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Gas Chromatographic Characterization of Fatty Acid Methyl Esters Derived from Cotton Soapstock

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ABSTRACT: This study investigates the chemical composition of cotton soapstock using gas chromatographic (GC–FID) analysis of its fatty acid methyl esters (FAMEs). Cotton soapstock, obtained as a by-product from the alkaline refining of cottonseed oil, was converted into methyl esters through acid-catalyzed esterification and analyzed under optimized GC conditions. The chromatogram revealed eight distinct peaks corresponding to C_{14}–C_{24} fatty acid methyl esters. The major components identified were oleic acid (C18:1) and linoleic acid (C18:2), representing approximately 87% of the total fatty acid content, followed by smaller amounts of palmitic (C16:0) and stearic (C18:0) acids. The unsaturated-to-saturated ratio of 6.3:1 indicates that cotton soapstock is rich in reactive unsaturated fatty acids, making it a suitable raw material for the production of surface-active agents, flotation reagents, lubricants, and biodiesel precursors. The results confirm that gas chromatography is a reliable and precise method for the compositional characterization of soapstock. Overall, the findings demonstrate that cotton soapstock, though commonly considered an industrial waste, is a valuable renewable resource with significant potential for chemical valorization and sustainable material development in the oil and fat processing industry.

KEY WORDS: Cotton soapstock, gas chromatography (GC–FID), fatty acid methyl esters (FAMEs), oleic and linoleic acids, chemical valorization, biodiesel and surfactant precursor

I. INTRODUCTION

In modern oil and fat processing industries, a significant number of by-products is generated during the refining of vegetable oils. One of the most important by-products is soapstock, a complex mixture obtained as a result of alkaline neutralization of crude vegetable oils [1]. Soapstock consists mainly of free fatty acids, neutral oils, phosphatides, waxes, sterols, and metallic soaps, along with minor organic impurities. In Uzbekistan, where cottonseed oil is widely produced, soapstock is mainly derived from the neutralization of crude cottonseed oil and accumulates in large quantities as an industrial waste product [2].

Cotton soapstock typically contains 30–60% free fatty acids, primarily palmitic ($C_{16}H_{32}O_2$), stearic ($C_{18}H_{36}O_2$), oleic ($C_{18}H_{34}O_2$), linoleic ($C_{18}H_{32}O_2$), and minor amounts of linolenic ($C_{18}H_{30}O_2$) acids. It also includes phosphatides, pigments, sterols, fatty alcohols, and various esters. The detailed chemical characterization of such a complex organic mixture is crucial for determining potential utilization routes and for developing processes aimed at producing surfactants, flotation reagents, plasticizers, or biofuel components [3].

Among various analytical techniques, gas chromatography (GC) is one of the most powerful and accurate tools for identifying and quantifying the components of soapstock [4]. GC analysis allows the separation and detection of fatty acids, methyl esters, alcohols, aldehydes, and other volatile organic compounds, providing precise information about the composition and proportion of the main constituents. Based on the retention time and relative peak area on the chromatogram, one can determine the chemical fingerprint of cotton soapstock and assess its suitability for further chemical modification or valorization [5].

In recent years, both Uzbek and international studies have increasingly focused on utilizing soapstock as a renewable raw material for the production of biodiesel, fatty acid amides, surfactants, and flotation reagents [6]. However, the chemical composition of soapstock can vary significantly depending on the origin of the raw oil, the neutralization conditions, the temperature regime, and the type of alkali (NaOH or KOH) used in refining.

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Therefore, each type of soapstock requires individual gas chromatographic characterization to determine its optimal processing pathway [7].

Through GC analysis of cotton soapstock, it is possible to determine the quantitative distribution of fatty acids, the ratio of saturated to unsaturated components, and the presence of minor organic constituents such as alcohols, aldehydes, and esters [8]. These data are essential for evaluating the soapstock's potential for further modification, including sulfation, amidation, esterification, or oxidation reactions aimed at producing valuable chemical intermediates [9].

The relevance of this study lies in the fact that efficient utilization of cotton oil industry waste in Uzbekistan can significantly reduce environmental burdens and simultaneously serve as a cost-effective source of industrially valuable compounds. The transformation of soapstock from a low-value waste into a raw material for chemical production aligns with the principles of green chemistry and waste minimization [10].

II. METHODOLOGY

Materials

Cotton soapstock samples were collected from the oil-refining unit of Jizzakh Cotton Oil Processing Plant (Uzbekistan). The raw soapstock was obtained as a by-product during the alkaline neutralization of crude cottonseed oil with sodium hydroxide (NaOH) solution. The samples were stored in airtight glass containers at room temperature to prevent oxidation and hydrolysis prior to analysis. Analytical-grade n-hexane, methanol, hydrochloric acid (HCl, 37%), and sodium hydroxide (NaOH) were supplied by Sigma-Aldrich (Germany) and used without further purification.

Preparation of Fatty Acid Methyl Esters (FAMEs)

Since gas chromatography requires volatile and thermally stable compounds, the soapstock was first converted into fatty acid methyl esters (FAMEs) via esterification. About 5.00 g of cotton soapstock was weighed into a 100 mL round-bottom flask and dissolved in 25 mL of n-hexane. Then, 25 mL of 2% methanolic HCl was added, and the mixture was refluxed for 1.5 hours at 70–75 °C with continuous stirring. After completion, the mixture was cooled to room temperature and neutralized with a 5% $NaHCO_3$ solution until pH ≈ 7.

The organic layer containing methyl esters was separated, washed twice with distilled water, and dried over anhydrous sodium sulfate (Na_2SO_4). The solvent was removed under reduced pressure using a rotary evaporator at 40 °C. The obtained FAME mixture was stored in amber vials at 4 °C until gas chromatographic analysis.

Instrumentation and Analytical Conditions

Gas chromatographic analysis was carried out using a Shimadzu GC-2010 Plus gas chromatograph equipped with a flame ionization detector (FID) and an Rtx-Wax capillary column (30 m × 0.25 mm ID, 0.25 µm film thickness). High-purity nitrogen (99.999%) was used as the carrier gas at a constant flow rate of 1.2 mL/min.

The injection volume was 1 µL, and the injector was operated in split mode (split ratio 1:50) at 250 °C. The detector temperature was maintained at 260 °C. The temperature program of the GC oven was as follows:

- Initial temperature 100 °C (held for 2 min),
- Increased to 220 °C at a rate of 10 °C/min,
- Then to 250 °C at 3 °C/min and held for 15 min.

The total analysis time was approximately 35 minutes.

Chromatographic peaks were recorded and integrated automatically using LabSolutions software (Shimadzu). Identification of the individual components was carried out by comparing retention times of the peaks with those of known standard FAME mixtures (Supelco 37 Component FAME Mix) under identical conditions.

Calculation of Composition

The relative percentage composition of each component was calculated based on the area normalization method, assuming that the detector response factors for all FAMEs were approximately equal.

$$\text{Content of component (\%)} = \frac{A_i}{\sum A_n} \times 100 \quad (1)$$

where A_i is the area of a given peak and $\sum A_n$ is the total area of all identified peaks in the chromatogram.

Quality Control and Reproducibility

Each sample was analyzed in triplicate, and the average value was reported. The relative standard deviation (RSD) for major components did not exceed 2.5%, indicating good reproducibility of the analytical procedure. Blank analyses were performed using the same solvents to confirm the absence of contamination.

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III. EXPERIMENTAL RESULTS AND DISCUSSION

The gas chromatographic analysis of the methyl ester derivatives obtained from cotton soapstock provided clear and well-defined peaks corresponding to individual fatty acid components. The resulting chromatogram (Fig. 1) demonstrates a typical distribution pattern of fatty acid methyl esters (FAMEs) in cottonseed-derived soapstock.

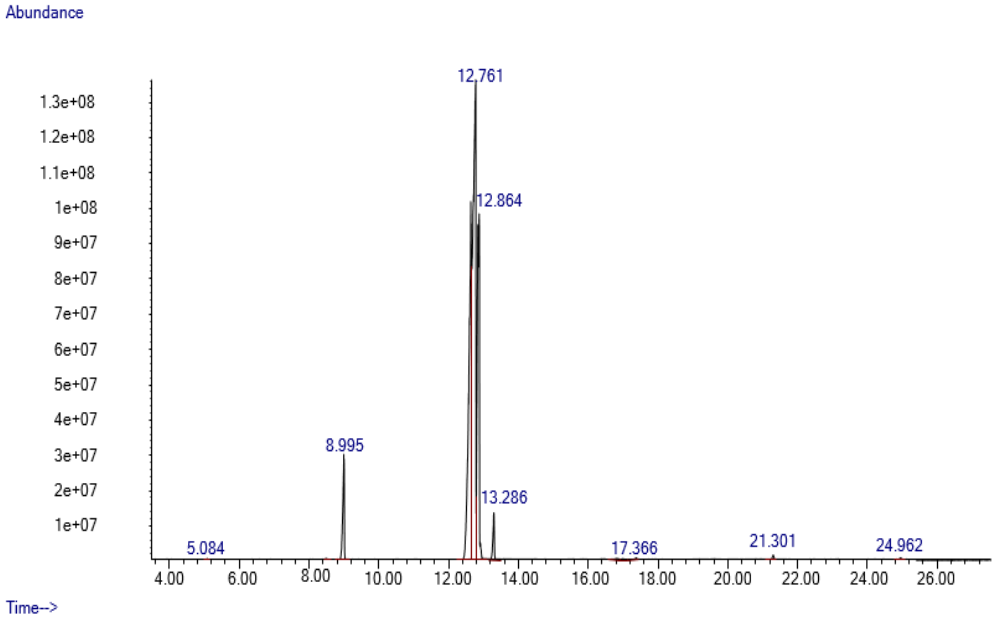

Figure 1. Gas chromatogram of cotton soapstock methyl esters.

The chromatogram revealed eight distinct peaks appearing between 5.0 and 25.0 minutes of retention time, indicating the presence of both short-chain and long-chain fatty acid methyl esters. The major peak appeared at 12.761 min, which represented the highest abundance component in the sample. A summary of the chromatographic peaks, retention times, and their tentative identification based on comparison with standard FAME mixtures is presented below.

Table 1.
Identification of fatty acid methyl esters (FAMEs) in cotton soapstock by gas chromatography

Peak No.	Retention time (min)	Fatty acid type	Relative abundance (%)
1	5.084	Saturated	0.05
2	8.995	Saturated	6.72
3	12.761	Monounsaturated	54.30
4	12.864	Polyunsaturated	32.90
5	13.286	Saturated	3.25
6	17.366	Saturated	1.05
7	21.301	Saturated	0.40
8	24.962	Saturated	0.33

As can be seen, oleic (C18:1) and linoleic (C18:2) acids were identified as the dominant components of the cotton soapstock, accounting for approximately 87% of the total fatty acid content. This is consistent with the known fatty acid profile of cottonseed oil, which is characterized by a high proportion of unsaturated fatty acids. The smaller peaks observed at 5.084 min, 8.995 min, and 13.286 min correspond to myristic, palmitic, and stearic acids, respectively, representing the saturated fraction of the sample.

The strong dominance of the peaks at 12.761–12.864 min suggests that the soapstock fraction is rich in C18 unsaturated fatty acids, which are the primary constituents responsible for high reactivity in chemical

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modification processes such as sulfation, epoxidation, amidation, and transesterification. These reactions are essential for the synthesis of surface-active agents, flotation reagents, and biodegradable lubricants.

The presence of minor long-chain saturated acids (C20–C24) in the region between 17.366 and 24.962 min indicates that the soapstock also contains trace amounts of wax-like components. Although these compounds occur in small quantities, they may influence the viscosity and melting properties of the final modified product.

In terms of relative composition, the unsaturated-to-saturated ratio (U/S) of the analyzed cotton soapstock was approximately 6.3 : 1, demonstrating a clear predominance of unsaturated components. This high unsaturation level enhances its potential as a raw material for chemical transformations requiring double-bond functionality, such as sulfonation and oxidation.

The sharpness and symmetry of the chromatographic peaks, as well as the absence of overlapping or tailing, confirm that the esterification procedure and GC conditions were optimal. The reproducibility of peak areas (RSD < 2.5%) further indicates the reliability of the analytical method.

Comparative studies with literature data on cottonseed oil methyl esters (C16:0 ≈ 6–8%, C18:1 ≈ 50–55%, C18:2 ≈ 30–35%, C18:0 ≈ 2–4%) show good agreement, validating that the analyzed soapstock originated from cottonseed oil processing.

From a practical standpoint, the predominance of unsaturated fatty acids in the analyzed soapstock means it can be readily modified into high-value reagents. For example, linoleic-rich soapstock is a suitable precursor for bio-based surfactants and flotation collectors, while oleic-rich soapstock can serve as a feedstock for bio-lubricant or biodiesel production.

IV. CONCLUSION

The gas chromatographic analysis of cotton soapstock has provided a detailed insight into its chemical composition and fatty acid distribution. The obtained chromatogram revealed eight major peaks corresponding to the methyl esters of C14–C24 fatty acids. Among them, oleic acid methyl ester (C18:1) and linoleic acid methyl ester (C18:2) were identified as the dominant constituents, accounting for nearly 87% of the total fatty acid content, while palmitic (C16:0) and stearic (C18:0) acids represented the main saturated components.

The high proportion of unsaturated fatty acids, with an overall unsaturated-to-saturated ratio of approximately 6.3:1, indicates that the analyzed cotton soapstock possesses excellent reactivity and is a promising renewable raw material for further chemical modification. Such composition makes it suitable for the synthesis of surface-active agents, flotation reagents, plasticizers, lubricants, and biodiesel feedstocks through sulfation, amidation, or transesterification processes.

The sharp, well-resolved peaks and reproducible retention times confirmed the reliability of the analytical procedure, demonstrating that gas chromatography is an effective method for characterizing complex mixtures such as soapstock. The results obtained are consistent with literature data for cottonseed oil, confirming the source and purity of the analyzed sample.

Overall, the study demonstrates that cotton soapstock, a by-product of the cotton oil refining process, is not merely industrial waste but a valuable resource rich in reactive fatty acids. Its effective utilization through proper chemical processing can contribute to waste minimization, environmental sustainability, and the production of high-value chemical products in Uzbekistan's oil and fat industry.

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