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Effect of Ultrasonic Waves on Aspire Biodiesel and Comparison of Its Properties with Petroleum Diesel

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ABSTRACT

This study aimed to determine the effect of ultrasonic sound waves on modifying the chemical structure of biodiesel to bring its physical properties closer to petroleum diesel. In this direction, safflower oil was selected because its fatty acid composition is similar to fatty acid esters of petroleum diesel and is a sustainable source. Refined safflower oil, the free fatty acid content of which was determined, was reacted with methanol under NaOH catalyst to perform the transesterification reaction. After biodiesel production, samples were incubated in an ultrasonic bath for 60, 120, and 180 minutes. FTIR, density, free fatty acid content, flash point, viscosity, and cloud point analyses investigated the effect of incubation times on biodiesel's chemical structure and properties. FTIR spectra showed that ultrasonic sound waves partially decomposed fatty acid methyl esters and increased the number of volatile components in biodiesel. The flash point of biodiesel has been associated with a decrease of 89°C, and the low flash point is expected to increase fuel efficiency. Kinematic viscosity values were measured in the 3.4583-3.5115 mm²/s range, and density values were measured in the 0.8820-0.8872 g/ml range. These values show that biodiesel complies with national and international standards. As a result, the ultrasonic bath process applied to biodiesel showed a similar result to chemical modification methods by affecting the structure of fatty acid chains. Thus, it brought the physical properties of biodiesel closer to petroleum diesel. It is seen that this method is a more efficient alternative for biodiesel production because it does not use additional chemicals, and the process is faster. In conclusion, by increasing the production of the drought-resistant safflower plant, sustainable energy resources will be contributed, while its waste can be evaluated as animal feed. Ultrasonicated safflower biodiesel can also be used as an efficient, environmentally and mechanically friendly alternative fuel source.

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Introduction

The rapid increase in the world population and the resulting increase in energy demand necessitate the use of fossil fuels, which in turn leads to the accumulation of CO_2 in the atmosphere, bringing severe environmental problems such as climate change. In addition to the ecological damage caused by fossil fuels, the rapid depletion of oil reserves has made it inevitable to seek alternative energy sources. In this context, interest in environmentally friendly and sustainable energy sources is increasing, and biodiesel stands out as a remarkable alternative among these sources (A. H. Demirbas & Demirbas, 2007; Moodley, 2021; Zaimes et al., 2015). Biodiesel is a fuel type obtained from sustainable vegetable and animal oils and can be mixed with petroleum diesel in diesel engines. A high cetane number, low toxic and sulfur content, and fewer greenhouse gas and exhaust emissions than diesel fuel are just a few reasons why biodiesel is preferred. In addition, the high flash point of biodiesel increases the safety of use and offers an advantage in terms of cost (A. H. Demirbas & Demirbas, 2007; Hasan & Rahman, 2017; Mofijur et al., 2016; Moon et al., 2010; Na et al., 2015; Simsek, 2020).

The source of the oil used in biodiesel production is critical to the quality and efficiency of the biodiesel (Aktaş et al., n.d.; Nogales-Delgado et al., 2021; Verma & Sharma, 2016; Zhenyi et al., 2021). In this context, the safflower plant (*Carthamus tinctorius L.)* has significant potential for biodiesel production. The safflower plant is widely used worldwide in various areas such as food, cosmetics, pharmaceuticals, and feed industries. However, despite its rich essential fatty acid content, safflower oil is not preferred much in edible products. Safflower, grown primarily for oil production, contains high unsaturated fatty acids. Safflower oil is known for its very high levels of linoleic acid, an essential fatty acid, which provides a suitable fatty acid profile for biodiesel production (Abbasi et al., 2024; Khalid et al., 2017; Nogales-Delgado et al., 2021; Yesilyurt et al., 2020). The remaining waste from biodiesel production is also used as animal feed (T. Şahin & Sural, 2020). For these reasons, safflower has become a preferred plant in biodiesel production.

In the production of biodiesel, the conventional methods are as important as the source of the oil used. Traditional biodiesel production methods are generally carried out by the transesterification reaction of triacylglycerols with methyl alcohol under the catalyst of NaOH (Eryılmaz & Erkan, 2015; S. Şahin & Yılmaz, 2024). These methods typically require long reaction times, which increases production costs. Recent studies have focused on advanced techniques to accelerate biodiesel production and improve efficiency. One of these techniques is ultrasonic bath applications (Fan et al., 2010; Hanh et al., 2009; Martinez-Guerra et al., 2014; Martinez-Guerra & Gude, 2014; Parida et al., 2012; Siatis et al., 2006; Stavarache et al., 2005). Ultrasonic bath is an innovative method to expedite the transesterification reaction in biodiesel production. Ultrasonic waves create cavitation in liquid media, allowing components such as oil and alcohol to mix more homogeneously. This cavitation causes micro bubbles to form in the liquid and high energy output when these bubbles burst. This energy accelerates chemical reactions, shortens the reaction time, and increases transesterification efficiency. The transesterification process, which can take one or two hours with traditional methods, can be completed in minutes using an ultrasonic bath (Badday et al., 2013; Fan et al., 2010; Hanh et al., 2009; Martinez-Guerra et al., 2014; Parida et al., 2012; Patil et al., 2021; Siatis et al., 2006; Stavarache et al., 2005). Using ultrasonic baths in biodiesel production speeds up the process and reduces energy consumption, reducing the environmental footprint of production. This makes biodiesel production more commercially attractive. In addition, ultrasonic bath applications can significantly improve biodiesel's chemical structure and physical properties. Positive effects of ultrasonic bath applications have been observed, especially in parameters that directly affect fuel performance, such as viscosity, density, and flash point (Badday et al., 2013; Patil et al., 2021; Stavarache et al., 2005). In this study, unlike the studies in the literature, the sonication process was applied to pure biodiesel after traditional methods produced biodiesel. Biodiesel samples were incubated in an ultrasonic water bath for different periods, and the effect of this incubation period on biodiesel's physical and chemical properties was investigated.

This study focuses on the safflower plant, one of the oils with potential biodiesel production. It is known that drought-resistant plant species are suitable for biodiesel production in various regions of Turkey, especially in areas with water shortages, and the safflower plant has an essential place in this context. With its drought-resistant structure, the safflower plant is a suitable raw material source for biodiesel production in regions with limited water resources. This feature of aspirin offers an alternative solution for biodiesel production, especially in areas with water shortages. In addition, increasing the cultivation of the safflower plant for sustainable energy production can contribute to local economies by utilizing dry areas and encouraging the use of local resources for biodiesel production (Abbasi et al., 2024; Aktaş et al., n.d.; Altıntop & Gidik, 2019; Khalid et al., 2017; Simsek, 2020).

In this study, biodiesel production from refined safflower oil using conventional chemical methods using NaOH catalyst was carried out, and the produced biodiesel samples were subjected to ultrasonic bath application for different durations. The effects of ultrasonic bath time on biodiesel's physical and chemical properties were investigated, and the potential of this process to improve the engine performance parameters and environmental impact of biodiesel was evaluated. The chemical structures of biodiesel samples treated with an ultrasonic bath were investigated by FTIR analysis, and physical properties such as viscosity, density, and flash point were evaluated using various qualitative and quantitative methods. As a result, it was aimed to reveal that using the ultrasonic bath method in biodiesel production from safflower oil not only improves the production process but also has the potential to increase the performance of biodiesel.

Materials and Methods

Safflower oil was used in refined form and was supplied from the market, while the chemicals needed for biodiesel production were used in analytical purity from Sigma (USA).

Free fatty acids are not desired in biodiesel production due to their saponification effect. Their amount should be at most 0.5% according to the EN 14104 European Standard (accepted as the Turkish Biodiesel Standard) (Sabudak & Yildiz, 2010). Therefore, the free fatty acid amount of refined safflower oil was determined first by volumetric analysis. The oil sample was titrated with 0.1 N KOH prepared in ethanol with a phenolphthalein indicator. The free acid number in safflower oil expresses the weight in mg of potassium hydroxide required to neutralize 1 gram of oil. Each ml of 0.1 N KOH consumed equals 0.028 g of oleic acid. The analysis sample's FFA (Free Fatty Acid, In terms of oleic acid) was calculated as the percentage of oleic acid according to Equation 1.

$$\%\text{FFA}\ () = \left({[V]}/{[m]} \right) \times 0.028 \times 100 \qquad (1)$$

V is the amount of 0.1 N KOH (in ethanol) solution used in the titration in ml; m is the mass of the safflower oil sample in g.

Since the results in determining free fatty acidity were, on average, 0.223% in terms of oleic acid, below 0.5% according to EN 14104 standards, the next stage, biodiesel production, was started without any additional processing.

Biodiesel Production

Biodiesel production is based on the esterification of fatty acids in the structure of triacylglycerols by interacting with low molecular weight alcohols and separating them from glycerol (Eryılmaz & Erkan, 2015; S. Şahin & Yılmaz, 2024; Zhenyi et al., 2021).

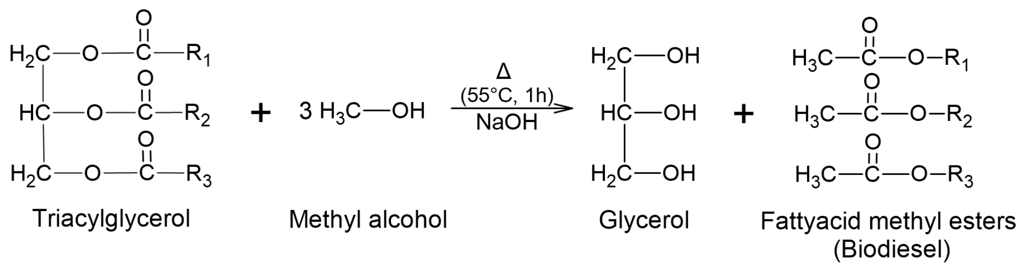

Figure 1. Transesterification reaction equation (Drawn with Chem Sketch program)

Table 1. Biodiesel samples and name abbreviations

Abbreviations	Explanation
S0	Non-ultrasonic treated safflower biodiesel
S1	60 minutes ultrasonic treated safflower biodiesel
S2	120 minutes ultrasonic treated safflower biodiesel
S3	180 minutes ultrasonic treated safflower biodiesel
OS	Safflower oil

Glycerol, which makes the oil thick and sticky, is removed from the medium after transesterification, leaving behind thinner, lower-viscosity fatty acid esters. Methyl alcohol (CH_3OH), a primary alcohol with the lowest molar mass and steric hindrance, and high volatility was preferred for transesterification. The transesterification process of safflower oil was done by reacting one mole of triacylglycerol with 3 moles of mono alcohol stoichiometrically.

In the transesterification process, NaOH was used as a catalyst. The method found suitable in our previous study was used in biodiesel production (Şimşek, 2024). Firstly, the catalyst was dissolved in methyl alcohol at room temperature with the help of a magnetic stirrer. Then, 1000 ml of safflower oil was placed in a narrow-mouthed boroxylate glass bottle and heated at 55°C. The alcohol-catalyst mixture was added to the hot oil at room temperature in a controlled molar ratio of 5:1 alcohol/oil. The system was completely closed to the atmosphere to prevent alcohol loss. The process was continued for 1 hour at 55°C on a heated magnetic stirrer to complete the reaction. After the reaction, the two main products obtained, glycerin and biodiesel, were taken into a separating funnel, kept at room temperature overnight, and separated using the density difference (Şimşek, 2024).

Application of Sonication to Biodiesel

While studies in the literature use sound waves in an ultrasonic bath for the transesterification process, previous study has shown that sonication applied to biodiesel, the final product of the transesterification, positively affects its characteristics (Şimşek, 2024). For this reason, the sonication process was used for safflower oil biodiesel, which has chemically different fatty acids from hazelnut oil, at the same frequency (80 Hz) and durations (60, 120, and 180 min) in a sonicated water bath at room temperature (Elmasonic P, Spain). For this process, 1000 ml of safflower oil biodiesel was placed in a closed glass container and then in a sonicated water bath. Ultrasonic sound waves at 100% power at 80 Hz were applied to the samples for 60, 120, and 180 min. At each time, 250 ml of safflower biodiesel was taken from the environment, placed in a clean boroxylate bottle, labeled as in Table 1, and stored at room temperature for analysis.

Analysis

According to ASTM D975 standards, petroleum-derived diesel fuel is used in diesel engines because it contains distillate gas oils with lower volatility and is suitable for use in relatively higher loads and single-speed engines. Therefore, biodiesel is generally compared with this fuel. According to ASTM D 975 diesel and biodiesel fuel standards, Diesel No.2 is accepted as a reference and compared with biodiesel as a fuel in diesel engines (Atabani et al., 2013). In the evaluation of the analysis results, the ASTM 975 standard was taken into consideration because it is the most accepted in biodiesel and diesel. In addition, ASTM D6751 and EN ISO 14104 standards prepared for the properties of biodiesel were also taken into consideration in the evaluation of some results.

The amount of free fatty acid in safflower oil was carried out before the biodiesel production experiments started. The amount of free acid in the synthesized biodiesel samples before and after ultrasonication was titrated with KOH and calculated in terms of oleic acid.

The chemical structures of biodiesel samples synthesized from safflower oil and the changes occurring in their structure depending on the ultrasonication time were investigated spectroscopically using Fourier-transform infrared spectroscopy (Bruker ALPHA ATR-FTIR, Germany) in the wavenumber range of 400-4000 cm^{-1} at a resolution of 4 cm^{-1}. The obtained results were evaluated comparatively.

The elemental content in the range of Na (11) to U (92) and the sulfur percentage of biodiesel samples were analyzed using a Spectro Xepos II (Germany) X-ray fluorescence spectroscopy (XRF) device.

The viscosity determination, which is one of the essential properties of biodiesel, was carried out using a Herzog HVM 472 (USA) automatic kinematic viscosity measuring device, compliant with ASTM D445, ASTM D446, IP 71, ISO 3104, ISO 3105, and GOST 33 standards. The flash point of biodiesel samples was measured using a Pensky-Martens FP93 5G2 model closed-cup flash point analyzer specific to diesel samples. The density of the samples was measured at 15°C using a Kem Kyoto 640B automatic density meter. The density values and API gravity were examined comparatively with relevant standards. The cloud point (cold filter plugging point) analysis of biodiesel samples was performed using a Koehler (Germany) device with three repetitions. The results were compared with standard values for petroleum diesel, and their compliance with the relevant standards was evaluated.

Results and Discussion

FTIR Analysis Results

Fourier Transform Infrared Spectroscopy (FTIR) is a spectroscopic analysis method that provides information about a substance's chemical structure and components. FTIR generates a spectrum by detecting the absorption or reflection of infrared radiation passing through a sample, identifying the characteristic vibration frequencies associated with different functional groups in molecules. This analysis is widely used to verify the chemical composition of biodiesel, oils, polymers, and other organic compounds. FTIR results indicate which functional groups are present in the structure of a sample (Berthomieu & Hienerwadel, 2009; Smith, 2011). For example, in biodiesel samples, a strong peak observed around 1740 cm^{-1} for the carbonyl group (C=O) indicates the presence of esters, commonly seen in biodiesel production. Asymmetric and symmetric stretching vibrations for methyl and methylene groups ($-CH_3$, $-CH_2$) are observed in the range of 2920-2850 cm^{-1}, indicating the presence of fatty acids. Peaks observed between 1180-1200 cm^{-1} corresponding to ester bonds ($-O-CH_3$) confirm the formation of biodiesel and the presence of methyl esters (Şimşek, 2024).

When comparing the FTIR analysis of biodiesel samples that did not undergo ultrasonic bath treatment with those subjected to different exposure times, it was determined that the transesterification reaction applied to safflower oil was completed. The esterification rate was found to occur at similar levels in biodiesel samples obtained from both oil sources. It was also identified that ultrasonic treatment applied for different durations after biodiesel production caused changes in the chemical structure of the samples. The overall FTIR spectra of biodiesel samples synthesized from safflower oil are presented in Figure 2 and examined in detail in Figure 3. Shifts were generally observed in the specific peak regions of the ultrasonically treated and untreated samples, with partial differences in peak intensities also noted. Additionally, the FTIR analyses showed that the proportion of fatty acid methyl esters changed with sonication time, and the chain structure was found to alter during the sonication process.

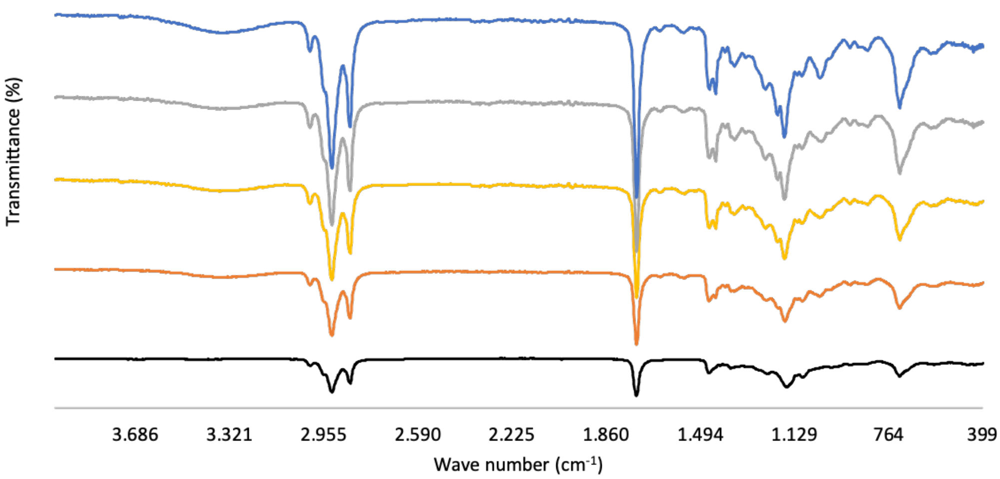

Figure 2. FTIR spectra of biodiesel samples obtained from safflower oil.

Table 2. Absorption peaks and their properties were observed for safflower in the FTIR spectrum.

Group Vibration	Vibration Type	Wave Number (cm^{-1})				
		OS	S0	S1	S2	S3
-O-H	Stretching band	-	3610-3024	3610-3024	-	3610-3024
=CH	Unsaturated bond stretching	3008	3010	3009	3008	3008
$-CH_2$	Asymmetric/ Symmetrical stretching vibration	2923	2924	2924	2924	2924
-C(=O)-	Saturated acyclic ester stretching	1743	1741	1741	1741	1741
$-CH_2$	Shear Type Stress	1460	1458	1458	1460	1458
$-CH_3$	Asymmetric bending: specific to biodiesel	-	1436	1436	1436	1436
$-O-CH_2-$	Glyceride bending vibration	-	1362	1362	1362	1362
O-H	O-H Deformation	1225	1239	1237	1237	1235
$-O-CH_3$	Stretching: Typical for biodiesel	-	1186	1190	1195	1199
C-O-C C-C	Symmetric stretching	1154	1164	1166	1166	1166
-C-OH	Primer Alcohol	-	1025	1021	1015	1023
Cis -C=C-	Planar vibration	719	721	719	721	717

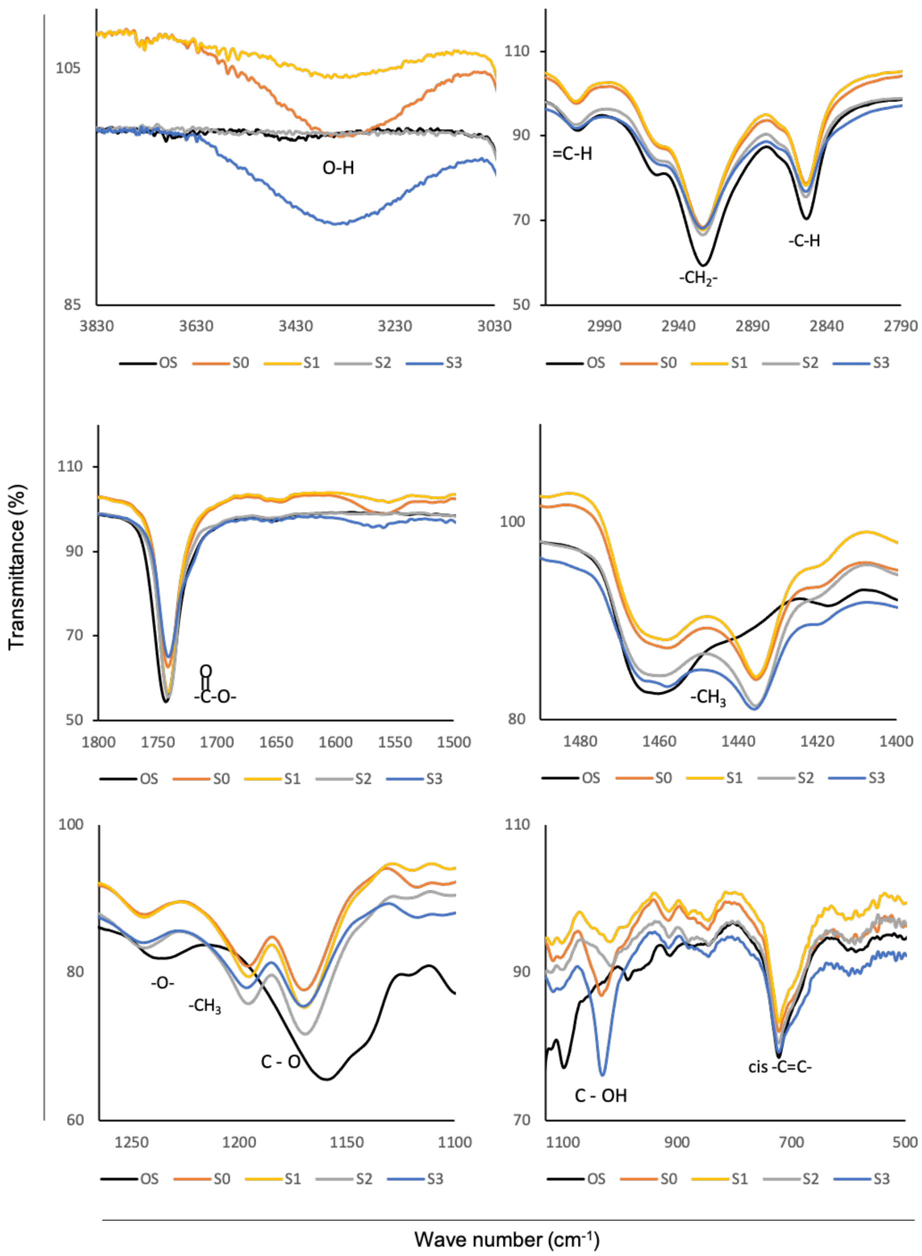

Figure 3. A detailed examination of FTIR spectra for biodiesel samples.

Although no changes were observed in the peak values within the fingerprint regions of the samples prepared from safflower oil, partial changes in the intensity of some peaks were noted. Additionally, it was determined that the chain structure altered during the sonication process. The significant absorption peaks and their characteristics are presented in Table 2.

In sample S3, an increase in peak intensity observed at the 1030 cm^{-1} region indicates an enhancement in C-O-C stretching vibrations. While this peak intensity decreases in samples S1 and S2, it is similar to the untreated sample S0. Additionally, the increase in bandwidth in the 3000-3600 cm^{-1} range suggests the presence of alcohol groups or unsaturated bonds in the structure. The FTIR spectrum shown in Figure 3 illustrates the structural analysis of the biodiesel samples. The peaks at specific wavenumbers reveal the biodiesel's chemical components and functional groups. Upon examining the spectra, peaks at 2922 cm^{-1} and 2853 cm^{-1} correspond to the asymmetric and symmetric stretching vibrations of $-CH_2$ groups, confirming the presence of the aliphatic chain structure and long carbon chains in biodiesel. The peak observed at 1741 cm^{-1} corresponds to the C=O stretching vibrations of esters, verifying the ester content in biodiesel. Its intensity indicates the successful completion of transesterification and a high degree of esterification. This peak, observed in the 1735-1750 cm^{-1} range, is characteristic of ester structures and appears independent of the ester type, thus seen in all samples. However, in sample S3, subjected to three hours of sonication, the decrease in peak intensity indicates a lower concentration of esters in this sample. The moderate intensity peaks observed in the 1460-1445 cm^{-1} range correspond to the stretching vibrations of $-CH_2$ and $-CH_3$ groups, representing the methyl ester components of biodiesel. Peaks at 1196 cm^{-1} and 1016 cm^{-1} represent the $O-CH_3$ and C-O-C stretching vibrations specific to biodiesel. Remarkably, the peak at 1196 cm^{-1} reflects the presence of methyl esters and highlights the typical characteristics of biodiesel. (Cazorla et al., 2002; Forfang et al., 2017; Miglio et al., 2013; Şimşek, 2024). These peaks provide essential clues for understanding the chemical structure of biodiesel samples. The FTIR spectrum confirms the esters formed due to the transesterification process, along with aliphatic hydrocarbon chains and other functional groups. In conclusion, the spectral details in Figures 2 and 3 demonstrate that the biodiesel samples were successfully produced, and their chemical components were accurately identified.

Figure 3, which provides a detailed examination of the FTIR spectrum, clearly shows some differences among the samples. The peak corresponding to the -CH_3 asymmetric bending vibrations observed in the 1425-1460 cm^{-1} range is present at similar levels across all samples, with an increased intensity in S3. Although this peak is characteristic of biodiesel, it also includes methyl types from alkyl and alcohol sources in the environment. It thus is not considered a primary indicator of transesterification. However, the -O-CH_3 stretching vibrations and –OH deformation peak in the 1188-1220 cm^{-1} range represent methyl esters and accurately reflect transesterification and biodiesel. This peak, not observed in OS but with higher intensity in S2 and S3, indicates a higher concentration of fatty acid methyl esters in these samples. The difference in intensity of the methyl ester peaks between S2 and S3 and between S0 and S1 in the same frequency range highlights the concentration difference of methyl structures not present in ester structures. The C–O–C symmetric stretching vibrations observed at 1154-1166 cm^{-1} with the highest intensity in OS are generally attributed to carbonyl structures, including glycerides and esters. The decrease in peak intensity observed in the biodiesel samples in this region is attributed to the breakdown of glyceride structures due to transesterification and the corresponding formation of methyl ester peaks in the 1188-1220 cm^{-1} range (Cazorla et al., 2002; Forfang et al., 2017; Miglio et al., 2013; Şimşek, 2024).

Comparing the FTIR analyses of biodiesel samples from safflower plant oils that did not undergo ultrasonic bath treatment with those subjected to different sonication times, it was found that the sonication process increased the formation rate of fatty acid methyl esters, thereby improving the biodiesel yield. Additionally, the physical properties of the sonicated biodiesel samples were closer to those of diesel fuel. In summary, the FTIR spectra revealed peaks consistent with the ester structure of biodiesel, and no peaks indicative of impurities or unremoved alcohol were observed.

Free Fatty Acid Measurements

The free fatty acid content of safflower oil was volumetrically measured before biodiesel production by titrating with a 0.1 N KOH solution, and the result was calculated as 0.223. Since the free fatty acid amount for safflower oil was below 0.5% according to the standard, no additional treatment was deemed necessary to reduce the free fatty acid content, and biodiesel production proceeded without any further processing.

$$R-COOCH_3 \xLeftrightarrow{\text{Sonication}} R-COOH + CH_3-OH$$

Figure 4. Possible equilibrium reaction of fatty acid methyl ester during sonication process.

Table 3. Percentage of free fatty acid content of safflower biodiesel samples as oleic acid.

Samples	Free Fatty Acids (%)
OS	0.233
S0	0.560
S1	26.43
S2	5.264
S3	7.060

FTIR spectra analysis of biodiesel samples from safflower oil (OS, S0, S1, S2, S3) revealed changes in the free fatty acid levels in the environment. The free organic acid amounts in these samples, both with and without sonication (S0, S1, S2, S3), were calculated as oleic acid percentages using Equation 1 and presented in Table 3. The highest free fatty acid content is expressed as oleic acid, at 26.43% in sample S1. Samples S2 and S3 showed values close to each other but significantly lower than S1. Among the sonicated samples, the free fatty acid content of S2 was closest to that of the non-sonicated sample S0, which is consistent with the FTIR results. This suggests that some fatty acids separated from glycerol during transesterification did not esterify with methyl alcohol. Additionally, it was observed that short-duration sonication could disrupt ester structures, while longer sonication times tended to favor the advancement of esterification reactions. Based on these results, it is recommended that sonication be applied for more than three hours to reduce the free fatty acid content in safflower biodiesel effectively.

Viscosity Measurement

According to the ASTM D975 standard, the viscosity range for No. 2 diesel is 1.9-4.1 mm^2/s (ASTM, 2017). In the ASTM D6751-08 standard (based on method D 445) (ASTM, 2008), this range is accepted as 1.9-6 mm^2/s for B100 biodiesel. One of the critical properties of biodiesel is its viscosity. As the viscosity increases, the fuel's flowability decreases, which is crucial for the fuel delivery system. Smaller fuel droplets injected into the combustion chamber mix more readily with air, heat more effectively, and ignite more readily. High viscosity at low temperatures can affect the fuel's flow and lead to issues with fuel injection, potentially requiring higher pumping pressures. Additionally, viscosity increases as the length of the hydrocarbon chains increases. Conversely, the presence of double bonds in the chains tends to reduce viscosity (A. Demirbas, 2005; Knothe & Steidley, 2005). As with diesel, the minimum viscosity value does not pose a problem in 100% biodiesel. However, maximum viscosity values can lead to issues such as poor combustion and accumulation of solid particulates. This results in the fuel penetrating deeper into the cylinder, which can cause incomplete combustion and more significant engine oil dilution, ultimately leading to higher pumping pressures (Edition, 2008).

Applying ultrasonic bath treatment not only provides chemical benefits to biodiesel but also affects the physical property of viscosity for safflower biodiesel. For safflower biodiesel, a decrease in viscosity was observed with one hour of exposure, which then increased in subsequent hours. This is reflected in the FTIR spectra, where ultrasonic cavitation is seen to enhance esterification and support regular trans geometric isomerism during the first hour. This situation suggests that the relative increase in molecular mass and tighter packing in molecular volume contribute to the rise in viscosity. Additionally, unlike our study with hazelnut oil (Şimşek, 2024), the FTIR spectra of safflower biodiesel exposed to short-duration sonication show an increase in the amount of mono alcohol present in the environment. In the case of hazelnut biodiesel, this increase in mono alcohol was observed with long sonication durations. This result demonstrates that the outcomes of sonication on plant-based biodiesel can vary depending on the chemical structure of the plant source. As a result, the kinematic viscosity values of sonicated and non-sonicated safflower biodiesel samples obtained in this study were closer to the standards than those reported in the literature. According to the values presented in Table 4, the application of ultrasonic bath treatment, in addition to the chemical benefits it provides to biodiesel, also slightly increases the viscosity of safflower biodiesel as the exposure time increases (ASTM, 2008, 2017; Knothe & Steidley, 2005).

Density and API Gravity

The density measurement results of the biodiesel samples are provided in Table 5. The API gravity was also measured using the same device, configured through the software according to Equation 2, and the results are presented in the same table. API gravity, which expresses the ratio of petroleum density to water density, decreases as density increases. Therefore, the relatively higher density of the obtained biodiesels resulted in API gravities lower than the natural diesel API gravity of 37.56. For diesel, the density value at 15°C is expressed as 820-860 kg/m³ according to ASTM D975 standards. For biodiesel, it is given as 880 kg/m³ according to ASTM D1298 test method and ASTM 6751 standard, and 860-900 kg/m³ according to EN ISO 3675/12185 test method and EN 14214 test method (ASTM, 2008, 2017; Atabani et al., 2013; Şimşek, 2024).

$$\text{API gravity} = \frac{141.5}{\text{specific gravity}} - 131.5 \qquad (2)$$

In the Eryılmaz et al. (2014) study, the density value of biodiesel produced from safflower seed oil was 0.885 g/ml at 15°C (Eryilmaz et al., 2014). In the study by Atabani et al., the density of safflower oil biodiesel was measured as 0.8885 g/ml (Atabani et al., 2013). In Demirbaş's study, the density value for safflower biodiesel was reported as 0.866 g/ml (A. Demirbas, 2005). According to Table 5, which presents the density values of sonicated and non-sonicated safflower samples, the density of non-sonicated safflower biodiesel was found to be 0.882 g/ml. The analysis results indicate that the density value obtained for the non-sonicated safflower biodiesel in this study is consistent with the values reported for safflower oil in the literature. However, it is slightly higher than the density value reported by Demirbaş (2005).

Similarly to viscosity and density measurements, API gravity yielded comparable results for the S1 and S2 samples and the S0 and S3 samples. The API gravity was measured as 27.95 for the S1 sample and 27.85 for the S2 sample. For the S0 and S3 samples, the API gravity was determined to be 28.79 and 28.82, respectively.

API gravity is a unit of measurement used to express the density of petroleum and petroleum products. Developed by the American Petroleum Institute (API), it is mainly used to determine the lightness or heaviness of petroleum products. API gravity indicates how much lighter or heavier a petroleum product is than water. It is calculated using Equation 2 based on the specific gravity of the petroleum product at 60 °F (15.6 °C) (Correa Pabón & Souza Filho, 2019; R. O. Dunn, 2011). If the API gravity is equal to 10, the density of the petroleum product is equal to that of water. If the API gravity is below 10, the product is heavier than water; if it is above 10, it is lighter than water. Petroleum products are classified by weight according to their API gravity, as shown in Table 6 (Correa Pabón & Souza Filho, 2019). According to this classification, all the safflower biodiesel samples obtained are in the medium petroleum product category, with the S3 product being the lightest. API gravity is an essential factor affecting the commercial value of petroleum and its products. Generally, petroleum with a higher API gravity is considered more valuable due to its easier processing (Alpaslan et al., 2012). Similarly, light diesel fuels are more accessible to process and are generally less expensive. Refining heavy diesel fuels is more challenging and costly, often resulting in a more expensive product (R. O. Dunn, 2011).

Table 4. Kinematic viscosity values of biodiesel samples

Biodiesel Samples	Viscosity (mm^2/s)
S0	3.4686
S1	3.4583
S2	3.5027
S3	3.5115
Diesel/ASTM D975	1.9 – 4.1
Biodiesel B100/ASTM D6751-D 445	1.9-6.0
EN 14214:2012-EN ISO 3104	3.5-5.0

Table 5. Density and API gravity values of biodiesel samples.

Biodiesel Samples	Density (g/ml)	15°C API/Specific Density	15°C API Gravity
S0	0.8822	0.88200	28.79
S1	0.8868	0.88664	27.95
S2	0.8872	0.88719	27.85
S3	0.8820	0.88183	28.82
Diesel/ASTM D975	0.82-0.88		30-42
B100/ASTM 6751	0.880		30-35

Table 6. Classification of petroleum and its products according to API gravity(Correa Pabón & Souza Filho, 2019)

Product Weight Classification	API Gravity Values
Light	≥31.1
Medium	22.3≤ … <31.1
Heavy	10≤ … <22.3
Very heavy	<10

Table 7. Cloud point changes of ultrasonicated safflower biodiesel samples.

Samples	Cloud Point (°C)	Freezing Point (°C)
S0	-7	-9
S1	-8	-10
S2	-8	-10
S3	-7	-10

Light diesel products with high API gravity have lower density, which results in cleaner combustion and more efficient energy production. This helps improve engine performance and reduces emissions. Lighter diesel fuels leave less residue in engines, and the combustion process is more efficient, leading to longer engine life and reduced maintenance needs. On the other hand, heavy diesel fuels can leave more residue and contribute to engine wear. API gravity affects the flow of diesel fuel in cold weather conditions. Fuels with lower density (high API gravity) generally exhibit better flow at lower temperatures, which improves engine starting in cold weather. Fuels with higher density (low API gravity) tend to solidify in cold conditions, potentially adversely affecting engine performance (Correa Pabón & Souza Filho, 2019; Tissot B.P. & Welte D.H., 2013).

Cloud and Freezing Point

Cloud point measurements are conducted to determine the flow characteristics of fuels under cold conditions. The cloud point is the temperature at which tiny solid crystals become visible as the fuel cools. The pour point is the temperature at which the fuel becomes so thick that it can no longer be pumped after filtering, potentially leading to engine shutdown. It is essential for fuel stations. The pour point is also known as the freezing or solidification point (Atabani et al., 2013). According to ASTM D975 standards for diesel fuels, the pour point is specified between -35°C and -15°C, while the cloud point is defined between -15°C and -5°C. For biodiesel, the pour point is defined by ASTM 6751 according to the ASTM D97 test method as between -15°C and -16°C, and the cloud point is determined by the ASTM D2500 method as between -3°C and -12°C (ASTM, 2008, 2017; Atabani et al., 2013; Candeia et al., 2009; Correa Pabón & Souza Filho, 2019; Edition, 2008; Knothe & Steidley, 2005). Table 7 presents the cloud point and freezing point values for biodiesel samples of castor oil subjected to ultrasonic treatment for 0, 60, 120, and 180 minutes. The table shows that the cloud point was measured as -7°C for S0 and S3 and -8°C for S1 and S2. The freezing point was -9°C for S0, which was not subjected to sonication, and -10°C for the other samples.

In the study by Eryılmaz et al. (2014), the cloud point of safflower biodiesel was found to be -5.3°C, and the freezing point was found to be -16.5°C (Eryilmaz et al., 2014). In the study conducted by Atabania et al., the cloud point for safflower biodiesel was given as -5°C (Atabani et al., 2013). This study determined that the cloud point values obtained from the same vegetable oils were lower than the literature, and the freezing point was between the extreme values . This situation shows that the temperature ranges in which sonicated safflower biodiesel samples can be used is more comprehensive.

Flash Point Determination

The flash point is the lowest temperature at which a liquid's vapor can ignite in the presence of an ignition source. One of the most significant advantages of biodiesel over diesel fuel is its higher flash point and combustion point. This characteristic also enhances biodiesel's ease of storage and transportation (Atabani et al., 2013; Moodley, 2021; Na et al., 2015; Zaimes et al., 2015). According to ASTM D975 standards for diesel, the flash point is accepted as between 52°C and 72°C. At the same time, biodiesel is defined as 93°C according to the ASTM D93 test method and ASTM 6751 standard and 101°C according to the EN ISO 2719 method and EN14214 standard (Atabani et al., 2013; Sakthivel et al., 2018; Shameer et al., 2016; Singh & Singh, 2010). In safflower biodiesel samples, the flash point was measured as 93°C for S0 and 89°C for the sonicated S1, S2, and S3 samples. In a study conducted by Atabania et al., the flash point for safflower was reported as 148°C (Atabani et al., 2013). In a research conducted by Demirbaş, the flash point of safflower was reported as 440 K (167°C) (A. Demirbas, 2005). The values obtained from the literature are significantly higher compared to petroleum diesel. However, in our study, it is evident that the transesterification method applied to safflower oil, followed by ultrasonic treatment, has affected the flash point, significantly lowering it compared to the values reported in the literature and bringing it closer to both petroleum diesel and the flash point values specified in relevant standards.

Sulfur Content

In the elemental analysis of biodiesel samples obtained from safflower oil using an XRF device, the sulfur content was primarily examined, and as expected, values close to zero (0.00077%) were obtained (Table 8). Compared to petrochemical diesel samples containing 0.2579% sulfur (ASTM, 2017; Candeia et al., 2009; İçingür et al., 2006), safflower biodiesel (B100) contains only 0.00068% sulfur. High sulfur content in fuels is undesirable, and in petrochemical diesel, sulfur is one of the most significant environmental pollution concerns. Therefore, the fact that biodiesel samples contain almost no sulfur is crucial for mitigating environmental pollution factors (Candeia et al., 2009; He et al., 2009).

Table 8. Sulfur percentages were tested with an XRF device.

Biodiesel Samples	Sulfur Ratio (%)
Safflower oil (OS)	0.00077
S0	0.00068
Diesel/ASTM D975	0.05
B100/EN 14104	0.02
B100/ASTM D6751-08-D445	Max. 0.05

Conclusion

In this study, the effect of exposing safflower biodiesel, obtained through the base-catalyzed transesterification method we developed, to ultrasonic waves was examined in detail, rather than performing the transesterification process using ultrasonic waves. The results showed that the ultrasonic bath application, carried out without using a mechanical stirrer or an external heating mechanism, caused changes in the chemical structure of the biodiesel, leading to improvements in its physical properties. Additionally, the effects of exposure time to ultrasonic waves on biodiesel were determined, and the critical characteristics of biodiesel were evaluated comparatively with petroleum diesel, considering international standards. Based on the findings, the following conclusions were reached.

- FTIR analyses have shown that the ratio of fatty acid methyl esters changes with sonication time. It has also been found that the chain structure changes the sonication process. Some physical parameters such as density, viscosity, flash point, cloud point, and pour point showed partial changes, and these values were found to be between those of non-sonicated biodiesel and petroleum diesel. As a result, the formation ratio of fatty acid methyl esters and changes in chain structures increased proportionally with the sonication time. It was determined that the physical properties of biodiesel obtained through base-catalyzed transesterification approached values closer to diesel fuel.
- As a result of the analyses, it was observed that fatty acid methyl esters were broken down by ultrasonic sound waves, leading to the formation of volatile components. Consequently, the physical properties were found to be by international standards. Notably, the flash point decreased to 89°C, and the pour point dropped to -10°C. The kinematic viscosity values also ranged from 3.4583 to 3.5115 mm²/s, while the densities ranged between 0.8820 and 0.8872 g/cm³. The samples, with API gravities around 28, are also close to light petroleum products in the medium-weight diesel class. The method used has proven to be as effective as chemical methods in shortening long fatty chains in biodiesel production, and it holds high application potential as a faster method due to the absence of a chemical removal step.
- As a result of short-term sonication of the aspire biodiesel, an increase in the amount of mono alcohol in the FTIR spectrum was observed. In contrast, our previous studies achieved this through long-term sonication in the biodiesel produced from hazelnut oil. This situation demonstrates that sonication applications in plant-based biodiesel production can yield variable results depending on the chemical composition of the plant source used.

Declarations

Fund Statement

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Conflict of Interest

The author declares no conflict of interest.

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