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STUDY ON OBTAINING OILY EXTRACT FROM LINDEN FLOWERS AND USING IT IN A COLD CREAM

BY

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Abstract. The extract of linden flowers was obtained by maceration in sunflower oil and, then, the extract was incorporated into a cold cream. The photodegradation, TGA-DTGA analyses as well as the iodine, acidity and saponification index, performed on the oily extract showed that it contains active plant compounds that can contribute to its long-term use. The cosmetic cream obtained with the oily extract of linden flowers demonstrated that, from a qualitative point of view, it is moisturizing and nourishing, beneficial for the skin but with a short shelf life (6 weeks).

Keywords: oily maceration, linden flowers, cold cream, photodegradation.

1. Introduction

Regardless of the species, linden flowers are one of the plants with the most uses since ancient times, due to their chemical composition rich in active substances and due to their therapeutic effects. Used both fresh and dried or in the form of extracts, linden flowers are a good remedy for treating a wide range

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of conditions such as: colds, migraines, insomnia, headaches, anxiety, rheumatism, cardiac arrhythmia, etc. (Coleta *et al*., 2001; Oniszczuk and Podgórski, 2015; Czerwińska *et al*., 2018; Hassanien *et al*., 2018). In addition to their specific actions, linden flowers are also used for skin treatments, having moisturizing, soothing, anti-inflammatory and emollient properties (Milică *et al*., 2005; Fan *et al.,* 2025).

In general, the Lindens tree is part of the *Tiliaceae* family, which includes about 40 genres and over 470 species. In Romania, the lindens tree is represented by a single genus (the genus *Tilia*) that includes a very large number of species and varieties, but only 3 of which are of particular importance, namely: the white lindens tree (*Tilia tomentosa Mill*.); the small-leaved lindens tree (*Tilia cordata Mill*.) and the large-leaved lindens tree (*Tilia platyphyllos Scop*.) (Milică *et al*., 2005; Ion, 2006). Also, lindens trees are found in the spontaneous flora of all natural areas of the country, in hardwood forests, or isolated on the edges of roads, in parks and gardens. Lindens tree flowers are rich in numerous active substances such as: phenolic compounds, vitamin C, flavonoids, volatile oil, etc. (Milică *et al*., 2005; Kruk *et al.,* 2022; Fan *et al.,* 2023; Ortiz-Islas *et al.,* 2024; Yankova-Nikolova *et al*., 2025).

In order to benefit from all those active compounds from a plant, various extraction techniques can be used, and studies show that, in general, the composition and biological activity of an extract depends on the extraction method and the solvent used (Sroka and Bełz, 2009; Gawlik-Dziki *et al*., 2012; Oniszczuk and Podgórski, 2015; Schoss and Glavac, 2024). Regarding the extraction of active compounds from linden flowers, especially phenolic compounds, literature studies show that ultrasonic and solvent-accelerated extraction are the most efficient techniques compared to microwave, Soxhlet extraction and infusion (Oniszczuk and Podgórski, 2015; Cittan *et al*., 2018). Both techniques are characterized by high extraction efficiency, reproducibility and rapid solvent recovery. In addition, the extractions are easy to perform, the equipment being relatively cheap and available in most laboratories.

These considerations justify the opportunity of our study, in which we propose to obtain an extract from linden flowers of the *Tilia cordata Mill*., variety by a simple method, namely by maceration at room temperature using a vegetable oil. The linden extract is then used to prepare a cosmetic product such as cold cream with therapeutic properties on the skin. We thus try to prove that through a simple technique it is possible to obtain an bioactive extract from linden flowers with minimal costs and which can be used in cosmetic products.

2. Experimental

2.1. Selection and preparation of plant material

For this study, linden flowers from the spontaneous flora of Botoșani County were used. They were naturally dried, in the sun, for 48 hours, being

turned periodically for uniform drying. Also, the sunflower oil purchased from the supermarket was used for extraction.

2.2. Obtaining oil extracts from linden flowers

The oily extract from linden flowers was obtained through a simple maceration process. In this regard, the dried and crushed linden flowers were previously placed in a glass container with a tight closure, over which sunflower oil was added in a mass ratio of 1:20. The mixture was left to macerate for 14 days at room temperature, shaking periodically (Fig. 1a).

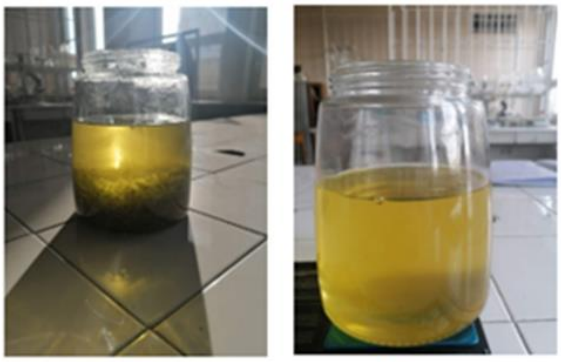

Fig. 1 − Maceration of dried linden flowers with sunflower oil.
a). 1st day. b). 14th day.

At the end of the maceration period, the mixture was filtered, and the oily extract from linden flowers was obtained with an extraction yield of 68% (Fig. 1b).

2.3. Analysis of oil extracts from linden flowers

The oil extracts from linden flowers were analyzed in terms of calculating acidity, saponification and iodine indexes as well as photodegradation and thermal degradation. The analyses regarding the acidity, saponification and iodine index were carried out in the Natural Extracts laboratory of the Faculty of Chemical Engineering and Environmental Protection "Cristofor Simionescu" Iași, according to the methods described in the specialized literature (Purcărea, 2015).

2.3.1. Determination of acidity index

A certain amount of sample was weighed into a Berzelius glass and then heated. A mixture of ethyl alcohol-benzene was added to the sample and then

titrated with a 0.1 N NaOH solution under continuous stirring in the presence of phenolphthalein, until a pink color appeared, that persisted for 30 seconds. For each type of oil, the titration was done three times, and the arithmetic mean of the results constituted the final value.

2.3.2. Determination of saponification index

A quantity of oily extract was introduced into a flask with ground glass joint, over which benzene and a KOH solution were added. An ascending refrigerant was added to the flask and its content was boiled on a water bath for 30 minutes, after which it was titrated with a HCl solution in the presence of phenolphthalein. In parallel, a blank sample was also obtained. Three titrations were performed, and the arithmetic mean of the results constituted the final value.

2.3.3. Determination of iodine index (Hanus method)

A quantity of oily extract was introduced into a vial to which chloroform and Hanus solution were added. In parallel, a control sample was prepared. The vials were sealed, shaken and kept in the dark for 30 minutes. Next, potassium iodide and distilled water were added, after which it was quickly titrated, under vigorous stirring, with sodium thiosulfate until a pale yellow color appeared. Starch was added and the titration was continued until the blue color due to the presence of iodine disappeared.

2.3.4. Photodegradation of linden oil extracts

The analysis was performed in the Inorganic Chemistry laboratory of the Faculty of Chemical Engineering and Environmental Protection "Cristofor Simionescu" Iași as follows: 50 mL of sample were irradiated with ultraviolet radiation generated by an 18W Hg UVB lamp, with the incident radiation intensity of 2.1 W/m^2. The distance between the UV radiation source and the samples is 2 cm. At periodic intervals, samples (approx. 4 mL) were taken and analyzed by UV-Vis spectroscopy (with a METERTECH SP 870 plus spectrophotometer). The samples were continuously magnetically stirred. The incident radiation intensity was determined to be 2.1 W/m^2 (Hamamatsu C9536-01, H9958 detector for 310-380 nm, calibrated between 1$\mu W/cm^2$ and 100 mW/cm^2).

2.3.5. Study of thermal degradation of linden oil extract

The study of the thermal degradation of the linden oil macerate was carried out at the National Institute for Research and Development for Cryogenic and Isotopic Technologies Rm. Vâlcea, by thermogravimetric measurements. For

this purpose, the oils were heated at 190°C for 0.5, 4.0 and 8.0 hours. Subsequently, TG/DTG curves were obtained using the Shimadzu TGA-50 apparatus in air (20 $mLmin^{-1}$) at a heating rate of 5°C/min in order to verify the influence of heating time and temperature.

3. Results and discussions

3.1. Determination of chemical indices in oily extracts from linden flowers

3.1.1. Determination of acid value and free acidity expressed as oleic acid

The free acidity of oils is an important index, which is due to the free fatty acids found in the product, and it must be low. The acidity index represents the amount of potassium hydroxide, expressed in milligrams, required to neutralize the free fatty acids in one gram of product. Table 1 presents the values of the acidity index and free acidity for the oily extract of linden flowers and sunflower oil.

Table 1
Acidity index and free acidity for the analyzed samples

No	Product	Acidity index mg KOH/g product	Acidity (acid oleic), %
1.	Linden flower oil extract	0.393	0.19
2.	Sunflower oil	0.605	0.304

In the case of the acidity index, it can be observed that its value is much lower than the one for the sunflower oil that was used for maceration. Therefore, it can be stated that simple maceration at room temperature still leads to an enrichment of the oil in active compounds observed by decreasing the amount of KOH necessary to neutralize the free fatty acids present in one milligram of the product. According to the specialized literature (Banu, 2002), oils whose acidity in oleic acid is 1% cannot be reused. In the case of the oily extract, the acidity is lower than that of the initial oil, which denotes the fact that the extract can be used.

3.1.2. Determination of saponification index

The saponification index represents the number of milligrams of potassium hydroxide required to saponify one gram of product. Table 2 presents the saponification index values for the linden flower extract and sunflower oil used in maceration.

Table 2
Saponification index for the analyzed samples

No	Product	Saponification index mg KOH/g product
1.	Linden flower oil extract	140.26
2.	Sunflower oil	189.351

As can be seen, the saponification index value for the extract is lower than that of the sunflower oil used for maceration, which denotes that the extract contains a smaller amount of saponifiable substances than the initial sunflower oil and implicitly a larger amount of active compounds.

3.1.3. Determination of iodine index (Hanus method)

The iodine value represents the amount of halogen, expressed in grams of iodine, added to 100 g of product. The method is based on the property of unsaturated acids that are part of the oils and fats to add halogens to the double bond. If the iodine value of an oil is known, an indication of its degree of unsaturation can be obtained. The presence of unsaturated acids gives oils certain properties, adversely affecting their resistance to storage for longer period of time. Table 3 presents the iodine value values for the two samples analyzed.

Table 3
Iodine index value for the analyzed samples

No	Product	*Iodine index*, g I_2/100 g product
1.	Linden flower oil extract	220.806
2.	Sunflower oil	194.157

From table 3 it can be seen that the iodine index value for linden extract is much higher, which indicates that, due to the content of active compounds, it cannot be preserved for a long time against oxidation. However, if it is still desired to preserve it for a long time, then a preservative must be added.

3.2. Determination of the stability of linden flower oil extracts to photodegradation

It is known that light is a determining factor in the chemical degradation of food products because the degradation reactions caused by it or the photodegradation reactions have a negative impact not only on the appearance and quality of food products but also on their chemical composition. Moreover, exposure to light poses the risk of rancidity, a phenomenon that can be accelerated by light, temperature, packaging, ionizing radiation, catalysts, etc. To test the

resistance of linden flower extracts to the action of UV light, they were subjected to photodegradation, and its effects were analyzed by UV-VIS spectroscopy.

Figure 2 shows the UV-VIS spectrum for the linden flower oil extract subjected to irradiation.

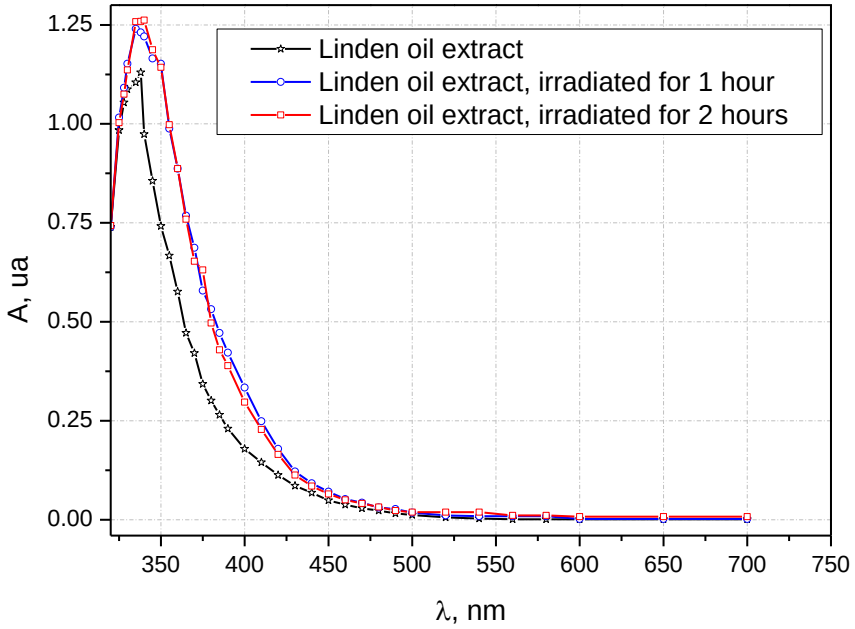

Fig. 2 − UV-VIS spectrum for linden oil extract.

As can be seen, the absorbance is found at the same wavelength for all three samples (oily extract, oily extract irradiated for 1 hour and oily extract irradiated for 2 hours), which demonstrates that the extract contains the same type of fatty acids that absorb UV light. The absorbance value is higher after photodegradation because more free fatty acids were obtained in the system under UV light. On the other hand, the value of absorbance for the two samples that undergoes photodegradation is almost the same, which conclude that the time of irradiation does not influence significatively the deterioration of triglicerides.

3.3. Study of thermal degradation of linden oil extract

Figure 3 shows the TG-DTG isotherms of the oily macerate of linden flowers.

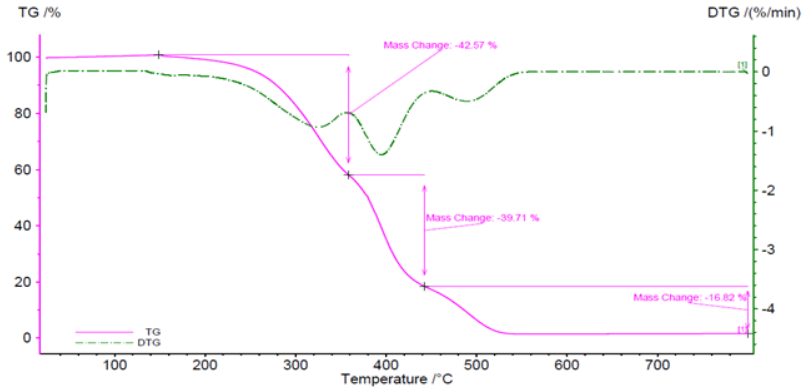

Fig. 3 − TG-DTG isotherms of linden oil macerate.

As can be seen, the first stage, in the range of 150-350ºC, corresponds to the decomposition of polyunsaturated fatty acids. Due to this fact, it can be stated that the thermal stability of the oily extract of linden flowers is dependent on the composition of fatty acids in the oil used for maceration. The second stage in the thermal decomposition of the oily macerate in the range of 350-450ºC corresponds to the decomposition of monounsaturated fatty acids, namely oleic acid. More specifically, during this reaction, the double bonds break, thus forcing the triglyceride molecules to become saturated. The third stage in the thermal decomposition of the oily extract 450-550ºC corresponds to the thermal decomposition of saturated fatty acids, such as palmitic acid.

3.4. Obtaining cold cream with linden flower extracts

The cold cream was obtained according to a recipe from the literature with some modifications, namely: the oil was replaced with linden oil extract and the water with an infusion of linden flowers. In addition, beeswax, borax, surfactant and alcoholic linden extract for smell were added in the quantities prescribed according to the recipe (Cernătescu, 2016). Finally, a product with a specific color, fine, creamy texture and a slight specific smell of linden flowers was obtained, which was stored in the refrigerator (Fig. 4).

Fig. 4 − Cold cream with linden flower extract.

This was tested on skin, demonstrating that, from a qualitative point of view, it is a homogeneous cream, with a pleasant smell and with moisturizing and nourishing properties. After 6 weeks of use, it was observed that it was no longer homogeneous and small changes in smell and texture appeared, which indicates that it may have a short shelf life due to the natural ingredients used and because we did not used preservatives.

4. Conclusions

The extract of linden flowers was obtained by maceration in sunflower oil, which was used in the preparation of a cold cream. The analyses regarding the iodine index, acidity and saponification, as well as the photodegradation and TGA-DTGA performed showed that it contains active compounds from the plant that can contribute to its long-term use. The cosmetic cream obtained with the oily extract of linden flowers demonstrated that, from a qualitative point of view, it is a moisturizing and nourishing cream, beneficial for the skin but with a short shelf life (6 weeks).

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STUDIUL PRIVIND OBȚINEREA ȘI UTILIZAREA EXTRACTULUI ULEIOS DIN FLORI DE TEI ÎNTR-O COLD CREMĂ

(Rezumat)

S-a obținut extractul din flori de tei prin macerare în ulei de floarea soarelui care a fost încorporat într-o cold cremă. Analizele de fotodegradare, TGA-DTGA precum și indicele de iod, de aciditate și de saponificare, realizate pe extractul uleios au arătat că este posibil ca această să conțină compuși activi din plantă care pot contribui la utilizarea sa un timp îndelungat. Crema cosmetică obținută cu extractul uleios din flori de tei a demonstrat că, din punct de vedere calitativ, este o cremă hidratantă și hrănitoare, benefică pentru piele dar cu durată de valabilitate scurtă (6 săptămâni).