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INFRARED SPECTROSCOPIC ANALYSIS OF THE PURIFICATION OF SUNFLOWER OIL FROM WAXY SUBSTANCES USING PERLITE AND VERMICULITE

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Abstract: This article investigates the effectiveness of perlite as a filtering agent for the removal of waxy substances from sunflower oil during the winterization process. Sunflower oil tends to become cloudy at low temperatures due to the presence of wax esters, phosphatides, and other wax-like compounds, which negatively affect its appearance and commercial value. In the experimental procedure, the oil was cooled to 7–8 °C to initiate wax crystallization and then filtered through perlite. The filtration efficiency was evaluated using infrared (IR) spectroscopy, which allowed for the identification of functional groups associated with waxy substances. The study also examined the regeneration potential of perlite by subjecting the used filter material to thermal treatment and reusing it in repeated cycles. Physicochemical properties such as porosity and structural stability were also assessed. The IR spectra confirmed that regenerated perlite retained its filtering capabilities. Overall, the results show that perlite is an effective and reusable natural adsorbent for improving the clarity and quality of sunflower oil.

Keywords: sunflower oil, waxy substances, perlite, vermiculite, regeneration, infrared spectroscopy, filtration, winterization, C=O group, C-H stretching vibration, thermal treatment, crystallization, surface activation.

Introduction. sunflower oil, waxy substances, perlite, vermiculite, regeneration, infrared spectroscopy, filtration, winterization, C=O group, C-H stretching vibration, thermal treatment, crystallization, surface activation.

Sunflower oil contains waxy phosphatides and other wax-like substances that cause turbidity at low temperatures, thereby reducing its commercial appeal. The removal of these substances is a critical step in the clarification process to ensure the oil is transparent and suitable for consumption. Typically, such waxy compounds are removed through cooling and crystallization (winterization), followed by filtration using adsorbent filter materials. Natural porous adsorbents such as perlite and vermiculite are widely utilized for this purpose due to their high surface area and adsorption capacity.

Sunflower oil is widely consumed due to its nutritional value and favorable fatty acid composition. However, it contains waxy substances such as wax esters, phosphatides, and sterols, which tend to crystallize at low temperatures, causing turbidity and reducing the oil's commercial quality. Therefore, the removal of these

substances is a critical step in refining processes, particularly for achieving clarity, stability, and extended shelf life.

Winterization is the conventional method used to eliminate these waxy impurities. It involves controlled cooling to induce crystallization, followed by filtration to separate the solidified wax particles. The efficiency of this process largely depends on the type and performance of the filter media used.

In this context, natural mineral-based adsorbents such as perlite and vermiculite have gained attention due to their high surface area, porosity, thermal stability, and low cost. Perlite is an amorphous volcanic glass that expands when heated and acts as a highly porous filtration aid. Vermiculite, on the other hand, is a hydrated layered silicate with excellent adsorption properties, which can be enhanced through thermal activation.

This study focuses on evaluating the effectiveness of perlite and thermally treated vermiculite in removing waxy substances from sunflower oil. The purification process was monitored using infrared (IR) spectroscopy, which allows the identification and quantification of functional groups associated with waxy compounds. By comparing IR spectra before and after filtration, the study aims to assess the adsorption efficiency of each material and determine their potential for industrial application in edible oil processing.

In recent years, growing consumer demand for high-quality, visually appealing edible oils has intensified the focus on effective dewaxing methods. Waxy substances in sunflower oil, such as long-chain esters and phosphatides, contribute to turbidity when the oil is stored or cooled, negatively affecting its marketability. Traditional winterization techniques involving cooling and filtration remain the most commonly employed industrial approach for wax removal.

However, the choice and reusability of filtering materials play a crucial role in the cost-efficiency and environmental sustainability of this process. Natural adsorbents like perlite and vermiculite have attracted attention due to their low cost, chemical inertness, high porosity, and potential for regeneration after thermal treatment. Moreover, advances in spectroscopic analysis, particularly Fourier-transform infrared (FTIR) spectroscopy, allow researchers to assess the molecular-level effectiveness of these materials in wax removal.

This study aims to evaluate the filtration performance of perlite and thermally treated vermiculite at different temperatures by comparing their wax adsorption efficiency through IR spectral analysis. Understanding the interaction mechanisms between these materials and waxy substances can inform more sustainable and effective industrial practices.

Methods. In the conducted experiment, sunflower oil was subjected to a controlled cooling process, gradually brought down to a temperature range of approximately 7–8 °C. This step was implemented in order to initiate the crystallization of waxy substances, which are naturally present in the oil and tend to solidify at low temperatures. Once the crystallization was achieved, the samples were subjected to a filtration process using three different types of adsorbents: perlite and two variants of vermiculite. The

vermiculite samples were pre-treated through thermal activation at two distinct temperatures—400 °C and 600 °C—aimed at modifying their surface and structural properties to enhance adsorption performance.

For analytical comparison, oil samples were taken both before and after the filtration process. These samples were then analyzed using Fourier-transform infrared (FTIR) spectroscopy across the spectral range of 4000 to 450 cm^{-1} . The IR spectra allowed for the identification and monitoring of characteristic functional groups associated with waxy compounds. In the control (unfiltered) sample, the IR spectrum (line 1) exhibited distinct absorption bands within the 2800–3015 cm^{-1} frequency range, which correspond to the stretching vibrations of C–H bonds. Additional absorption peaks were observed at 1485 and 1390 cm^{-1} , indicating C–H bending (deformation) vibrations. A prominent absorption band near 1760 cm^{-1} was attributed to the stretching of carbonyl (C=O) functional groups, while the spectral region between 1086 and 1165 cm^{-1} corresponded to C–O–C asymmetric and symmetric stretching vibrations typically associated with ester and ether functionalities. The obtained IR spectra and detailed analysis outcomes are illustrated and discussed in Figures 1 and 2.

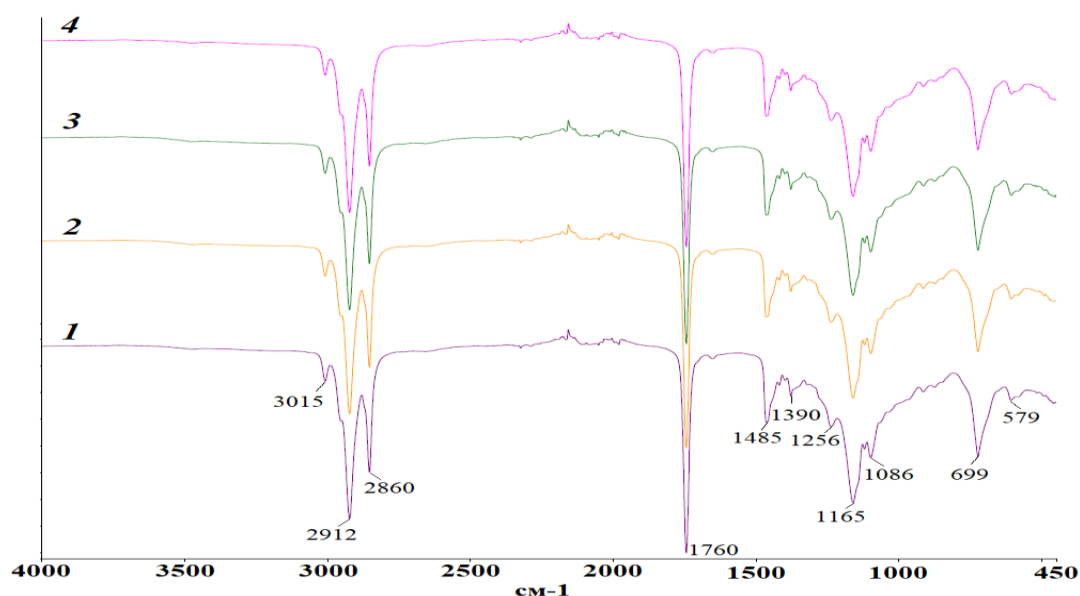


Figure 1. IR analysis of sunflower oil after wax removal using perlite and vermiculite (treated at 400 °C and 600 °C)

1) Control sample, 2) Sample treated with perlite, 3) Sample treated with vermiculite (400 °C), 4) Sample treated with vermiculite (600 °C)

The IR spectral results of the samples treated with perlite (line 2), vermiculite at 400 °C (line 3), and vermiculite at 600 °C (line 4) indicate that they are nearly identical. Based on these results, it can be concluded that the oil does not undergo any chemical reaction with the treatment materials under the experimental conditions.

The spectra also exhibit characteristic absorption bands corresponding to $[\text{SiO}_4]$ Si–O groups with tetrahedral structures: 1005 cm^{-1} (perlite, line 1) and 994 cm^{-1} (vermiculite

treated at 400 °C and 600 °C, lines 2 and 3). The bands around 759 cm⁻¹ correspond to the vibrations of the Si–O–Al group, while the bands at 673 cm⁻¹ are attributed to the vibrations of the structural fragment Si–O–Mg.

In the IR spectra, characteristic stretching vibrations of waxy substances were prominently observed, particularly around ~2915 cm⁻¹ and ~2849 cm⁻¹ (C–H stretching vibrations), as well as ~1735 cm⁻¹ (associated with the C=O group), showing high intensity. In the post-filtration samples, these regions exhibited noticeably diminished peaks, indicating a significant reduction in waxy components.

During filtration with perlite, the intensity of these peaks decreased by approximately 30–33%, while vermiculite treated at 600 °C resulted in a 35–40% reduction. The most significant effect was observed with vermiculite treated at 400 °C, where peak intensity dropped by up to 45%. This clearly indicates a substantial reduction in the concentration of waxy substances.

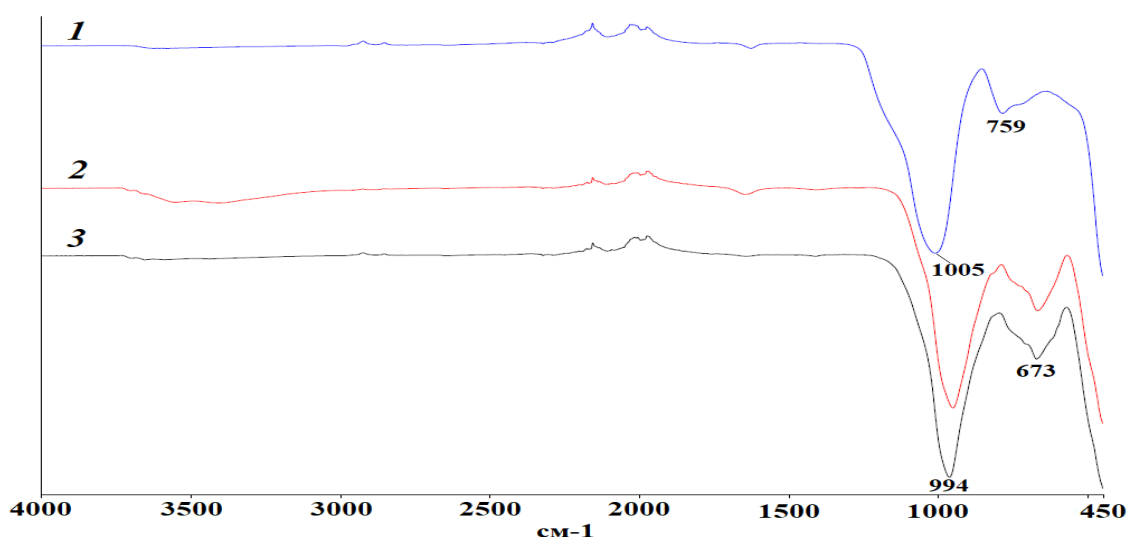


Figure 1. IR analysis of sunflower oil treated with perlite and vermiculite (400 °C and 600 °C)

1) Sample treated with perlite, 2) Sample treated with vermiculite (400 °C), 3) Sample treated with vermiculite (600 °C)

Spectral analysis confirmed the effective removal of waxy substances from sunflower oil. The high porosity and surface activity of perlite make it an efficient adsorbent in this process. Vermiculite, due to its layered structure and surface reactivity, provides additional adsorption capacity. The combination of these materials demonstrated a synergistic effect, resulting in enhanced purification efficiency.

Infrared spectroscopy proved to be a reliable method for determining the amount of waxy substances in sunflower oil purified using perlite and vermiculite. According to the results, vermiculite showed the highest efficiency in removing waxy impurities from sunflower oil. This approach may be beneficial for improving purification efficiency at an industrial scale.

These findings validate the effectiveness of IR spectroscopy in assessing purification quality and demonstrate that thermally treated vermiculite, particularly at 400 °C, offers superior performance in removing waxy substances from sunflower oil.

Results. Infrared spectroscopic analysis provided compelling evidence of the effectiveness of perlite and thermally treated vermiculite in the removal of waxy substances from sunflower oil. In the control (unfiltered) sample, distinct absorption bands were observed at approximately 2915 cm^{-1} and 2849 cm^{-1} , corresponding to the C–H stretching vibrations of methylene and methyl groups. A strong absorption band at $\sim 1735 \text{ cm}^{-1}$ was attributed to the C=O stretching vibration, typically associated with esters and phosphatides – the primary components of waxy impurities in edible oils.

Following filtration, a marked reduction in the intensity of these characteristic bands was recorded for all treatment groups:

Perlite-treated samples showed a decrease in intensity by approximately 30–33%, indicating moderate wax removal.

Vermiculite treated at 600 °C exhibited a slightly higher reduction of 35–40%, suggesting improved adsorption capacity with increased thermal activation.

Vermiculite treated at 400 °C demonstrated the highest efficiency, with up to 45% reduction, indicating enhanced interaction between the waxy components and the adsorbent surface.

This trend suggests that all adsorbents contributed to effective dewaxing, with 400 °C-treated vermiculite showing the most pronounced performance, likely due to optimal surface activation and porosity.

In addition to wax-related peaks, the IR spectra exhibited consistent structural bands associated with the filter materials themselves:

A Si–O stretching band appeared at 1005 cm^{-1} for perlite and at 994 cm^{-1} for both vermiculite samples.

Si–O–Al bond vibrations were observed near 759 cm^{-1} , while Si–O–Mg vibrations appeared at 673 cm^{-1} .

These structural peaks remained unchanged before and after filtration, suggesting that the filtration process did not alter the chemical structure of the adsorbents or induce reactions with the oil.

Discussion. The spectral analysis revealed a clear correlation between thermal treatment of adsorbents and their wax-removal efficiency. Vermiculite activated at 400 °C showed the highest level of wax adsorption, likely due to the optimal opening of its layered structure and increased surface area. Interestingly, further thermal treatment at 600 °C slightly reduced this efficiency, possibly due to partial structural collapse or sintering effects that limit access to adsorption sites.

Perlite, despite its comparatively lower adsorption rate, proved to be chemically inert and structurally stable, which are desirable traits for multi-cycle filtration. The 30–33% reduction in wax-specific IR bands confirms its suitability for industrial applications where cost-effectiveness and reusability are important.

Moreover, the absence of new functional groups or spectral shifts post-filtration indicates that the interaction between the oil and adsorbents is purely physical—likely van der Waals forces and surface entrapment—rather than chemical bonding. This is advantageous as it helps preserve the chemical composition and quality of the oil.

These findings align with previous studies that emphasize the role of adsorbent microstructure, porosity, and thermal treatment in determining filtration performance. However, this study highlights the potential of using thermally optimized vermiculite as a more efficient alternative to conventional materials. Further research could explore regeneration limits and scalability for continuous industrial use.

Conclusion. The study demonstrated that infrared (IR) spectroscopy is a reliable analytical tool for monitoring the removal of waxy substances from sunflower oil using natural adsorbents such as perlite and vermiculite. Spectral analysis revealed characteristic absorption bands corresponding to waxy components, particularly in the regions around 2915 cm^{-1} , 2849 cm^{-1} (C–H stretching), and 1735 cm^{-1} (C=O stretching). These bands showed significant intensity reductions after filtration, indicating effective purification.

Perlite exhibited a 30–33% reduction in peak intensity due to its high porosity and surface activity, while vermiculite treated at $600\text{ }^{\circ}\text{C}$ achieved a 35–40% reduction. Vermiculite treated at $400\text{ }^{\circ}\text{C}$ demonstrated the highest efficiency, reducing the peak intensity by up to 45%. Additionally, spectral features associated with structural groups such as Si–O, Si–O–Al, and Si–O–Mg confirmed the chemical stability of the adsorbents and their non-reactivity with the oil.

The synergistic use of perlite and vermiculite, leveraging their complementary structural and surface properties, led to enhanced purification performance. Overall, vermiculite—particularly thermally treated at $400\text{ }^{\circ}\text{C}$ —proved to be the most effective in removing waxy impurities from sunflower oil. These findings suggest that such an approach can be beneficial for improving filtration efficiency in industrial-scale oil processing applications.

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