# INNOVATIVE TECHNOLOGIES FOR OBTAINING STRUCTURED EMULSIONS, BASED ON SEA BUCKTHORN EXTRACT AND TENSIDES

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**INNOVATIVE TECHNOLOGIES FOR OBTAINING STRUCTURED EMULSIONS, BASED ON SEA BUCKTHORN EXTRACT AND TENSIDES**

ABSTRACT. New structured emulsions were obtained by innovative technologies based on sea buckthorn extract (oil) and two surfactants, sodium dodecyl sulfate and Tween® 80 mixture: Es – sea buckthorn oil/sodium dodecyl sulfate/water; Et – sea buckthorn oil/Tween® 80/water; Est – sea buckthorn oil/sodium dodecyl sulfate and Tween® 80 (ratio 1:1)/water, for different concentrations of sea buckthorn extract oil, in order to improve surface properties with applications in leather industry. Sea buckthorn extract (oil) has a strong antimicrobial and antifungal effect due to its content in: vitamins C and E, phytosterols, fatty acids, antioxidants and amino acids. The order of introduction of the components in innovative technologies, the working conditions and especially the choice of the concentration of surfactants >CMC, are essential in the solubilization of sea buckthorn oil and in obtaining the structured emulsions. The emulsions were characterized by optical microscopy with sea buckthorn oil at 23-50°C. The changes in the aggregation process were observed for each type of emulsion, the influence of temperature and the solubilization of sea buckthorn oil. Dynamic light scattering (DLS) for the emulsions showed the stability, concentration, particle size, polydispersity, zeta potential. The antimicrobial properties were analyzed by microbiological tests. FTIR measurements highlighted the interaction mechanism of surfactants with sea buckthorn oil from the structured emulsions. The leather samples were microbiologically tested, and antimicrobial and antifungal effects were observed. The new structured emulsions are original due to the successful inclusion of sea buckthorn extract (oil) with high potential for improved surface properties with applications in the leather industry.
KEYWORDS: new structured emulsions, innovative technologies based on sea buckthorn extract and tensides, leathers processed

**TEHNOLOGII INOVATOARE PENTRU OBȚINEREA UNOR EMULSII STRUCTURATE PE BAZĂ DE EXTRACT DE CĂTINĂ ȘI TENSIDE**

REZUMAT. S-au obținut noi emulsii structurate prin tehnologii inovatoare bazate pe extract de cătină (ulei) și doi agenți tensioactivi, dodecil sulfat de sodiu și amestec Tween® 80: Es – ulei de cătină/dodecil sulfat de sodiu/apă; Et – ulei de cătină/Tween® 80/apă; Est – ulei de cătină/dodecil sulfat de sodiu și Tween® 80 (raport 1:1)/apă, pentru diferite concentrații de extract de cătină, în scopul îmbunătățirii proprietăților de suprafață cu aplicații în industria de pielărie. Extractul (uleiul) de cătină are un puternic efect antimicrobian și antifungic datorită conținutului său în: vitaminele C și E, fitosteroli, acizi grași, antioxidanți și aminoacizi. Ordinea introducerii componentelor în tehnologiile inovatoare, condițiile de lucru și mai ales alegerea concentraţiei de surfactanţi >CMC sunt esenţiale în solubilizarea uleiului de cătină şi obţinerea emulsiilor structurate. Emulsiile au fost caracterizate prin microscopie optică cu ulei de cătină la 23-50°C. S-au observat modificările procesului de agregare pentru fiecare tip de emulsie, influența temperaturii și solubilizarea uleiului de cătină. Dispersia dinamică a luminii (DLS) a arătat stabilitatea, concentrația, dimensiunea particulelor, polidispersitatea și potențialul zeta ale emulsiilor. Proprietățile antimicrobiene au fost analizate prin teste microbiologice. Măsurătorile FTIR au evidențiat mecanismul de interacțiune al agenților tensioactivi cu uleiul de cătină din emulsiile structurate. Probele de piele au fost testate microbiologic și s-au observat efecte antimicrobiene și antifungice. Noile emulsii structurate sunt originale datorită includerii cu succes a extractului de cătină (ulei), cu potențial ridicat de îmbunătățire a proprietăților de suprafață cu aplicații în industria de pielărie.
CUVINTE CHEIE: noi emulsii structurate, tehnologii inovatoare bazate pe extract de cătină și tenside, piei prelucrate

**TECHNOLOGIES INNOVANTES POUR L'OBTENTION D'ÉMULSIONS STRUCTURÉES À BASE D'EXTRAIT D'ARGOUSIER ET DE TENSIOACTIFS**

RÉSUMÉ. De nouvelles émulsions structurées ont été obtenues par des technologies innovantes à base d'extrait (huile) d'argousier et de deux tensioactifs, dodécylsulfate de sodium et mélange Tween® 80 : Es – huile d'argousier/dodécylsulfate de sodium/eau ; Et – huile d'argousier/ Tween® 80/eau ; Est – huile d'argousier/dodécylsulfate de sodium et Tween® 80 (rapport 1:1)/eau, pour différentes concentrations d'extrait d'argousier, afin d'améliorer les propriétés de surface avec des applications dans l'industrie du cuir. L'extrait (huile) d'argousier a un fort effet antimicrobien et antifongique grâce à sa teneur en : vitamines C et E, phytostérols, acides gras, antioxydants et acides aminés. L'ordre d'introduction des composants dans les technologies innovantes, les conditions de travail et surtout le choix de la concentration en tensioactifs >CMC sont essentiels à la solubilisation de l'huile d'argousier et à l'obtention des émulsions structurées. Les émulsions ont été caractérisées par microscopie optique avec de l'huile d'argousier à 23-50°C. Les changements dans le processus d'agrégation ont été observés pour chaque type d'émulsion, l'influence de la température et la solubilisation de l'huile d'argousier. La diffusion dynamique de la lumière (DLS) a montré la stabilité, la concentration, la taille des particules, la polydispersité et le potentiel zêta des émulsions. Les propriétés antimicrobiennes ont été analysées par des tests microbiologiques. Les mesures FTIR ont mis en évidence le mécanisme d'interaction des tensioactifs avec l'huile d'argousier dans les émulsions structurées. Les échantillons de cuir ont été testés microbiologiquement et des effets antimicrobiens et antifongiques ont été observés. Les nouvelles émulsions structurées sont originales en raison de l'inclusion réussie d'extrait (huile) d'argousier avec un potentiel élevé d'amélioration des propriétés de surface avec des applications dans l'industrie du cuir.
MOTS CLÉS : nouvelles émulsions structurées, technologies innovantes à base d'extrait d'argousier et de tensioactifs, traitement des cuirs

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## INTRODUCTION

This paper presents innovative technologies to create new structured emulsions, based on sea buckthorn extract (oil) and two tensides: sodium dodecyl sulfate and/or Tween® 80, in order to improve surface properties with applications in leather industry.

Sea buckthorn (*Hippophae*) has its origins in China, but we can also find it in temperate areas of the world: Russia, Mongolia, Finland, France [1-4]. Sea buckthorn is a thorny shrub with yellow or orange berries, the leaves are long lanceolate and can reach a height of 7 m. *Hippophae rhamnoides* has nine subspecies that differ based on genetic variations [5-7]. Sea buckthorn is also called *Siberian pineapple* because of berries that have a bitter-sour taste similar to that of pineapple. Due to the dense arrangement of the berries and thorns (Figure 1), harvesting the berries is cumbersome and is done once every two years [8-10].

Figure 1. Sea buckthorn branch with berries [11]

Sea buckthorn is rich in bioactive compounds present in fruits, seeds and leaves. In sea buckthorn fruits we find a rich content of: ascorbic acid, phytosterols, carotenoids, flavonoids, polyphenols, caffeic acid, ferulic acid [12-24]. Buckthorn berries contain: 23% seeds; 7% peel, 69% pulp. Due to its nutritional properties and beneficial effect on health, sea buckthorn is used in cosmetics, medicine, pharmacy and food [25-32].

Tween 80 [33] is a polyethylene sorbitol ester, also known as Polysorbate 80, PEG (80) sorbitan monooleate, polyoxyethylenesorbitan monooleate. It has been used as emulsifying agent for the preparation of stable oil-in-water emulsions. Tween is a group of non-volatile surfactant derivatives derived from glycerol esters. The most important usage of Tween is its application as an oil absorber and emulsifier.

Sodium dodecyl sulfate (SDS), also called sodium lauryl sulfate (SLS), having the formula: $CH_3(CH_2)_{11}OSO_3Na$ is an anionic tenside used as a cleaning and hygiene products [33].

In this research the new structured emulsions created and leathers processed with them were analyzed by FTIR-ATR spectroscopy, DLS, optical microscopy and microbiological tests.

## EXPERIMENTAL

### Materials and Methods

In order to obtain new structured emulsions the following materials were used: sodium dodecyl sulfate and Tween 80 from Sigma-Aldrich; sea buckthorn oil from "BIOCA" company.

The experimental techniques used in this paper consist in:

- "MALVERN" zetasizer-nano equipment, with measuring range between 0.3 nm-60.0 microns and zeta potential determination with an accuracy of +/-2%;
- JASCO FTIR-ATR spectrophotometer;
- optical microscopy with an ELTA 90 Medical Research S.R.L. equipment.

A number of three samples of emulsions – Es, Et, Est – were prepared in the following working conditions: sodium dodecyl sulfate or/and Tween 80 at 1:1 ratio, temperature=50°C for 30 minutes with sea buckthorn extract (oil), Figure 2.


a) b) c) d)

Figure 2. Image of new structured emulsions: a) Es – sea buckthorn oil/sodium dodecyl sulfate/water; Et – sea buckthorn oil/Tween® 80/water; Est – sea buckthorn oil/sodium dodecyl sulfate and Tween® 80 (ratio 1:1)/water; b) sea buckthorn oil; c) sodium dodecyl sulfate; d) Tween 80

## RESULTS AND DISCUSSION

### Obtaining New Structured Emulsions Based on Sea Buckthorn Oil and Tensides

Aqueous emulsions were obtained using two tensides, sodium dodecyl sulfate and Tween 80, in which sea buckthorn oil was introduced. According to novel innovative technologies in Figure 3, three types of new structured emulsions were made: Es, Et, Est.

The antimicrobial and antifungal effects were improved with the increase in the amount of sea buckthorn oil.

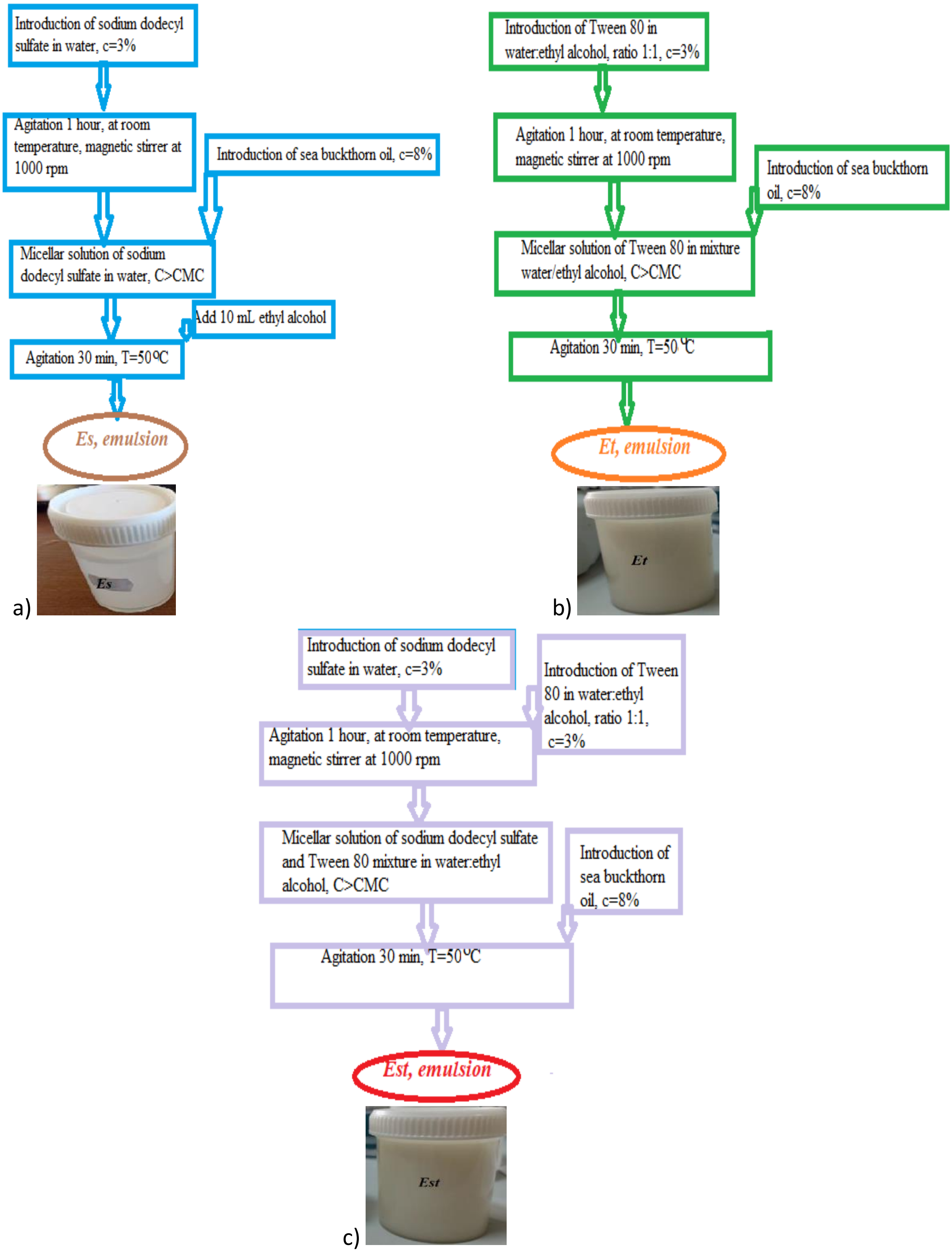



Figure 3. Innovative technologies for obtaining three types of emulsions with sea buckthorn oil:

a) Es; b) Et; c) Est

The Est emulsion variant with the two tensides sodium dodecyl sulfate and Tween 80 in a 1:1 ratio was selected because it is the most stable over time (1 month). The way of introducing tensides and sea buckthorn oil in obtaining emulsions is very important. The surfactant micellar solution is always made in water at a concentration above the micellar critical concentration – CMC and then the vegetable oil is added drop by drop and mixed. The chosen temperature is 50°C for a good solubilization of the vegetable oil in the surfactant micelles. When there are two tensides, micellar solutions in water are made separately for them, then the two solutions are mixed and mixed micelles in water are obtained. In the solution of mixed micelles, the vegetable oil is introduced drop by drop, stirring at the appropriate temperature. In the end, the emulsion is obtained with sea buckthorn oil solubilized in the mixed micelles. The yield of multiple drop formation decreases rapidly as the homogenization time increases. Structured emulsions are formed and the properties derive from the surfactants used, as well as the conditions and working parameters. This phenomenon is controlled by the concentration of: sea buckthorn oil, surfactants**,** temperature, pH=4.

## Mechanism of Sea Buckthorn Oil Solubilization in Tenside Micelles

In this research, the interaction of sea buckthorn oil with two tensides, sodium dodecyl sulfate and Tween 80, was investigated. A mechanism for the solubilization of sea buckthorn oil in tenside micelles was proposed, Figure 4. The effect of the length of the carbon chain on the interaction was analyzed by FTIR-ATR spectroscopy. The experimental results suggested that Tween 80 was most efficient out of the two tensides taken for the study. The order of stability is given as sea buckthorn oil – Tween 80 > sea buckthorn oil – sodium dodecyl sulfate.

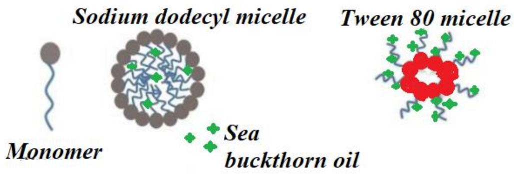


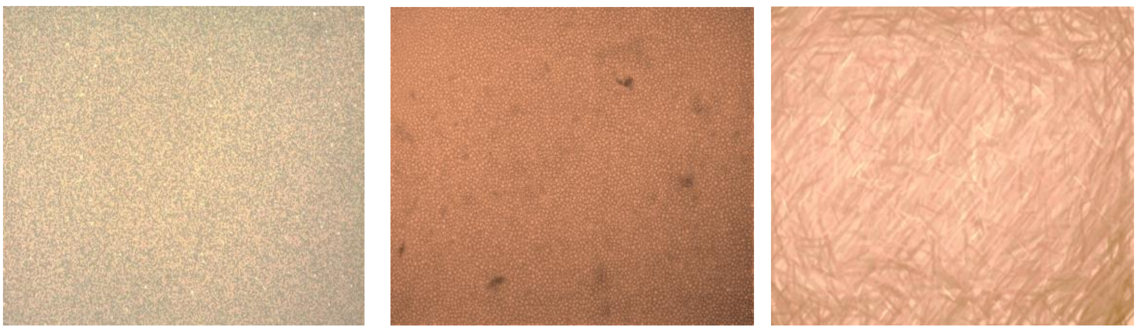

Figure 4. Proposed mechanism for solubilization of sea buckthorn oil in tensides: sodium dodecyl sulfate or Tween 80

Sea buckthorn oil is hydrophobic and gets stuck in the core of the micelles but also on the alkyl ends of the hydrophobic chains. For Tween 80, the amount of solubilized sea buckthorn oil is higher than in the case of sodium dodecyl sulfate, due to interaction forces.

## Characterization of the Structured Emulsions Obtained and the Leathers Processed

### *Optical Microscopy Analyses of Structured Emulsions*

The optical microscopy images from Figure 5 (a-f) show that all three emulsions obtained (at room temperature or 50 degrees) are structured like: irregular shapes (a, d), layer of balls (b, e) or bunches of needles (c, f) due to the influence of interaction between tensides and sea buckthorn oil.



a) b) c)

d) e) f)

Figure 5. Optical microscopy images of emulsions: a) Es at room temperature; b) Et at room temperature; c) Est at room temperature; d) Es at T=50ºC; e) Et at T=50ºC; f) Est at T=50ºC

*Dynamic Light Scattering (DLS) of Structured Emulsions*

The average particle sizes of new structured emulsions showed dimensions between (10-2075 nm), confirming the formation of the complex aggregates, Table 1.

The three types of emulsions were analysed by dynamic light scattering (DLS), Table 1.

Table 1: Results of DLS for three emulsions: Es, Et, Est

| Sample | Average diameter (nm) | % Intensity | Zeta Potential (mV) |
|---|---|---|---|
| Es | 21 | 10 | -55 |
| | 15 | 70 | |
| | 307 | 20 | |
| Et | 58 | 15 | -38 |
| | 400 | 85 | |
| Est | 800 | 29 | -70 |
| | 2075 | 71 | |

*Characterization by FTIR-ATR Spectroscopy of Leathers Processed with Emulsions*

The leathers were processed by spraying with the three obtained emulsions: Es, Et, Est, and were marked Esl, Etl, Estl (Figure 6) and then analyzed spectrophotometrically by FTIR-ATR.

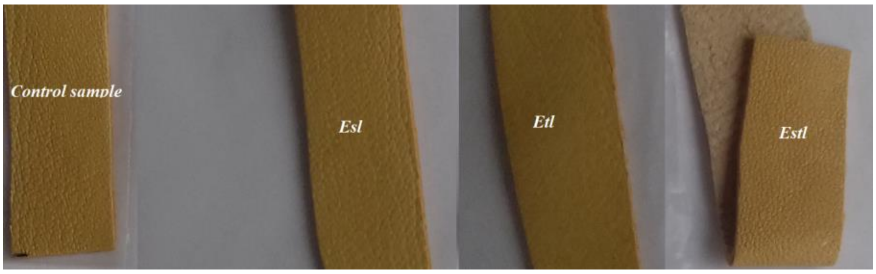



Figure 6. Image of the leathers processed with three structured emulsions: Es, Et, Est, and a control sample

From Figure 7 it can be seen that the largest amount of sea buckthorn oil is found in the leather treated with the Est emulsion (the spectrum intensity is the highest in the entire spectral range). The order of the sea buckthorn oil on the leather for the three emulsions is as follows: Estl>Etl>Esl.

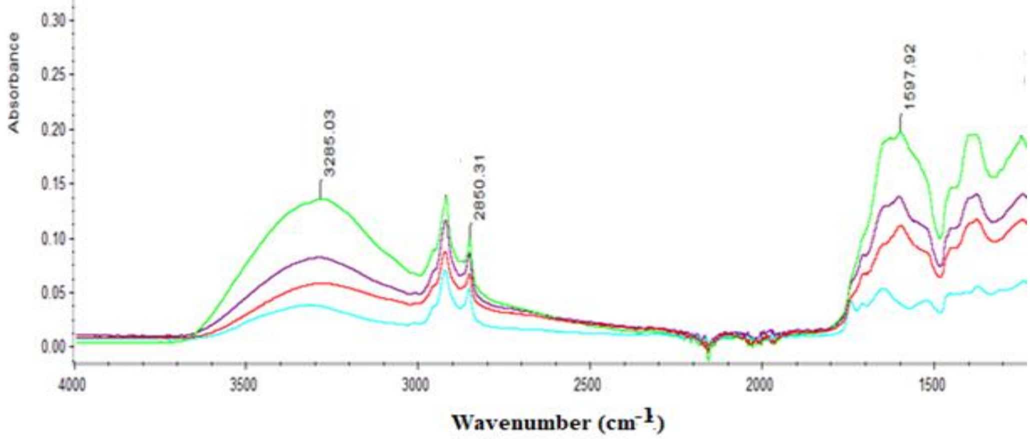



Figure 7. Overlay of FTIR-ATR spectra for leathers processed with emulsions: Esl---, Etl---, Estl--- control sample---

The Est emulsion is also the most stable, >1 month. The absorption maximum at the wavenumber ~1597 cm$^{-1}$ is the result of the overlap of the $CH_2$ deformation with the asymmetric $CH_3$ deformation (the intensity of the absorption maximum being proportional to the number of $CH_2$ and $CH_3$ groups present).

The range of wavenumbers: 3400–3500 cm$^{-1}$ is specific to sea buckthorn oil that has a high content of phenolic compounds and flavonoids. The absorption maximum at the wavenumber 2850 cm$^{-1}$ represents a weak skeletal vibration of isopropyl ($R_1R_2C=CHR_3$), the deformation being out of plane for undeformed, weakly strained systems, i.e. for cyclohexene derivatives.

*Microbiological Tests of Leathers Processed with Emulsions*

The microbiological tests of leathers processed with three emulsions against the attack of *Staphylococcus aureus* ATCC 6538 and *Aspergillus niger* are presented in Table 2.

Table 2: Results of microbiological tests of leathers processed with three emulsions: Es, Et, Est

| Sample | Result, UFC/mL | R% | $Log_{10}$ red. | Sample | Result, UFC/mL | R% | $Log_{10}$ red |
|---|---|---|---|---|---|---|---|
| *Aspergillus niger*, Inoculum concentration | $T_0$=9.8x10$^3$ | - | - | *Staphylococcus aureus,* Inoculum concentration | $T_0$=9.3x10$^3$ | - | - |
| Esl | $T_{24}$=3.4x10 | 99.65 | 2.46 | Esl | $T_{24}$=4.2x10 | 99.55 | 2.35 |
| Etl | $T_{24}$=0 | 100 | 4 | Etl | $T_{24}$=2 | 99.98 | 3.67 |
| Estl | $T_{24}$=2.18x10$^2$ | 97.78 | 1.44 | Estl | $T_{24}$=1.98x10$^2$ | 97.87 | 1.67 |

## CONCLUSIONS

1. The aim of this research was fulfilled to develop new emulsions and to study the influence of tensides and sea buckthorn oil in obtaining structures like: irregular shapes, layer of balls or bunches of needles. The structures of new emulsions were demonstrated by optical microscopy.

2. The emulsions with particle sizes of 10-2075 nm were obtained by DLS tests.

3. The new multiple structured emulsions are original due to the successful inclusion of sea buckthorn oil, with applications in the leather industry.

4. A mechanism of solubilization of sea buckthorn oil in tenside micelles was proposed. Sea buckthorn oil is hydrophobic and gets stuck in the core of the micelles but also on the alkyl ends of the hydrophobic chains. For Tween 80, the amount of solubilized sea buckthorn oil is higher than in the case of sodium dodecyl sulfate, because it has a larger hydrophobic chain, for which Van der Waals interaction forces are responsible.

5. The changes in the aggregation (structured) process were observed for each type of emulsion (Es, Et, Est), the solubilization

of sea buckthorn oil by dynamic light scattering and optical microscopy.

6. In the process of finishing the leathers by spraying with three types of structured emulsions obtained compared to an untreated leather, the antifungal and antimicrobial properties, as well as the softness and appearance of the leathers were improved.

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