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RHEOLOGICAL STUDY OF THREE COSMETIC CREAMS OBTAINED WITH DIFFERENT EXTRACTS OF LEMON

BY

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Abstract. In this study, three cosmetic creams containing/with different lemon extracts were formulated and studied from a rheological point of view. Rheological tests such as: the flow test, amplitude sweep and frequency sweep were determined by specific measurements and showed that the products formulated based on lemon extracts are stable at different temperatures and can easily be applied to the skin. Additional microbiological tests and clinical studies are required for cosmetic creams to be marketed.

Keywords: rheological study, lemon extract, cosmetic cream, oily maceration.

1. Introduction

Creams are frequently used in cosmetics to clean, nourish, protect and soothe the skin. In order to perform these functions, cosmetic creams must meet several conditions such as: being harmless to the skin and body, being easily absorbed, not being rancid, having a very high degree of homogenization and being able to form a protective and emollient layer in any season (Merica, 2003).

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The composition and structure of cosmetic creams is closely related to the behaviour of emulsions because the vast majority of cosmetic creams are heterogeneous emulsion-type systems. In addition to the basic components, water and oil that form the emulsion, the creams also contain emulsifiers and surfactants that ensure the stability of the emulsion, but also different active substances extracted from plant materials that give them superior properties. All this contributes to the rheological behaviour of creams, and studies in the specialized literature confirm this (Morávková and Stern, 2011; Cobzaru *et al*., 2017; Cobzaru *et al*., 2022; Biglarian *et al*., 2022; Cobzaru *et al*., 2023; Andonova *et al.,* 2024; Mariano *et al*., 2024). Moreover, Lungu and Merica (2000) state that the rheological characteristics of a cream must be known by both producers and consumers because they give information about the flow and stability of the products.

Citrus fruits are plant materials that have a composition rich in active compounds such as vitamin C, citric acid, minerals, essential oils, alkaloids, flavonoids, carotenoids, enzymes and phenolic compounds, giving the final creams antioxidant, antimicrobial, anti-inflammatory and antiviral activities (Palazzolo *et al*., 2013; Oikeh *et al*., 2016; Moosavy *et al*., 2017; Rafique *et al*., 2020; Soares Mateus *et al*., 2024; Munir *et al.,* 2024; Sudeshna *et al*., 2024). Due to these properties, citrus fruits can be used to manufacture cosmetic creams or other cosmetic products (Buccellato, 2000; Klimek-Szczykutowicz *et al.,* 2020; Ben Hsouna *et al*., 2023; Šafranko *et al*., 2023; d'Avanzo *et al*., 2024; Leong *et al.,* 2024).

Thus, in the present study the rheological behaviour of three cosmetic creams formulated using different lemon extracts was measured. Lemon extracts were obtained by the means of three extraction methods, namely infusion, maceration in sunflower oil and Soxhlet extraction. The obtained cosmetic creams were tested from a rheological point of view, measuring characteristics such as: flow test, amplitude sweep and frequency sweep at different temperatures.

Through this study, the importance of using extracts in the preparation of cosmetic creams is analysed, because they improve the properties of the cream, but also can be easily obtained from different plant materials. For example, a series of active compounds from lemon extracts such as vitamin C, citral, pinene, limonene, etc., are considered powerful antioxidants that can support collagen production and remove pigment spots from the skin (Klimek-Szczykutowicz *et al.,* 2020). However, if the cosmetic creams prepared in this study are to be used for commercialization/selling on the market, further microbiological tests and clinical studies are required.

2. Experimental

2.1. Selection and preparation of plant material

The lemons used in this study were purchased from the supermarket. In order to obtain the extracts, first, the fruits were washed, dried and then grated. The obtained peel was dried in an oven at t = 40°C for 60 minutes in order to remove moisture. After drying, the material was stored in a closed jar in the fridge, until used.

2.2. Extraction apparatus and solvents

Lemon peel extracts were obtained by the means of three extraction methods, namely infusion, maceration in sunflower oil and Soxhlet extraction. Infusion and maceration were performed in a glass flask with ground glass joint, and Soxhlet extraction was performed in a specific laboratory construction.

Distilled water was used for the infusion, and the sunflower oil used for the maceration was purchased from the supermarket and it was from the 2022 production. The solvent used for the Soxhlet extraction was 96^{o} ethyl alcohol purchased from Merck and diluted to 70^{o}, according to the specialized literature (Horoba and Horoba, 2010). The alcohol was diluted to 70^{o}, because this is the optimum concentration needed to extract the active principles from the plant materials (Petkova-Parlapanska *et al*., 2014).

The beeswax required for the preparation of the cosmetic cream was purchased from local producers, production year 2021, and substances such as borax and sodium dodecyl sulphate (surfactant) were purchased from Merck.

The rheology tests were carried out with an "Anton Paar Physica MCR 501" modular rheometer, equipped with a CTD 600 system, or with a Peltier system for temperature regulation. The measurements were performed with a plane-plane geometry with striations, with a 50 mm diameter. The tests were carried out both in oscillatory mode and in rotational mode in the Rheology Laboratory on the Interdisciplinary Training and Research Platform "High-performance multifunctional polymeric materials for medicine, pharmacy, microelectronics, energy/information storage, environmental protection (MATMIP)" from the Department of Natural and Synthetic Polymers of the "Cristofor Simionescu" Faculty of Chemical Engineering and Environmental Protection Iasi.

2.3. Obtaining extracts from dried lemon peel

a) Maceration in sunflower oil. The dry product was placed in a glass container with a lid where sunflower oil was added in a ratio material:oil=1:12.5. The mixture was kept in a dark and cold place for 14 days, stirring periodically.

At the end of the extraction period it was filtered and the oily extract was kept in a glass container for use.

b) Infusion in warm water. The dried lemon peel was placed in a Berzelius flask, together with distilled water heated to 70°C, for 15 minutes, in a ratio of material:solvent=1:10, after which it was filtered and the residue was pressed. Finally, the aqueous extract was refrigerated for 24 h before use.

c) Extraction with ethyl alcohol in a Soxhlet extractor. Dry lemon peel was inserted into the Soxhlet cartridge, it was covered with cotton wool and then inserted into the extractor part of the apparatus, on the upper side. Through the extractor, ethyl alcohol of 70^{o} was introduced over the sample cartridge in a ratio material: alcohol=1:30, performing a first siphoning in order to moisten the plant material and release the active compounds more easily. Next, the ascending refrigerant was connected and the heating was turned on until the boiling temperature of the solvent (approx. 80^{o}C). The extraction was carried out for 4 hours with a speed of 2 siphonings/hour.

2.4. Obtaining cold cream with lemon peel extract

The cosmetic creams were obtained according to the method described in literature (Cernatescu, 2016). Basically, beeswax and vegetable oil are introduced into a laboratory flask, in a ratio of 1:30 (m/m), and the mixture is heated to t = 68^{o}C, under continuous stirring. Separately, a solution is prepared by diluting the borax and surfactant in the amount of water prescribed in the recipe. The two substances are added in proportions of 0.2% and 0.4% (m/m), respectively. When the wax and oil mixture has warmed to the temperature indicated in the recipe, the solution of borax and surfactant was added, under vigorous stirring. Next, the cream is removed from the heat source and stirred continuously until it reaches the room temperature, then it is transferred into plastic containers.

For the creams that are the subject of the present study, some changes were made, namely, the necessary water was replaced with infusion, and as vegetable oil with oily maceration and coconut oil were used, both individually and in a mixture according to Table 1.

Table 1

Ingredients of cosmetic creams obtained

Name	Bees wax	Vegetable Oil	Water	Borax	Tensioactive	Concentrated lemon peel Alcoholic Extract
Cream 1	yes	coconut oil	infusion	yes	yes	No
Cream 2	yes	coconut oil /oily maceration, ratio 1:1	infusion	yes	yes	No
Cream 3	yes	oily maceration	infusion	yes	yes	1-5 drops

3. Results and Discussions

Figure 1 shows the cosmetic creams with lemon peel extracts prepared for the study.

Fig. 1 − Cosmetic creams with lemon peel extracts.

From Fig. 1 it can be seen that each cosmetic cream has its own distinct colour. Thus, the cream with coconut oil (Cream 1) has a very light-yellow colour, the cream with a mixture of coconut oil and oily lemon maceration (Cream 2) has a more pronounced yellow colour and the one in which only the oily maceration was used (Cream 3) has a dark yellow colour. All creams are homogeneous and have a good viscosity. As an informative note, the creams perform very well when tested on the skin, being easily absorbed, giving a feeling of softness and a fresh citrus smell that persist for a long time.

Rheological testing of creams formulated with dried lemon peel extracts

For the analysed samples, the *flow test* was firstly carried out, which is part of the category of rotational tests, materializing through a flow curve or more precisely, through a graphic representation of the flow behaviour of the samples. For this purpose, the sample to be analysed is subjected to an increasing or decreasing shear rate, the shear stress and viscosity being calculated by the parameters of the apparatus. The shape of the obtained curve indicates the type of flow behaviour exhibited by the sample, and it can be: ideal viscous, pseudoplastic or dilatant. The graphic representation of the flow behaviour of the three creams is presented in Fig. 2.

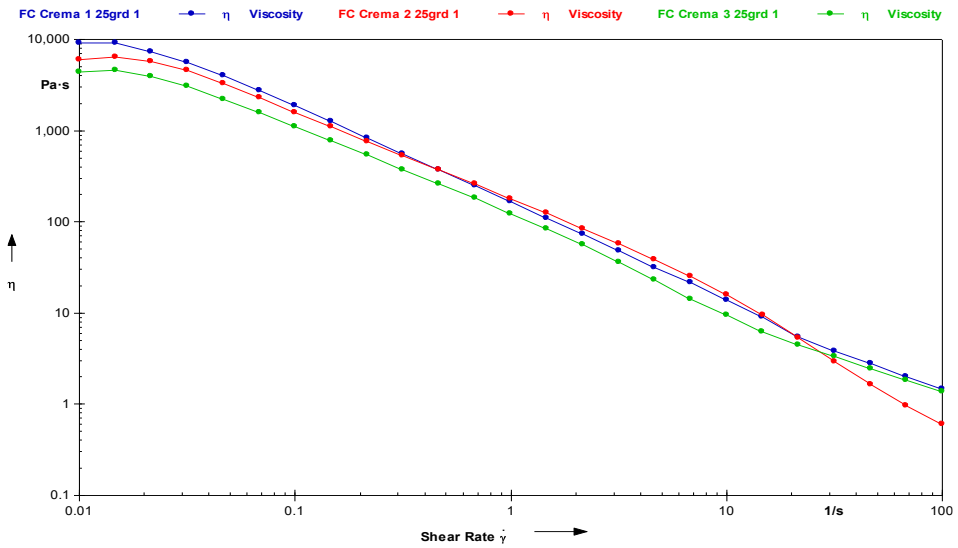
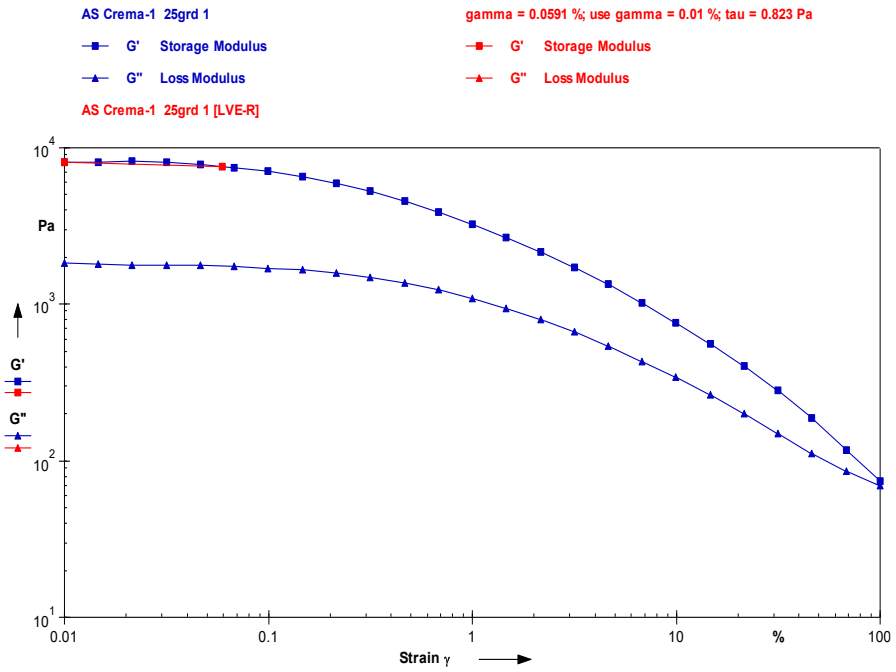


Fig. 2 − Flow curve for the three cream samples with lemon extract.

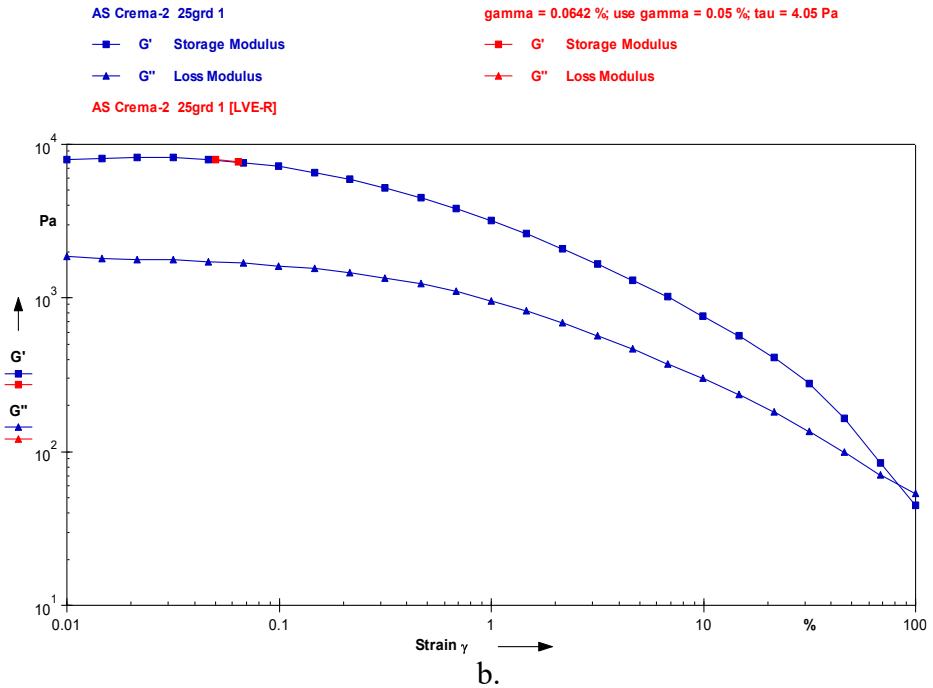
The graph show that the viscosity value decreases with the increasing of shear rate, which indicates a pseudoplastic behaviour. According to literature, this behaviour is specific, in general, to materials characterized by an irregular internal structure that opposes flow, until an external force acts on them (Lungu and Merica, 2000).

Another rheological test performed on cosmetic creams with lemon peel extract was the *amplitude sweep*, which is part of the class of oscillatory tests that describe the microstructure of the materials. The measured parameters specific to this test are: *the storage modulus* (G') - a measure of the deformation energy accumulated in the sample during shearing or more concretely, which represents the elastic behavior of the material, *the loss modulus* (G") - a measure of the energy of deformation used by the sample during shear or in other words, it represents the viscous behavior of the material, *the phase angle* (δ), the damping or loss factor that describes the ratio between the viscous and the elastic component of a viscoelastic behavior and the *complex viscosity* (η*) (Lungu and Merica, 2000).

The amplitude sweep test determines the linear viscoelastic range of the material (LVE), further establishing the parameters for the oscillatory tests to which the samples are to be subjected. In the case of the three creams, the test was carried out at a constant frequency and variable amplitude, as well as at a temperature of 25°C. Figure 3 shows the graphs of the amplitude sweep of the three analysed creams.


AS Crema-1 25grd 1
G' Storage Modulus
G" Loss Modulus
AS Crema-1 25grd 1 [LVE-R]
gamma = 0.0591 %; use gamma = 0.01 %; tau = 0.823 Pa
G' Storage Modulus
G" Loss Modulus
Pa
G'
G"
Strain γ
%

a.


AS Crema-2 25grd 1
G' Storage Modulus
G" Loss Modulus
AS Crema-2 25grd 1 [LVE-R]
gamma = 0.0642 %; use gamma = 0.05 %; tau = 4.05 Pa
G' Storage Modulus
G" Loss Modulus
Pa
G'
G"
Strain γ
%

b.

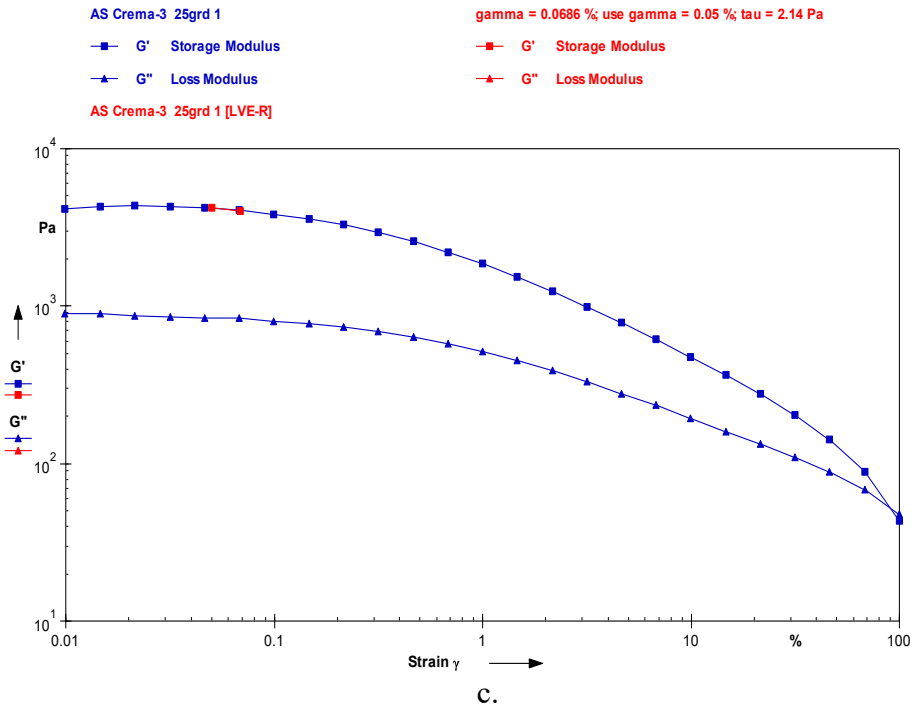


Fig. 3 − Amplitude sweep for: a) Cream 1; b) Cream 2; c) Cream 3.

As shown in Fig. 3, the samples exhibit a structural stability in the range of small deformations, the limit value of the linear viscoelastic range being in the range of 0.01-0.07%. However, in the case of cream 3 (Fig. 3c) a slight decrease in the values of the two dynamic moduli is observed, which indicates that this cream is characterized by a slightly softer consistency, with a softer and lighter appearance to apply on the skin. This can be attributed to the alcoholic extract that was introduced into the composition of the cream and the viscosity of the oily macerate.

The last rheological test carried out on the three creams is the *frequency sweep* through which information is obtained regarding the internal structure and molecular mass of the samples. In principle, the frequency sweep aims to vary the shear moduli (G' and G") and the tangent of the loss angle over a wide range of frequencies. The storage modulus (G') describes the elastic behaviour of the sample, providing information related to the stability and structural strength of the sample, and the loss modulus (G"), provides information regarding the viscous behaviour of the sample. By the ratio of the two modules, the tangent of the loss angle is obtained, tan(δ) = G"/G'. Figure 4 shows the graphs of the frequency sweep for the three analysed creams.

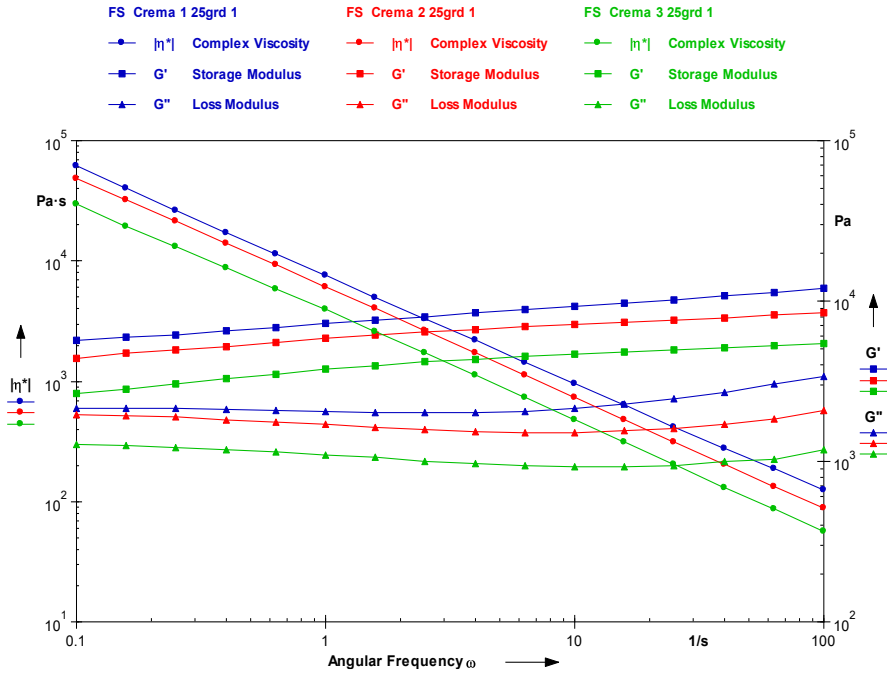


Fig. 4 − Frequency sweep for the three creams.

4. Conclusions

Three cosmetic creams with lemon peel extracts were formulated and studied, from a rheological point of view. Rheological tests such as flow test, amplitude sweep and frequency sweep were made by specific measurements and showed that the products formulated with lemon peel extracts are stable at different temperatures and do not flow, which makes them usable in skin care. The cream obtained only with lemon peel extracts is clearly superior in colour and texture to the other creams. Additional microbiological tests and clinical studies are required for cosmetic creams to be marketed.

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STUDIUL REOLOGIC A TREI CREME COSMETICE OBȚINUTE CU DIFERITE EXTRACTE DE LĂMÂIE

(Rezumat)

În acest studiu s-au formulat și studiat, din punct de vedere reologic, trei creme cosmetice cu extracte diferite de lămâie. Testele reologice precum testul de curgere, baleiaj de amplitudine și baleiaj de frecvență au fost determinate prin măsurători specifice și au arătat că, produsele formulate pe bază de extracte de lămâie sunt stabile la diferite temperaturi și că se pot aplica pe piele. În cazul comercializării cremelor cosmetice sunt necesare teste microbiologice și studii clinice suplimentare.