

Advanced characterization of biodiesel produced from spent Greek coffee grounds using gas chromatography-tandem mass spectrometry analysis

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Introduction

Waste biomass valorization involves converting organic waste materials into valuable energy products, contributing to sustainable energy systems and effective waste management. Biomass sources, such as agricultural residues, forestry by-products, industrial organic waste, and municipal solid waste, represent significant renewable resources (Emmanouilidou et al., 2023; Okolie et al., 2022). Coffee is among the most traded commodities globally and ranks as one of the most widely consumed beverages worldwide. Greek coffee, with its robust flavor and thick texture, may take some getting used to for those unaccustomed to its unique taste. Greece ranks 17th among the world's top coffee-consuming nations, with an average annual consumption of 5.5 kilograms of coffee per person (*Visitgreece.gr*, 2018). The increased generation of spent coffee grounds (SCG) due to coffee consumption poses both challenges and opportunities. SCG, with their high residual oil content, offer a promising opportunity for biodiesel production, contributing to sustainable waste management practices and renewable energy objectives (Gu et al., 2023; Passadis et al., 2020). Gas chromatography coupled with Tandem Mass Spectrometry (GC-MS/MS) is considered an advanced method in analytical chemistry. The tandem configuration enhances sensitivity, selectivity, and the ability to analyze trace amounts of compounds, making it particularly useful for analyzing biodiesel components, like fatty acid methyl esters (FAMES), contaminants, and other impurities in biodiesel and its feedstock (Musharraf, Ahmed, & Zehra, 2015). The current study examines the FAME analysis and quantification in biodiesel samples derived from spent Greek coffee grounds (SGCG) using an optimized GC-MS/MS method employing multiple reaction monitoring (MRM) mode.

Materials and Methods

SGCG samples were provided by local businesses. Each sample was pretreated and thoroughly prepared before undergoing any conversion processes. All chemicals and FAME calibration standards used in the investigation were of analytical grade. The extraction of SGCG to obtain the coffee oil was performed using n-hexane as the solvent in a Soxhlet apparatus, with the solvent being recovered and reused for all extraction cycles. Acid-catalyzed esterification and alkali-catalyzed transesterification processes were conducted under the optimum selected conditions. Biodiesel samples were characterized and compared to their physicochemical properties based on the test methods outlined in the EN 14214 standard (Atabani et al., 2018; Emmanouilidou et al., 2024). GC-MS analysis was carried out using an Agilent 8890 GC system equipped with a 7000D GC/TQ triple quadrupole mass detector and a 7693A autosampler injector (1 µL sample volume), with data acquisition managed by the MassHunter workstation. The separation was performed on an Agilent DB 5MS column with helium as the carrier gas. The oven temperature was initially set to 150 °C for 1 minute, then increased to 190 °C at a rate of 20 °C/min for 5 minutes. It was subsequently raised to 230 °C at 4 °C/min and held for 1 minute before finally reaching 320 °C at 20 °C/min, where it was maintained for 2 minutes. All standards and samples were injected in split mode with a split-to-column flow ratio of 100:1. The collision energy of 5 eV was used to fragment precursor ions into product ions and optimized for each specific compound, where the dwell time was set at 50 ms.

Results and Discussion

The determination of the physicochemical properties of biodiesel samples revealed that most of them met the specifications outlined by EN 14214 methods. The identification and quantification of FAMES in biodiesel samples were achieved with high specificity and sensitivity using the MRM mode of GC-MS/MS. Each FAME was monitored through a set of transitions, where a precursor ion was fragmented into product ions at an optimized collision energy. One product ion was selected as the quantification ion, while additional product ions served as qualifiers. This approach provided excellent confidence in compound identification by ensuring that the monitored ion transitions were unique to each FAME, reducing the likelihood of interference from co-eluting compounds or background noise. The most abundant FAMES identified in biodiesel samples were methyl palmitate (C16:0), methyl stearate (C18:0), methyl oleate (C18:1), methyl linoleate (C18:2) and methyl linolenate (C18:3).

Conclusions

Spent Greek coffee grounds (SGCG), like the spent grounds left after brewing, is a byproduct of the coffee-making process. Instead of discarding it as waste, it can be repurposed in several environmentally friendly and innovative ways, contributing to waste reduction and sustainability. This study underscores the successful valorization of spent coffee grounds derived from Greek coffee into biodiesel, demonstrating their potential as a renewable feedstock within the framework of a circular economy. Utilizing this waste biomass supports sustainable energy objectives while contributing to effective waste management by converting organic waste into a valuable energy resource. The optimized GC-MS/MS method, employing multiple reaction monitoring (MRM) mode, is a robust analytical approach for identifying and quantifying FAMES in biodiesel. The MRM mode exhibits significant advantages, particularly in detecting minor and less stable components. This is due to MRM's ability to use both precursor and product ion transitions, enhancing selectivity and sensitivity, especially in complex matrices. The improved sensitivity and specificity ensure a comprehensive profile of FAMES, providing insights into the quality and composition of the biodiesel.

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