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Utilization of Sunflower Oil Winterization Waxes as Functional Ingredients in Cosmetic Cream Formulations

Umirova Zilola[a], Akhmedov Azimjon[b*], Khakimova Zulfiya[a], Ruzibayev Akbarali[a]

[a] Department of Food and Perfumery-Cosmetic Products Technology, Tashkent Institute of Chemical Technology, Tashkent 100000, Uzbekistan
[b] Department of Food Products Technology, Karshi State Technical University, Karshi 180119, Uzbekistan
*a.ahmedov80@mail.ru

The present study focuses on the separation, purification, and application of waxy substances obtained as waste from the winterization of sunflower oil, with the aim of partially or completely replacing stearic acid in cosmetic cream formulations. A key challenge addressed in this research is the removal of perlite particles and the isolation of high-purity wax fractions suitable for cosmetic use. In the experimental work, a mixture of waxy substances and perlite obtained after filtration during winterization was subjected to different separation techniques. Conventional extraction with hexane was used as a control method, while aqueous solutions of sodium chloride (1–10%) were investigated as an alternative, environmentally friendly approach. The efficiency of separation was evaluated by varying solution concentration, temperature, and settling time. The isolated waxy substances were further characterized using chromatographic analysis, infrared (IR) spectroscopy, refractometry, and differential scanning calorimetry (DSC) to determine their composition and physicochemical properties. The results demonstrated that an 8% sodium chloride solution at a temperature of 20 °C and a settling duration of 10 hours provided optimal conditions for perlite removal. Under these conditions, the yield of purified waxy substances reached 98.5% relative to the conventional hexane extraction method. Importantly, the use of aqueous salt solutions significantly reduces the emission of toxic organic solvents, thereby improving environmental safety and process sustainability. The purified waxy substances were subsequently incorporated into cosmetic cream formulations as a partial or full substitute for stearic acid. It was found that replacing up to 50% of stearic acid with the recovered waxy material did not adversely affect the rheological or sensory properties of the creams. However, complete substitution led to a noticeable decline in product performance, particularly in terms of spreadability and skin absorption characteristics.

1. Introduction

Sunflower oil contains a significant amount of waxy substances that negatively affect the clarity and stability of refined oil. To ensure product quality, the oil undergoes a winterization process, during which wax crystals are removed using filtration aids such as perlite. As a result, a solid waste consisting of perlite and waxy substances is generated and typically disposed of, posing environmental and economic concerns. Meanwhile, waxy substances, including components similar to stearic acid, possess desirable physicochemical properties such as hardness, partial elasticity, and the ability to act as structure-forming and stabilizing agents in cosmetic formulations. Therefore, the recovery and utilization of these waxy materials represent a promising approach for sustainable resource management and value addition.

Waxes are lipophilic organic substances that are solid yet pliable at room temperature. They typically consist of high-molecular-weight alkanes and lipid-like compounds that melt at temperatures above approximately 40 °C, forming low-viscosity liquids upon melting. Waxes are insoluble in water but readily dissolve in nonpolar organic solvents such as hexane, benzene, and chloroform. Various natural waxes are found in plants, animals, and petroleum sources [Krendlinger et al., 2023, Morrison et al., 1983].

In the food industry, sunflower wax is widely used as a structuring agent in confectionery and fat-based products. It plays an important role in stabilizing crystal structure, reducing oil separation, and increasing melting point in products such as chocolate coatings, creams, spreads, and margarines. In particular, it is highly valuable in the

development of solid fat systems free of trans fatty acids. In this regard, sunflower wax is classified among plant-derived waxes recognized as safe by the European Food Safety Authority[Azmi et al., 2022, Ramos et al., 1985].
In cosmetics and pharmaceuticals, sunflower wax is used in formulations such as lipsticks, balms, creams, and emulsions as a consistency enhancer, emulsion stabilizer, and film-forming (occlusive) agent. It can serve as an alternative to carnauba wax in certain applications. According to Cosmetic Ingredient Review assessments, it is considered safe for cosmetic use. In pharmaceutical applications, it is employed in controlled-release matrices for tablets and capsules, as a coating material, and as a hydrophobic barrier. Sunflower wax is biologically inert and exhibits high oxidative stability[Umirova et al., 2025, Mariani et al., 1989].
In coatings and polishing applications, sunflower wax is utilized to impart gloss and water resistance to furniture, flooring, paper, and food packaging materials. Due to its natural origin and biodegradability, it offers advantages over synthetic paraffins. Furthermore, in the development of fat-based emulsions and oleogels, sunflower wax functions as a structuring agent, gelator, and rheological modifier, enabling the conversion of liquid vegetable oils into solid, trans-fat-free systems [Akhmedova et al., 2024, Brevedan et al., 2000].
Sunflower wax consists of plant-derived wax-like compounds naturally present in seeds and oils as complex mixtures of low-volatility molecules. Chemically, these substances are primarily composed of esters formed from long-chain fatty acids and long-chain alcohols. Their general chemical structure can be expressed as R–COO–R′, where R represents the fatty acid chain and R′ denotes the alcohol moiety. Typically, esters containing carbon chains in the range of C40–C48 constitute the major fraction of sunflower wax, contributing to its high molecular weight and elevated melting point [Morrrison, 1982].
For example, when linoleic acid (C18:2) reacts with cetyl alcohol (C16H33OH) via esterification, cetyl linoleate (C18H31O2–O–C16H33) is formed, representing a typical wax ester. Due to their long hydrocarbon chains, these compounds exhibit relatively high melting temperatures (approximately 70–80 °C) and tend to crystallize upon cooling of sunflower oil, leading to turbidity. Gas chromatography (GC) analysis has confirmed that C40–C42 esters constitute a significant portion of sunflower wax [Rafael et al., 2023, Carelli et al., 2002].
Several methods are employed for the isolation of sunflower waxes. Extraction is commonly used for recovery from seeds, whereas both extraction and cooling (winterization) techniques are applied for separation from oil. Winterization is one of the most widely used methods and is based on cooling the oil to induce crystallization of high-melting components, including waxes. Studies have shown that cooling sunflower oil to temperatures around 5–10 °C over an extended period increases viscosity and promotes the formation of wax crystals of various morphologies. These crystals can then be separated by filtration.
During filtration, filter aids such as perlite or adsorbents like bentonite are often used to enhance separation efficiency. The isolated wax fraction typically consists predominantly of C36–C48 wax esters, accounting for approximately 60–80% of the total wax content. This combined cooling and filtration approach is a scientifically established and industrially viable method for efficient wax removal from sunflower oil [Sytnik et al., 2020].
In addition to oil, wax-like substances are also present in sunflower seed hulls and other structural components. In such cases, cooling methods are not effective, and extraction techniques are required. Organic solvents such as hexane or supercritical CO_2 may be used. Scientific studies indicate that supercritical CO_2 extraction provides superior efficiency. In this process, CO_2 in its supercritical state penetrates the matrix and selectively extracts wax components at the molecular level.
Due to its high diffusivity and tunable solvating power, supercritical CO_2 effectively separates waxes from oil fractions. Upon pressure reduction, the dissolved compounds precipitate, yielding a purified wax product. Waxes obtained via this method are enriched in C42–C48 esters and exhibit a more uniform molecular composition. An additional advantage of this technique is the absence of organic solvent residues, making the product safer for food and cosmetic applications [Carelli et al., 2002, . Demirci & Pehlivanoğlu, 2016].
Both winterization and extraction methods are effective; however, each has distinct advantages and limitations. Winterization is a relatively simple and cost-effective physical process suitable for large-scale industrial applications. However, co-crystallization of minor components such as sterol esters and ketones may reduce wax purity. In contrast, supercritical CO_2 extraction yields higher purity and more uniform products but requires higher investment costs, more complex process control, and generally lower throughput.
Therefore, despite its limitations, winterization remains the most widely used method for industrial-scale wax removal from sunflower oil [Chalapud et al., 2016,].
Based on the above analysis, the present study employed the winterization method for the separation of wax-like substances from sunflower oil.

2. Methods

2.1 Materials and sample preparation

2.1.1. Separation of Waxes from Sunflower Oil

Refined and bleached sunflower oil was used for the isolation of wax-like substances. Initially, the oil was heated to 60 °C and then cooled to 25 °C under continuous stirring using a magnetic stirrer. Subsequently, the cooling process was continued using a cooling system. The temperature was maintained at 5–10 °C for 24 hours.

After the formation of wax crystals, the wax-like substances were separated by filtration using filter paper. Perlite was used as a filtration aid. Two different methods were applied to separate perlite from the wax fraction.

In the first method, the wax–perlite mixture was subjected to extraction with hexane at 60 °C for 1 hour. The extraction process was carried out using a Soxhlet apparatus. The obtained solution was allowed to settle for 10–12 hours, after which the precipitated perlite was separated from the solvent by filtration.

In the second method, an aqueous sodium chloride solution was used. For this purpose, the sample was placed in a flask, and an aqueous solution of sodium chloride of a specified concentration was added. The flask was heated on an electric heater and boiled for 1 hour. After completion of the process, the mixture was gradually cooled and then separated by sedimentation at controlled temperature and time conditions. The settling temperature was regulated and maintained using a heating device.

As a result, the system separated into four distinct phases: precipitated perlite, an aqueous layer, a wax layer, and an upper oil layer. After settling, the wax layer was separated, washed with hot water, and dried under vacuum. In this study, the effects of sodium chloride concentration, process temperature, and settling time on the yield and melting point of wax-like substances were investigated.

2.1.2. Cream preparation

A conventionally used **moisturizing and nourishing cream formulation** was chosen as the base formulation for the study. The formulation composition (per 100 g, %) was as follows: oil phase consisting of stearic acid (14.0%), liquid paraffin (6.0%), glyceryl monostearate (3.0%), and oil (10.0%); and aqueous phase consisting of glycerin (4.0%), propylene glycol (4.0%), isopropyl myristate (2.0%), triethanolamine (0.2%), propyl paraben (0.1%), fragrance (0.03%), and water (56.67%).

The cosmetic cream formulation was developed based on a prototype triethanolamine-based cream, using only stearic acid and sunflower wax as structuring agents. The oil phase of the emulsion was initially prepared by melting sunflower wax at 70–80 °C, followed by the addition of stearic acid, liquid paraffin, lanolin, and glyceryl monostearate into the molten mass. The aqueous phase was prepared separately by mixing propylene glycol, isopropyl myristate, triethanolamine, glycerin, and water, which was heated to the same temperature as the oil phase. Both phases were then gradually combined under continuous stirring to obtain a homogeneous dispersion. When the temperature decreased to 35 °C, a fragrance was added. Preservatives were incorporated into the aqueous phase prior to mixing [15].

2.2 Physicochemical analyses of waxes

The acid value of the waxes was determined according to standard methods ISO 660 or GOST 5476. The saponification value was determined in accordance with ISO 3657 or GOST 5478. The mass fraction of moisture and volatile substances was determined according to GOST 11812. The melting point of the wax was determined using the open capillary method in accordance with GOST ISO 6321.

2.3. Evaluation of cream

The prepared cream formulations were evaluated in terms of pH, spreadability, viscosity, and particle size, as reported in previous studies [18–28].

2.3.1. pH measurement

The pH of the cream was determined using a pH meter. A 10% (w/v) cream suspension was prepared and measured at 25 °C. Prior to analysis, the instrument was calibrated using standard buffer solutions at pH 4.0 and 7.0. Measurements were performed in triplicate, and the average value was reported.

2.3.2. Spreadability

The spreadability of the formulations was evaluated using a standard method. Approximately 0.5 g of the sample was placed within a marked circle on a glass slide, and a second glass plate was placed on top. A weight of 500 g was applied and maintained for 5 minutes.

Spreadability was defined as the area covered by the sample after uniform spreading between the glass plates. The increase in diameter due to spreading was recorded, and the average of three measurements was reported.

2.3.3. Viscosity

Rheological properties were determined using a viscometer. Approximately 50 g of the sample was placed in a beaker and allowed to equilibrate for 5 minutes. Measurements were performed using a T-D spindle at rotational speeds of 10, 20, 30, 50, 60, and 100 rpm.

The dial readings corresponding to each speed were recorded. Subsequently, the spindle speed was gradually decreased, and the corresponding readings were again noted. All measurements were conducted at room temperature and repeated three times.

2.3.4. Thermal stability

The thermal stability of the prepared creams was evaluated at 20 °C, 30 °C, and 40 °C in accordance with standard testing procedures.

3. Results and discussion

3.1 Separation of Perlite from Wax Fractions

To separate perlite from wax-like substances obtained by filtration of sunflower oil, experiments were carried out using aqueous sodium chloride solutions with concentrations ranging from 1% to 10%. The optimal concentration of the salt solution was determined based on the yield of the recovered wax fraction. The experimental results are presented in Figure 1.

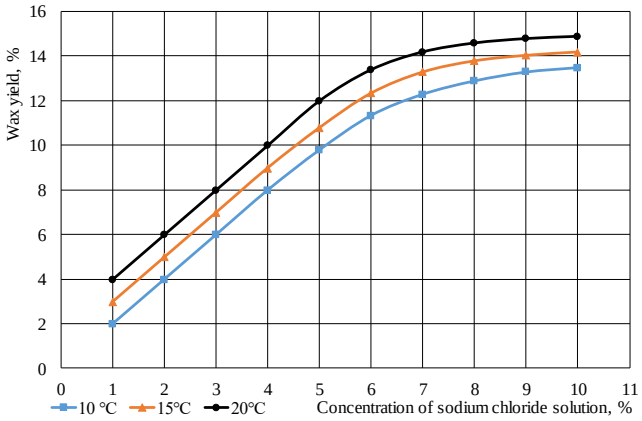


Figure 1: Effect of salt solution concentration and deposition temperature on separation of waxy substance from a mixture of waxes and perlite

As shown in Figure 1, when the concentration of the sodium chloride solution increased from 1% to 10%, the yield of wax recovered from the wax–perlite mixture increased from 2% to 15%. Analysis of the curve indicates that the yield increased rapidly up to a salt concentration of 8%, after which the growth rate slowed down and approached a plateau.

In addition to concentration, the temperature during the sedimentation process after treatment with the salt solution also influenced the wax yield. Increasing the temperature from 10 °C to 20 °C resulted in an approximately 2% increase in wax recovery.

The duration of the sedimentation process is another important factor affecting the separation efficiency. Therefore, in subsequent experiments, samples treated under optimal concentration conditions were subjected to sedimentation at 20 °C for 5–20 hours in order to determine the optimal settling time. The results are presented in Figure 2.

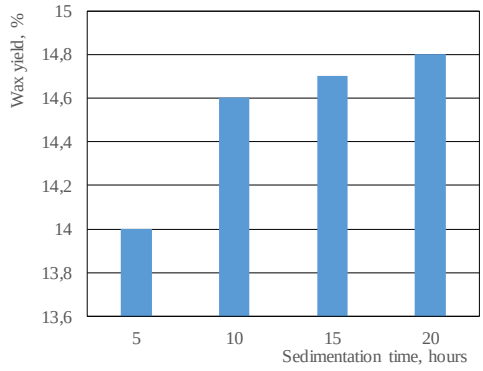


Figure 2: Effect of perlite deposition duration on wax yield

According to the data shown in Figure 2, the yield of wax increased from 14% to 14.6% as the sedimentation time increased up to 10 hours. Beyond 10 hours, no significant increase in yield was observed.
Thus, based on the obtained experimental results, it can be concluded that the optimal conditions for separating perlite from wax-like substances derived from sunflower oil using an aqueous sodium chloride solution are: salt concentration of 8%, sedimentation temperature of 20 °C, and sedimentation time of 10 hours.
The physicochemical properties of the wax obtained under these optimal conditions were compared with those of wax extracted using hexane. The results are presented in Table 1.

Table 1: Physico-chemical characteristics of waxes extracted from used perlite

№Index name	Separated with saline solution	Separated with hexane
1 Melting temperature, ℃	69	70
2 Acid value, mg KOH/g	2,8	2,6
3 Saponification value, mg KOH/g	110	114
4 Mass fraction of moisture and volatile substances, %	0,84	0,80

As shown in Table 1, the physicochemical characteristics of waxes obtained by both methods were found to be comparable.
The physicochemical and structural characterization of the isolated waxy substances demonstrated that they predominantly consist of long-chain saturated compounds and wax esters. Chromatographic analysis confirmed that the main components include saturated fatty acids (52–58%), fatty alcohols (18–24%), and wax esters (15–20%), with minor amounts of hydrocarbons and sterols (3–5%).
The relatively low acid value (2.8 mg KOH/g) indicates a minimal content of free fatty acids, while the saponification value (110 mg KOH/g) confirms the presence of esterified structures typical for natural waxes. The refractive index (n = 1.442–1.448 at 40 °C) further supports the high degree of saturation and the predominance of long-chain aliphatic compounds.
Infrared (IR) spectroscopy revealed characteristic absorption bands at 2915–2848 cm^{-1} (C–H stretching), 1735 cm^{-1} (C=O stretching of esters), and 1170–1240 cm^{-1} (C–O stretching), confirming the presence of ester functional groups and long hydrocarbon chains.
Differential scanning calorimetry (DSC) analysis showed a sharp melting peak at approximately 69 °C, with a melting range of 66–72 °C and an enthalpy change of 120–150 J/g, indicating a well-defined crystalline structure with a high degree of order. The crystallization peak observed at 60–64 °C further confirms the uniformity and stability of the wax structure.
Overall, the obtained results demonstrate that the isolated wax possesses high thermal stability, a low level of impurities (moisture content 0.84%), and a structurally ordered composition, making it suitable for application as a structuring agent in cosmetic and food formulations.
The melting point of wax is relatively high, similar to that of stearic acid. Therefore, it can be used as a substitute for stearic acid in various applications. In this study, sunflower wax was investigated as a partial or complete replacement for stearic acid in a cosmetic cream formulation. For this purpose, stearic acid in the cream recipe was partially or fully replaced with sunflower wax, and the physicochemical properties of the resulting creams were evaluated.
As shown in the selected cosmetic cream formulation, the oil phase accounts for more than 33% of the total composition, of which 14% is stearic acid. Stearic acid represents more than 40% of the oil phase and plays a key role in determining the structural properties of the cream. The effect of varying concentrations of stearic acid and sunflower wax on the viscosity and spreadability of the cream was investigated. The obtained results are presented in Figure 1.

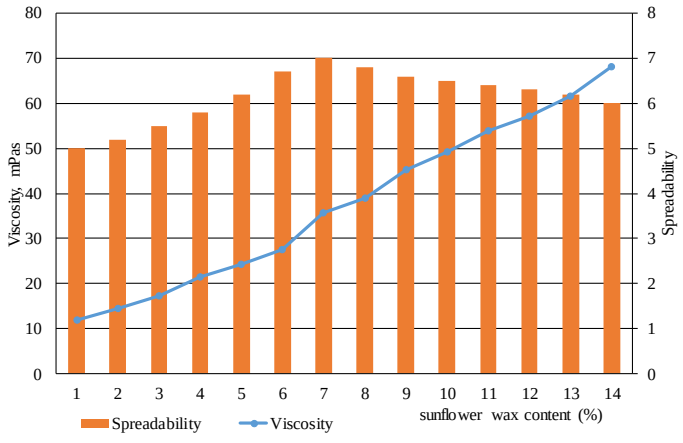


Figure 2: The effect of sunflower wax on the viscosity and spreadability of cosmetic cream

As can be seen from Figure 1, replacing stearic acid with sunflower wax leads to an increase in viscosity as the mass fraction of wax increases. However, the spreadability increases with wax content up to 7%, and then gradually decreases when the wax content rises from 7% to 17%. This behavior can be explained by the significant reduction in the mass fraction of stearic acid in the formulation.
The pH values of the obtained creams were analyzed and found to be in the range of 7.2–7.4.
Thermal stability, viscosity, and spreadability are key parameters affecting the suitability of cosmetic creams during storage and processing. All creams containing sunflower wax exhibited improved thermal stability at 20 °C, 30 °C, and 40 °C. However, formulations containing more than 7% sunflower wax showed reduced thermal stability and lower spreadability, which led to structural instability and phase separation.

4. Conclusions

In conclusion, the proposed method enables effective purification and valorization of waxy waste generated during sunflower oil winterization. The partial replacement of stearic acid with recovered waxy substances not only reduces raw material costs but also contributes to environmental protection by minimizing waste and eliminating the use of hazardous solvents. This approach offers a sustainable and economically viable solution for the cosmetic industry.
Wax-like substances were successfully recovered from sunflower oil processing waste using an optimized NaCl-assisted separation method (8%, 20 °C, 10 h), achieving a yield of up to 14.6%. The obtained wax showed physicochemical properties comparable to hexane-extracted samples. Analytical results (GC, IR, DSC, refractometry) confirmed that the wax consists mainly of long-chain saturated compounds and wax esters, with a melting point of 69 °C, low acid value (2.8 mg KOH/g), and refractive index of 1.442–1.448.
In cosmetic applications, sunflower wax effectively replaced stearic acid, increasing viscosity and improving spreadability up to 7% concentration, while higher contents reduced stability. The results demonstrate the potential of recovered wax as a sustainable structuring agent in cosmetic formulations.

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