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Biodiesel Production by Supercritical Methanol from Jojoba Seeds Harvested in the Mesaoria Plain of Cyprus

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Abstract: Nowadays, interest in renewable energy sources such as biodiesel is increasing due to the limited resources and environmental impacts of fossil fuels. Jojoba plant (Simmondsia chinensis) is one of the oilseeds with significant potential for biodiesel production. This study investigates the viability of producing biodiesel from jojoba oil extracted from plants grown in the Mesaoria Plain of Cyprus. The Mesaoria Plain, with its favorable climate for jojoba cultivation, presents an ideal location for investigating the usability of this biodiesel feedstock. Supercritical methanol, known for its efficiency in transesterification reactions, offers a novel approach to converting jojoba oil into biodiesel. Through analysis and experiments, this work assesses the crucial variables—temperature, pressure, and reaction time—involved in the supercritical methanol process. Furthermore, the quality attributes of the biodiesel produced, like its fatty acid methyl ester content, viscosity, density, and cetane number, were determined in compliance with international biodiesel standards. In the supercritical methanol transesterification at 240°C and 8.3 MPa, the % achieved conversion in a separate portion of fatty acid esters was 97.8 %. The findings of this research contribute valuable insights into the potential of jojoba-derived biodiesel as a sustainable energy solution in Cyprus and beyond.

Keywords: Biodiesel, Jojoba seeds, Supercritical methanol, Energy engineering

Introduction

The limitation of energy resources, one of today's main problems, requires energy production from sustainable raw materials. Biodiesel, due to its biodegradability, reduced emissions, and compatibility with existing diesel engines, has proven to be a solution to the fuel problem due to its ability to be produced from sustainable energy sources (Knothe, 2010). Biodiesel is produced by a chemical reaction called transesterification, in which vegetable oils or animal fats react against alcohols of low molar weight (Al-Shanableh, 2017). Unlike traditional methods used in the transesterification process, the supercritical methanol method stands out for its higher efficiency and less by-product formation. The supercritical methanol (SCM) method that achieves a faster and more efficient transesterification process is based on the reaction of methanol with oils under supercritical conditions (under high temperature (>200 °C) and pressure (>5 MPa)) (Saka & Kusdiana, 2001; Al-Shanableh & Savas, 2023). Additionally, biodiesel produced by the supercritical methanol method may contain higher purity and fewer contaminants, providing reassurance about the effectiveness of this research.

Jojoba (Simmondsia chinensis), a perennial shrub native to arid regions, is particularly suited for biodiesel production, with its high oil content ranging from 45% to 60% and adaptability to semi-arid climates (Bilin, 2020). Jojoba oil is an oil obtained from the seeds of the Jojoba plant. This oil's chemical and thermophysical

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properties include many that are important for its industrial and cosmetic uses. Jojoba oil is an unsaturated liquid wax that is clear, golden and has no greasy or perfumed aftertaste. While the phrase "jojoba oil" is commonly used to refer to it, it is more appropriately called "wax," as it is a mixture of long-chain monounsaturated esters (97–98 weight percent) of fatty acids and fatty alcohols rather than a triglyceride like vegetable and animal fats are (Canoira et al., 2006). According to the standard of the International Jojoba Export Council (IJEC), the significant component of wax esters in jojoba oil with C40 and C42, of which monounsaturated fatty acids should be oleic acid (C18:1), eicosenoic acid (C20:1) and erucic acid (C21:1) (IJEC, 2018). The fatty acid compositions of jojoba oil would vary significantly depending on the soil and climate where the jojoba shrub is grown, when it is harvested and how the oil is processed.

Jojoba oil has the exceptional qualities of a liquid wax with low and consistent viscosity at varying temperatures, good lubricity, and other physicochemical characteristics such as high boiling, smoke, flash point, and low chemical reactivity. In lubricants and other industrial applications, jojoba oil has supplanted whale sperm oil as the primary natural source of liquid wax esters (Lei & Li, 2015). Lubricants, medications, and cosmetics are among the industries that use jojoba oil and its derivatives. Jojoba oil is an excellent lubricant for metal cutting, tool work, and high-speed machinery in the automobile industry. Furthermore, the viability of using jojoba methyl ester for biodiesel on a commercial scale is highly dependent on the added value (about 1.16 $/kg) achieved for the fatty alcohols that are created as a side product during the transesterification process (Bouaid et. al., 2007). Different studies showed jojoba oil was successfully used as a raw or biodiesel in different proportions in diesel engines. Both Canoira et al. (2006) and Bouaid et al. (2007) studied to convert jojoba oil to biodiesel using base catalyst transesterification at moderate operation conditions, and they obtained 97 % and 83.5 % conversion yield of esters, respectively. In 2015, Lei and Li transesterified jojoba oil, an enzymatic lipase catalyst, and the maximum conversion they obtained was 97 %. With the SCM method, Singh et al. (2022) achieved 95.7 % conversion within 23 min at 123 bar and 287 °C. In their work, Selim et al. (2022) mixed raw Jojoba oil in different proportions with diesel fuel, and they showed that the mixture could serve as a fuel for diesel engines without any modifications. The raw Jojoba oil was found to be a suitable, promising alternative to the diesel fuel used in diesel engines with viscosity adjustment.

This study aimed to convert jojoba oil extracted from plants grown in the Mesaoria Plain into biodiesel using the SCM method in a supercritical reactor designed and constructed in previous studies.

Method

Extraction of Jojoba Oil

The jojoba seeds were harvested from the Mesaoria Plain test plantation. A typical jojoba seed was an oblong to oval shape produced by a female jojoba plant. The process of extracting oil involves using a screw machine that applies pressure to the seeds within a cylindrical chamber, causing the jojoba oil to be collected through outlet holes located at the bottom of the chamber by preventing the solid press cake from passing through (Al-Shanableh et al., 2020). The jojoba oil was allowed to settle and precipitate for three days following extraction. Several physicochemical properties of the jojoba oil extracted were also examined and listed in Table 1, as required by IJEC quality standards.

Table 1. Various physicochemical properties of Jojoba oil extracted

Physicochemical characteristics	**Values obtained**	**Method**	**IJEC Standards**
Specific gravity (g/cm^3)	0.86	AOCS Cc 10 a	0.86-0.87
Kinematic viscosity at 40 ˚C (mm^2/s)	24.5	ASTM D 445	-
Kinematic viscosity at 100 ˚C (mm^2/s)	6.45	ASTM D 445	-
Refractive Index	1.47	AOCS Cc 7 25	1.45-1.47
Saponification (mg KOH/g)	92.6	AOCS Cd 3-25	88.0-96.0
Acid Value (mg KOH/g)	0.5	AOCS Ci 4-91	≤1.0
Iodine Value (gram)	88.9 /100	AOCS Cd 1-25	882-87 /100
Peroxide Value ($meqO_2/kg$)	<1.0	AOCS Cd 8-53	≤ 2.0

Table 1 illustrates the fatty acid compositions of the jojoba oil samples produced from the seeds of jojoba shrubs grown on the Mesaoria Plain in TRNC.Jojoba oil is a wax ester comprising esters of fatty acids and fatty alcohols. The predominant constituents found in jojoba oil in this study were the wax esters C40 and C42, which align with the specifications outlined in the IJEC standard (IJEC, 2018). C40 wax ester consists of C22:0

alcohol and C18:1 fatty acid, while C42 consists of C22:0 alcohol and C20:1 fatty acid. Eicosenoic acid, also known as C20:1, emerges as the primary component within the specified limits of the IJEC standard.

Table 2. Fatty acids and wax esters compositions of jojoba extracted

Fatty acid and wax ester composition	%	IJEC Standards Limits
Palmitic acid (C16:1)	1.89	≤3.0
Palmitoleic acid (C16:1)	0.07	≤1.0
Stearic acid (C18:0)	0.46	-
Oleic acid (C18:1)	12.3	5.0-15.0
Linoleic acid (C18:2)	0.21	-
Linolenic acid (C18:3)	0.59	-
Arachidic acid (C20:0)	0.13	-
Eicosenoic acid (C20:1)	71.1	65.0-80.0
Behenic acid (C22:0)	0.18	≤1.0
Erucic acid (C22:1)	11.7	10.0-20.0
Nervonic acid (C24:1)	1.15	≤3.0
Wax Ester Compositions		
C40 wax ester	28.2	26.0-34.0
C42 wax ester	46.3	44.0-56.0
C44 wax ester	8.0	8.0-12.0
C46 wax ester	0.5	0.0-3.0

Experimental Setup for Supercritical Methanol

The experimental set-up consisted of a bench-scale, batch-type reactor designed and manufactured to manage SCM's demanding transesterification process conditions, which are characterized by high temperature and pressure, as depicted in Figure 1 (Al-Shanableh & Savas, 2022). This figure's letters are designated as follows: A: Pressure vessel, B: Electrical external heater, C: Hatch (vessel cover), D: Thermocouple, E: Adjusting lever, F: Extruded fins, G: Reactor pressure gauge.

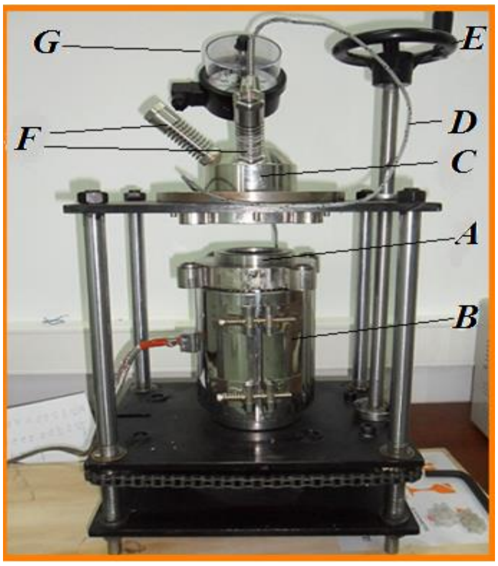

Figure 1. The reactor designed and manufactured for supercritical biodiesel production

The experimental configuration for the one-step supercritical methanol transesterification process was tailored to function effectively under elevated temperatures and pressures. Nitrogen gas was an inert medium to generate pressure, and a pliable high-pressure hose linked the supercritical reactor. This reactor was outfitted with various components, including an external heater, insulating mantle, electromagnetic stirrer, safety valve, pressure gauge, and thermocouple for temperature monitoring. Additionally, a laboratory-grade double-pipe heat exchanger was utilized as the condenser.

Experimental Procedure for Biodiesel Production by Supercritical Methanol

The process of supercritical methanol transesterification of jojoba oil was conducted utilizing the previously outlined experimental arrangement above and adhering to the procedural steps illustrated in the flowchart depicted in Figure 2.

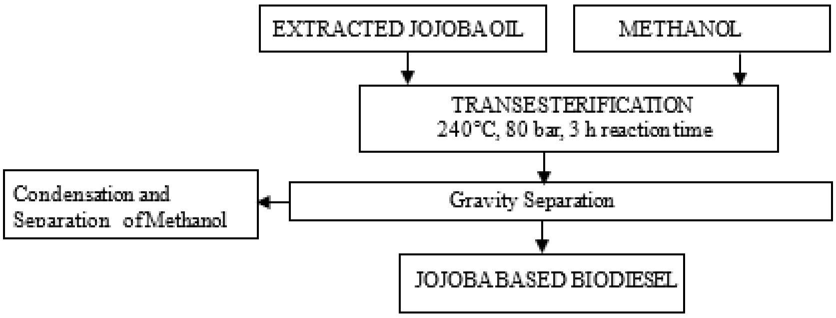

Figure 2. Flowchart for the experimental supercritical methanol procedure of jojoba biodiesel production

The procedure commenced by introducing a blend of extracted jojoba oil and methanol (the oil-to-alcohol molar ratio of 1:41 approximately corresponded to a 1:2 volume ratio) into the supercritical reactor, followed by thorough mixing to achieve a uniform mixture. Subsequently, the reactor was sealed, and temperature and pressure were adjusted to enter the supercritical fluid phase. The reaction duration was arranged as 120 min because this was the optimum duration obtained in the authors' previous study (Al-Shanableh & Savas, 2023). After the designated reaction time was completed, the reaction vessel was removed from the heating jacket to initiate the cooling process. Excess methanol was then transferred to the condenser. At the same time, the product mixture was poured into the distillation column to separate volatile fatty alcohol fraction from fatty acid methyl esters of jojoba (FAME). All other types of vegetable oils/animal fats are made up of triglyceride molecules, so the results of the transesterification process will be glycerol and FAME, unlike the transesterification of jojoba oil. Following the production and separation process, quality assurance measures were implemented to assess the biodiesel's properties, such as viscosity, cloud point, and pour point, following relevant ASTM and EN-ISO standards.

Results and Discussion

Biodiesel Yield by Supercritical Methanol Transesterification

Several physicochemical properties of the end product of supercritical methanol transesterification of jojoba were analyzed to assess its suitability as fuel for diesel engines. Testing methods from ASTM, EN, or ISO standards were employed, and the obtained values for the sample were compared with reference values for biodiesel according to ASTM D6751 and EN 14214. The results are presented in Table 3.

Table 3. Physicochemical fuel properties of the jojoba-biodiesel produced

	Method	**Limits**	**Jojoba based FAME**
Kinematic viscosity at 40 ˚C (mm^2/s)	ASTM D 445	1.9-6.0	5.210
Higher heating value (MJ/kg)	ASTM D 4809	--	44.3
Ester contents (wt %, max.)	EN 14103	96.5	97.8
Linoleic acid methyl esters (wt %, max.)	EN 14103	12.0	0.4
Iodine value (g I_2 / 100 g, max)	EN 14111	120	54
Cloud point (˚C)	ASTM D 2500	--	12
Cold filter plugging point (˚C)	ASTM D 6371	--	10
Pour point (˚C)	ASTM D 97	--	8.6

All measured properties were within the range of ASTM/ EN standards. The authors' previous studies with various vegetable oils have proven that high unsaturated fatty acids content shows low cold flow properties (Al-Shanableh et al., 2023). Three cold flow properties, namely cloud point, cold filter plugging point, and pour point of jojoba-based biodiesel, are relatively high despite having 97% monounsaturated fatty acids content. This is because jojoba oil is a wax, and jojoba-based biodiesel not only contains FAME but is also a mixture of fatty acid methyl esters and fatty alcohols.

Conclusion

Utilizing supercritical methanol to produce biodiesel from jojoba seeds harvested in the Mesaoria Plain of Cyprus offers a promising pathway toward sustainable fuel production, an alternative to traditional diesel. The supercritical fluid approach demonstrated notable efficiency and decreased process time compared to base-catalyzed transesterification. The single-step procedure in the bench-scale reactor achieved considerable conversion rates within a comparatively brief timeframe. These findings suggest that the supercritical method holds promise for scaling up biodiesel production, primarily owing to its abbreviated reaction duration.

Recommendations

Further investigation and fine-tuning of process parameters can elevate conversion rates further and enhance overall efficiency in biodiesel production utilizing supercritical methanol. With adaptability to arid climates and an efficient conversion process, jojoba presents a viable feedstock for biodiesel production in Cyprus. However, successful implementation requires careful consideration of technical, economic, and environmental factors and long-term commitment to sustainable development goals..

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

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