obtaining of stable, efficient and ecological systems, capable of optimally utilizing bioactive compounds from plant extracts and ensuring improved functional performance of treated textile materials.

EXPERIMENTAL

Materials and Methods

The following materials have been used: collagen hydrolysate from Sigma-Aldrich; Lauryl glucoside, Tween 20, Tween 80 from SERVA Feinbiochemica GmbH & Co; sage, thyme, lemongrass, and mint extracts from Hofigal Export Import SA. The structured bioemulsions are characterized by FTIR-ATR spectroscopy, scanning electron microscopy and microbiological tests. The experimental techniques used in this research were performed using:

- a “SEM QUANTA 200” equipment from FEI company, with EDS/EDX coupled. The samples were prepared by slow evaporation in clean atmosphere at room temperature;
- a JASCO FTIR-ATR spectrophotometer.

The plant extract concentration experiments were performed on a KMS Laboratory Cell CF-1 module, equipped with prefiltration, microfiltration and ultrafiltration membranes from Sigma-Aldrich (Figure 1).

Figure 1. Image of a KMS Laboratory Cell CF-1 module

RESULTS AND DISCUSSIONS

Obtaining Membrane-Concentrated Plant Extracts

In the first stage, the vegetable extracts of sage, thyme, lemongrass and mint are obtained, used in the mixture, in equal proportions. The extraction is carried out in an aqueous or hydroalcoholic medium (water: ethanol ratio 1:1), at temperatures of 20-60°C, for 30-120 minutes, using a solid:liquid ratio of 1:10-1:20. The extracts obtained are subsequently subjected to a concentration process by coupled membrane technology, using microfiltration, ultrafiltration and/or nanofiltration modules. The process is carried out at working pressures between 2 and 8 bar, temperatures of 23-30°C and durations of approximately 30-60 minutes. Under these conditions, a concentrated retentate is obtained, with a content of bioactive compounds approximately 1.5-2 times higher than the initial extract, characterized by an increased content of bioactive compounds compared to the initial extract (Figure 2).

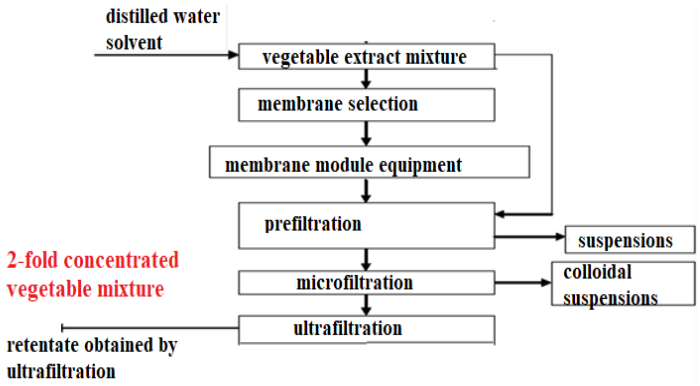
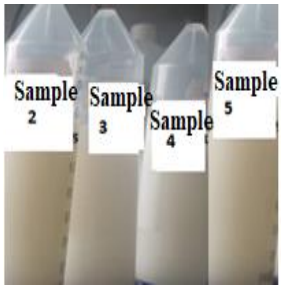


Figure 2. Technological flow for obtaining the mixture of vegetable extract from: sage, thyme, lemongrass, mint, concentrated through membranes

Obtaining Structured Bioemulsions Based on a Mixture of Membrane-Concentrated Plant Extracts

In the next stage, the concentrated extracts are integrated into a dispersed system by formulating bioemulsions. For this purpose, a system of surfactants is used, consisting of Lauryl glucoside, Tween 20 and Tween 80, in concentrations of 3% each, together with collagen hydrolysate in a concentration of 1%. The dispersion medium is represented by a water:ethanol mixture in a 1:1 ratio. The emulsification process is carried out under controlled temperature and stirring conditions, in two successive stages: i) in the first stage, the surfactants are dissolved and homogenized by stirring at room temperature (20-25°C), for 30-60 minutes, at speeds of 800-1200 rpm; ii) in the second stage, the collagen hydrolysate and the mixture of membrane-concentrated plant extracts are added, with subsequent adjustment of the pH to values close to neutral (5.5-7). Subsequently, the system is homogenized at temperatures up to 50°C, for 30-60 minutes. Under these conditions, multiple structured bioemulsions are formed, of the "emulsion in emulsion" type, with particle sizes in the nano- and micrometric range, of approximately 50-200 nm (Figure 3).


a)



b)

Figure 3. Photographic images of: **sample 1** – multiple emulsion based on Tween 20/Tween 80/Lauryl Glucoside and collagen hydrolysate mixture and membrane-concentrated vegetable extract mixture of: sage, thyme, lemongrass, mint; **sample 2** – multiple emulsion based on Tween 20/Tween 80/Lauryl Glucoside and collagen hydrolysate mixture and membrane-concentrated lemongrass extract; **sample 3** – multiple emulsion based on Tween 20/Tween 80/Lauryl Glucoside and collagen hydrolysate mixture and membrane-concentrated thyme extract; **sample 4** – multiple emulsion based on Tween 20/Tween 80/Lauryl Glucoside and collagen hydrolysate mixture and membrane-concentrated sage extract; **sample 5** – multiple emulsion based on Tween 20/Tween 80/Lauryl Glucoside and mixture of collagen hydrolysate and membrane-concentrated mint extract

These are stabilized by the interaction between surfactants and collagen hydrolysate, forming a "bio" type fixation system, which facilitates the anchoring of active compounds on the surface of textile fibers.

Functionalization of Textile Materials Using the Structured Bioemulsions Created

Sample 1 from Figure 3 was selected, dilutions of 2%, 10%, 20%, 50% and 100% were prepared and applied, by the pad method, on 100% cotton textile material. The treatment is followed by drying at temperatures of 60°C and 100°C, for 2-5 minutes. The 100% cotton textile material was tested for antibacterial activity, according to the standardized method SR EN ISO 20645:2004, using reference bacterial strains (*Escherichia coli* ATCC 11229 and *Staphylococcus aureus* ATCC 6538). To evaluate the durability of the applied treatments, the textile materials that showed the best results in terms of antibacterial effect were selected and subjected to 1, 2 and 5 washing cycles, these being represented by textile materials made of 100% cotton fibers, cotton fibers treated with 50% and 100% bioemulsion dilutions, dried at 60°C and 100°C. Microbiological tests carried out according to EN ISO 20645/2004 confirm good antibacterial, antifungal effects (with inhibition zones of 1-18.5 mm) before washing. They were carried out on selected textiles, 1, 2 and 5 washing cycles. After 5 washing cycles, the effects are maintained satisfactorily (lack of bacterial or fungal growth under the sample), demonstrating the efficiency of fixing bioemulsions on textiles, impregnated (Table 1).

The following notation was made for the sectioned samples, initially with the applied emulsion dilution of 50% and 100%, on 100% cotton (code A) by drying at 60°C and 100°C: Ai-A50/100, Ai-A100/100, Ai-A50/60, Ai-A100/60 and after one wash cycle with: Ai-A50/100S1, Ai-A100/100S1, Ai-A50/60S1, Ai-A100/60S1, after two wash cycles: Ai-A50/100S2, Ai-A100/100S2, Ai-A50/60S2, Ai-A100/60S2 and after 5 wash cycles with: Ai-A50/100S5, Ai-A100/100S5, Ai-A50/60S5, Ai-A100/60S5.

Table 1: Experimental results for determining the antibacterial effect of samples immobilized on 100% cotton textile material (code A) before washing and after 1, 2, 5 washing cycles

Sample	*Escherichia coli*		*Staphylococcus aureus*	
	Inhibition zone (mm)	Effect evaluation	Inhibition zone (mm)	Effect evaluation
Control sample	0	Insufficient	0	Insufficient
Ai-A50/100	11	Good antibacterial effect	14	Good antibacterial effect
Ai-A100/100	18	Good antibacterial effect	16	Good antibacterial effect
Ai-A50/60	12	Good antibacterial effect	10	Good antibacterial effect
Ai-A100/60	18.5	Good antibacterial effect	17	Good antibacterial effect
After 1 wash cycle				
Ai-A50/100	4	Satisfactory	2.5	Satisfactory
Ai-A100/100	15	Satisfactory	15.5	Satisfactory
Ai-A50/60	4	Satisfactory	5	Satisfactory
Ai-A100/60	6	Satisfactory	10	Satisfactory
After 2 wash cycles				
Ai-A50/100	3	Satisfactory	3	Satisfactory
Ai-A100/100	5	Satisfactory	3.5	Satisfactory
Ai-A50/60	2	Satisfactory	1	Satisfactory
Ai-A100/60	1	Satisfactory	2.5	Satisfactory
After 5 wash cycles				
Ai-A50/100		Satisfactory		Satisfactory
Ai-A100/100		Satisfactory		Satisfactory
Ai-A50/60		Satisfactory		Satisfactory
Ai-A100/60		Satisfactory		Satisfactory

Characterization of Functionalized Textile Materials with the Structured Bioemulsions Created

The characterization of the treated textile materials was carried out by specific methods, such as microbiological tests, FTIR-ATR spectroscopy and scanning electron microscopy (SEM) coupled with EDS/EDX analysis (Table 2). A method for validation of durability (persistence) was presented, characterized by the use of FTIR-ATR spectroscopy for the identification of markers: thymol from thyme extract at ν =1580 cm^{-1} (aromatic ring); aldehydes from lemongrass extract at ν =1672 cm^{-1}; menthol from mint extract at ν =1710 cm^{-1} (Ketone); 1,8-Cineole from sage extract at ν =1080 cm^{-1} (ether) and for the calculation of retention rates and the analysis of the decomposition curves, respectively (Figures 4-7).

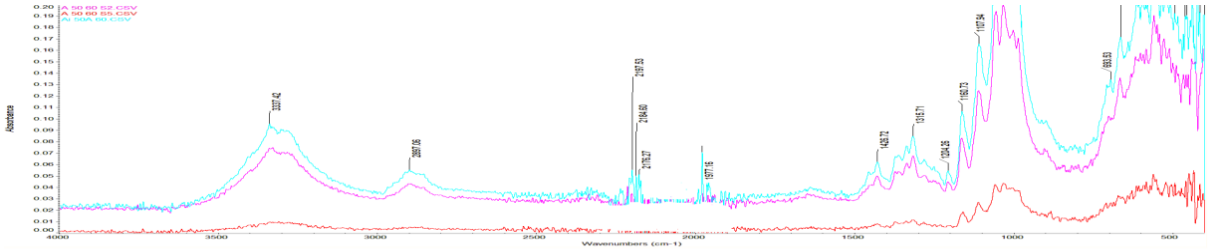


Figure 4. Detail of the FTIR-ATR spectra overlay for samples: Ai-50A/60, A-50/60S2, A-50A/60S5

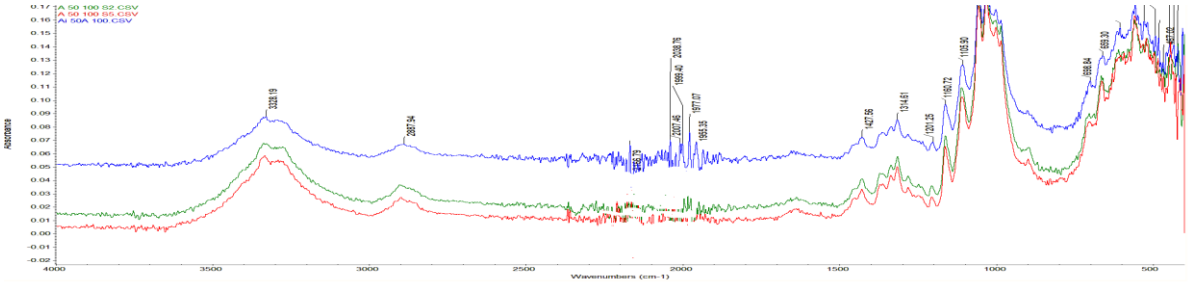


Figure 5. Detail of the FTIR-ATR spectra overlay for samples: Ai-50A/100, A-50A/100S2, A-50A/100S5

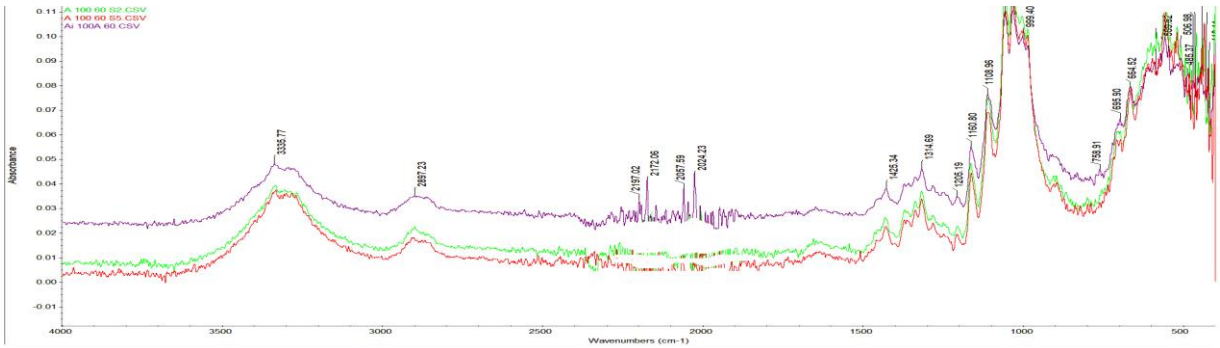


Figure 6. Detail of the FTIR-ATR spectra overlay for samples: Ai-100A/60, A-100A/60S2, A-100A/60S5

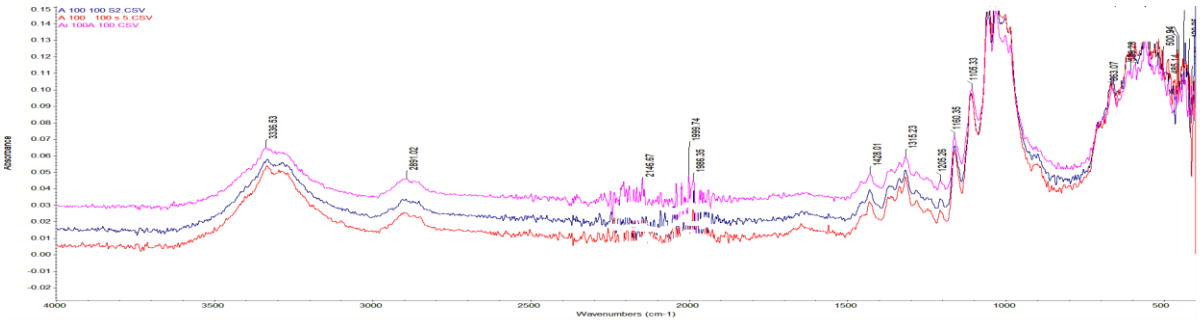


Figure 7. Detail of the FTIR-ATR spectra overlay for samples: Ai-100A100, A-100A/100S2, A-100A/100S5

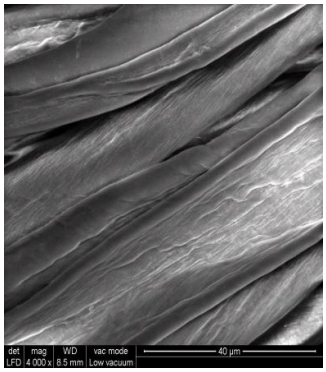
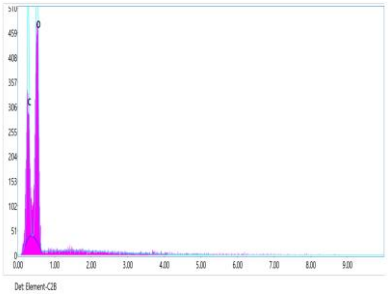
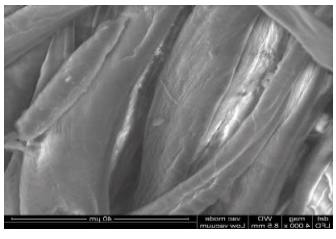
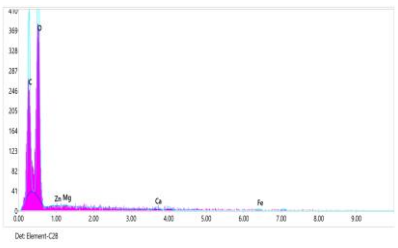
The calculation of retention rates shows that a significant percentage of vegetable mixture remains on cotton treated at 100°C (for sample A-50A/100S5 – 32.95%) after 5 washing cycles, compared to impregnation at 60°C (for sample A-50A/60S5 – 4%), for the same initial mixture concentration but different drying temperatures (Table 2).

Table 2: Calculation of retention rates for samples selected

Sample	Absorbance at 2897 cm^{-1}	Retention rates (%)
Ai-A50A/60	0.082	50
A-A50/60S2	0.038	46
A-A50/60S5	0.003	4
Ai-A50A/100	0.088	50
A-50/100S2	0.068	38.64
A-50/100S5	0.058	32.95
Ai-A100A/60	0.035	100
A-A100A/60S2	0.018	51.4
A-A100A/60S5	0.014	40
Ai-A100A/100	0.046	100
Ai-100A/100S2	0.033	71.7
Ai-100A/100S5	0.025	54.3

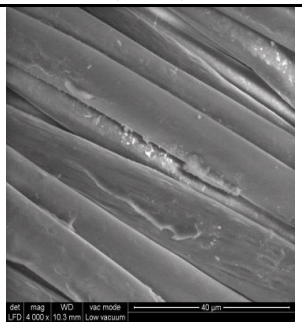
Morphological analysis performed by scanning electron microscopy (SEM) highlights the uniform distribution of the bioemulsified system on the surface of textile fibers. EDS/EDX analysis of selected samples highlights the presence of the majority of mineral elements, specific to each extract (sage, mint, lemongrass, thyme) even after 5 washing cycles, which demonstrates good resistance to washing of the treatments performed, even in the absence of commonly used fixing agents (Table 3). Influence of technological parameters – increasing the drying temperature to 100°C – leads to more efficient fixing and increased stability to washing, with possible variations in the intensity of the antibacterial effect.

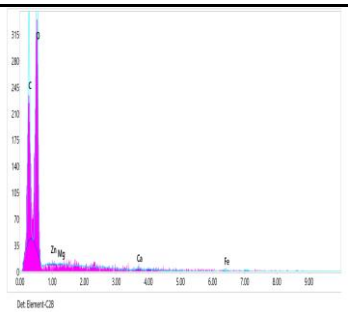
Table 3: SEM and EDS/EDX images for all samples analyzed before and after 2 and 5 washing cycles, respectively

Sample	SEM image (x4000)	EDS/EDX image	eZAF Quant Result - Analysis Uncertainty: 9.40 %
Control sample (code A)			Element / Weight % / Atomic % / Error % C K 40.9 48.0 9.7 O K 59.1 52.0 9.7
Ai-A50/100 (100°C)			Element / Weight % / Atomic % / Error % C K 37.9 47.5 10.3 O K 53.1 49.9 9.8 Mg K 0.2 0.2 73.5 Ca K 1.4 0.5 58.7 Fe K 7.1 1.9 24.3 Zn L 0.2 0.0 74.8

Sample	SEM image (x4000)	EDS/EDX image	eZAF Quant Result - Analysis Uncertainty: 9.40 %

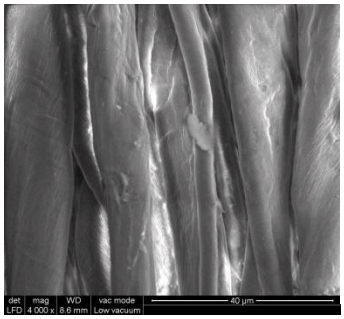
Ai-A100/100 (100°C)

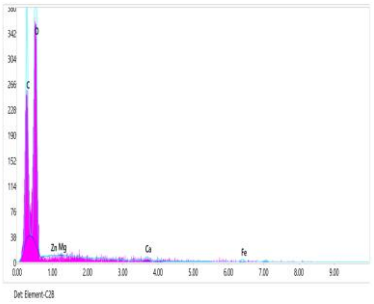




Element	Weight %	Atomic %	Error %
C K	37.2	46.8	10.5
O K	53.4	50.5	9.8
Mg K	0.2	0.1	77.4
Ca K	1.5	0.5	65.0
Fe K	7.4	2.0	17.4
Zn L	0.3	0.1	74.8

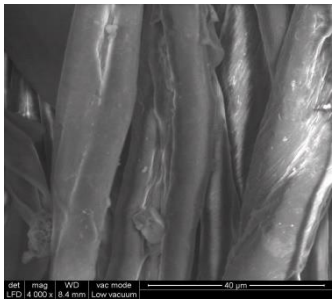
Ai-A50/60 (60°C)

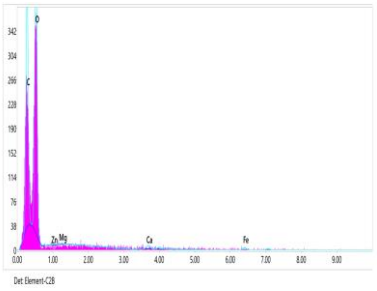




Element	Weight %	Atomic %	Error %
C K	37.6	47.4	10.4
O K	52.3	49.6	9.8
Mg K	0.2	0.1	74.1
Ca K	1.8	0.7	46.1
Fe K	8.0	2.2	21.2
Zn L	0.1	0.0	99.0

Ai-A100/60 (60°C)

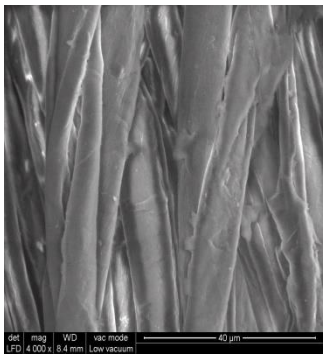


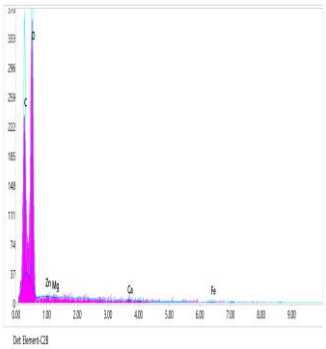


Element	Weight %	Atomic %	Error %
C K	39.0	48.9	10.3
O K	51.3	48.2	9.9
Mg K	0.2	0.1	77.8
Ca K	1.6	0.6	46.5
Fe K	7.9	2.1	20.3
Zn L	0.1	0.0	86.7

After 2 wash cycles

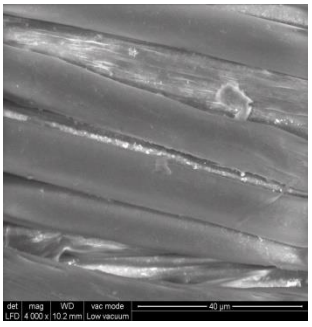
A-A50/100S2 (100°C)

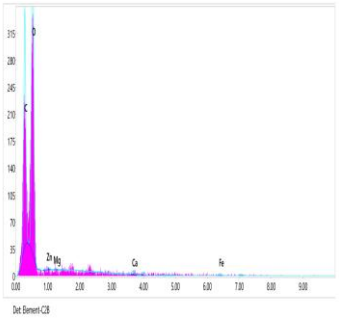




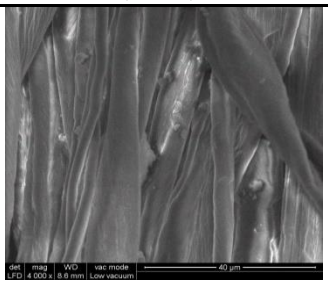
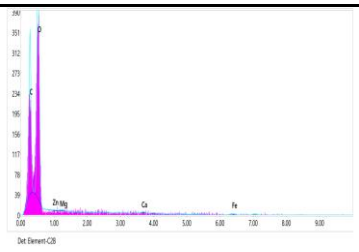
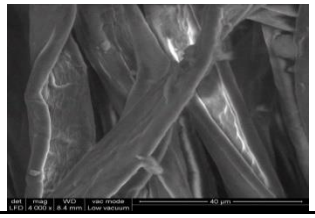
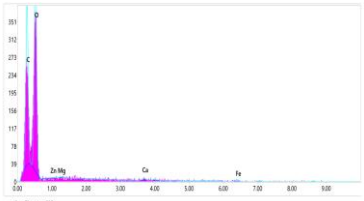
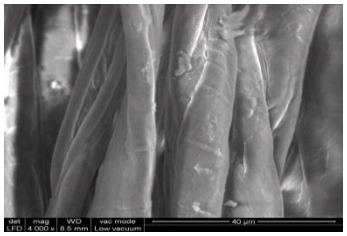
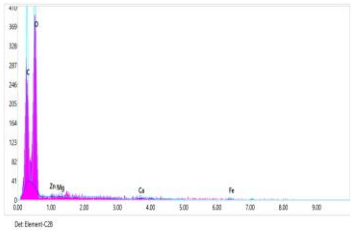
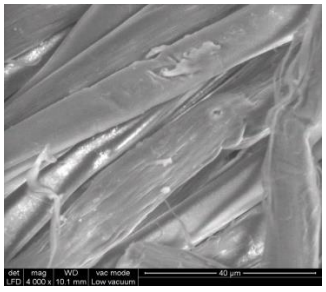
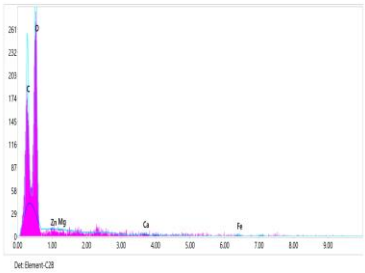
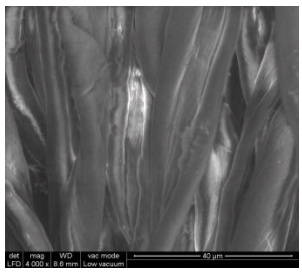
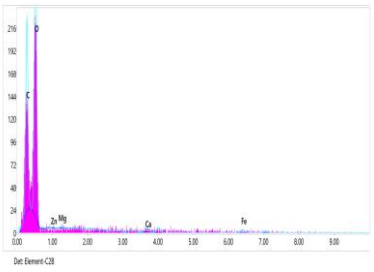
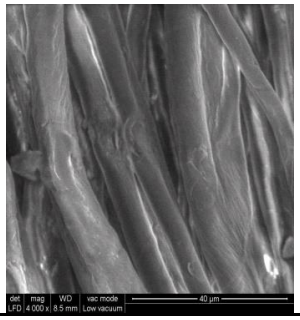
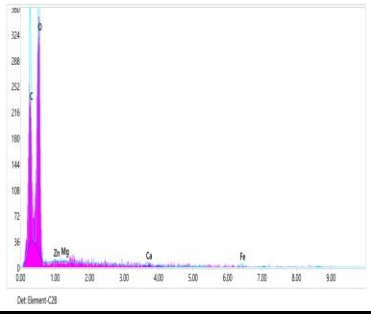
Element	Weight %	Atomic %	Error %
C K	37.4	47.0	10.5
O K	53.3	50.3	9.8
Mg K	0.3	0.2	73.9
Ca K	1.2	0.4	65.4
Fe K	7.6	2.1	33.9
Zn L	0.3	0.1	75.5

A-A100/100S2 (100°C)





Element	Weight %	Atomic %	Error %
C K	37.3	47.0	10.5
O K	53.0	50.2	9.9
Mg K	0.1	0.1	80.8
Ca K	1.7	0.7	49.0
Fe K	7.3	2.0	20.7
Zn L	0.5	0.1	73.9

Sample	SEM image (x4000)	EDS/EDX image	eZAF Quant Result - Analysis Uncertainty: 9.40 %
A-A50/60S2 (60°C)			Element / Weight % / Atomic % / Error % C K 37.4 46.0 10.4 O K 56.5 52.2 9.7 Mg K 0.2 0.1 74.7 Ca K 0.9 0.3 69.5 Fe K 4.9 1.3 68.6 Zn L 0.1 0.0 84.9
A-A100/60S2 (60°C)			Element / Weight % / Atomic % / Error % C K 38.0 47.8 10.4 O K 52.4 49.4 9.8 Mg K 0.2 0.1 72.2 Ca K 1.4 0.5 63.9 Fe K 7.8 2.1 32.2 Zn L 0.2 0.0 75.3
After 5 wash cycles			
A-A50/100S5 (100°C)			Element / Weight % / Atomic % / Error % C K 37.7 47.8 10.4 O K 51.4 48.9 9.8 Mg K 0.4 0.3 67.5 Ca K 2.0 0.8 40.8 Fe K 8.0 2.2 28.8 Zn L 0.4 0.1 72.7
A-A100/100S5 (100°C)			Element / Weight % / Atomic % / Error % C K 32.1 41.8 12.7 O K 55.7 54.6 9.8 Mg K 0.1 0.1 92.6 Ca K 1.4 0.6 66.8 Fe K 10.4 2.9 22.8 Zn L 0.3 0.1 87.4
A-A50/60S5 (60°C)			Element / Weight % / Atomic % / Error % C K 38.1 47.2 10.3 O K 54.7 50.8 9.8 Mg K 0.1 0.0 89.7 Ca K 1.3 0.5 63.8 Fe K 5.7 1.5 41.5 Zn L 0.1 0.0 76.0
A-A100/60S5 (60°C)			Element / Weight % / Atomic % / Error % C K 37.8 48.0 10.5 O K 51.2 48.8 9.9 Mg K 0.1 0.1 74.8 Ca K 1.5 0.6 62.1 Fe K 9.2 2.5 20.3 Zn L 0.2 0.0 82.9

CONCLUSIONS

After 1, 2 and 5 washing cycles, textile samples treated with bioemulsions based on plant extract mixtures continue to exhibit antibacterial effect, which indicates good fixation of the bioactive compounds on the support. The intensity of this effect is maintained for all the analyzed variants, but is mainly influenced by the concentration of the bioemulsion, the samples treated at higher concentrations showing both a more pronounced antibacterial activity and a superior durability to washing.

Technological parameters play an essential role in determining the efficiency of the treatment and its durability, influencing both the degree of immobilization of the active compounds on the textile support and their stability to washing, aspects highlighted by correlating the results obtained by FTIR-ATR and SEM coupled with EDS/EDX, as follows:

- the amount of immobilized bioactive compounds, highlighted by FTIR-ATR spectroscopy, increases with the concentration of the bioemulsion;
- the drying operation performed after padding, at 100°C, leads to more efficient fixation and increased washing stability, through improved anchoring of the compounds on the surface of the textile support;
- high temperatures may cause a partial decrease in antibacterial activity, associated with the volatilization or degradation of some bioactive compounds from the composition of the plant extracts constituting the bioemulsion.

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