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Effects of ultrasonic mixing time and microwave irradiation time on biodiesel production from crude tung oil

Kittikorn Sasujit[*], *Nigran Homdoung, Surapon Riyana, and Natthawud Dussadee*

School of Renewable Energy, Maejo University, Chiang Mai 50290, Thailand

***Corresponding author**:
Kittikorn Sasujit
k.sasujit@yahoo.com

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ABSTRACT

Biodiesel is an eco-friendly alternative energy source for diesel engines that can be synthesized by the transesterification of vegetable oil or animal fat with alcohol. Tung oil (*Vernicia montana* Lour.) has poor oxidation stability due to the reactivity of the conjugated carbon–carbon double bonds in the constituent α-eleostearic acid. The ultrasonic and microwave-assisted transesterification of tung oil in the presence of an alkaline oxide catalyst was investigated. A laboratory batch process was optimized within parameter ranges for 28 kHz ultrasound mixing (5–20 min), microwave heating (0.5–3 min), methanol-to-oil molar ratio (3–9:1), and KOH catalyst concentration (0.5–1.5% w/w). The maximum transesterification yield was 93.68 ± 1.79%, with a higher heating value of 42.33 MJ/kg, a viscosity of 7.86 cSt, and a fatty acid methyl ester content of 98.83% w/w. The optimal procedure involved 15 minutes of ultrasonic mixing and 2 minutes of microwave heating at a methanol-to-oil molar ratio of 8:1 and a KOH concentration of 1.25% w/w at 60 ± 5°C. The optimized combination of ultrasonic mixing and microwave heating significantly improved the process conditions and product profile, indicating this technique to be an alternative to conventional methods of producing biodiesel.

Keywords: biodiesel; transesterification; tung oil; ultrasonic mixing; microwave irradiation

1. INTRODUCTION

Petroleum products from crude oil and natural gas are critical worldwide energy resources with limited supply. Increased combustion of petroleum-based fuels contributes to environmental issues, including global warming and pollution (Meher et al., 2006; Nakpong & Wootthikanokkhan, 2010). Biodiesel is a renewable and sulfur-free alternative comprising fatty acid methyl esters (FAME). These contain oxygen in the range of 10–11% w/w, which compared to diesel oil, significantly reduces emissions in the exhaust gas. Biodiesel can also be used in modified diesel engines. Biodiesel is produced from agricultural plant oils such as soybean, oil palm, sunflower seed, and peanut, as well as animal fats and waste cooking oils (Harish et al., 2021).

Tung refers to two plant species, *Vernicia montana* (Lour.) and *Vernicia fordii* (Hemsl.), that grow in open woodlands in China and Vietnam (Chen et al., 2010). Crude tung seed oil is extracted in yields of 25.10–26.20% w/w and has a higher heating value of 38.89 MJ/kg (Zhang et al., 2019). Crude tung oil contains approximately 60% fatty acids, including α-eleostearic acid (C18) with three conjugated double bonds (Park et al., 2008; Shang et al., 2010).

Recent studies have aimed to improve biodiesel production and utilization. Biodiesel production yields of more than 96% have been achieved using a methanol-

to-oil molar ratio of 6:1 at 40–60°C with 3% w/w KOH as a catalyst (Chen et al., 2010; Yang et al., 2017). Tung oil biodiesel properties include a high acid value of more than 7.12 mg KOH/g, a density of 903 kg/m^3 at 15°C, and a viscosity of 7.84 mm^2/s at 40°C (Chen et al., 2010).

Conventional biodiesel production processes employ an acid or alkaline catalyst to create a transesterification reaction between free fatty acids or oil and an alcohol, producing esters and glycerol. A catalyst is used to accelerate the process and improve ester yield. Methanol and ethanol are the most acceptable polar short-chain alcohol feedstocks due to their low cost (Sivaprakasam & Saravanan, 2007). Lab-scale biodiesel production with a magnetic stirrer and hotplate has several drawbacks, including low product conversion, excess reactant concentration, slow and inefficient heating, and a large byproduct yield (Maddikeri et al., 2012; Talebian-Kiakalaieh et al., 2013). Typically, biodiesel is produced in a batch reactor using electrical heating coils, requiring significant energy and long reaction times (Tippayawong & Sittisun, 2012). Appropriate heat and mass transfer rates of reagents and products during purification, separation, and extraction are crucial for viable biodiesel yields.

Numerous technologies have been proposed to enhance biodiesel production yields, including reactive distillation (Pradana et al., 2017), membrane reactors (Dubé et al., 2007), microwave irradiation and ultrasonic mixing, or their combination (Fan et al., 2010; Talebian-Kiakalaieh et al., 2013). Ultrasonic mixing or microwave heating in the transesterification process have provided high biodiesel yields (Yang et al., 2017). Microwave irradiation is an alternative heating method that reduces energy usage and enhances efficiency. According to literature reviews, microwave irradiation for batch transesterification provides biodiesel yields of 96.5% from tallow fat oil (Tippayawong & Sittisun, 2012), 92.14% from castor oil (Kord et al., 2016), and 96.5% from soybean oil (Hsiao et al., 2010). Ultrasonic mixing techniques for biodiesel production have been shown to significantly improve yield and homogenization. Biodiesel yields from rapeseed oil using ultrasound are reported to be 87.17% (Almasi et al., 2019) and in the range 91.15–94.03% for oils blended with tung oil (Manh et al., 2012).

However, while microwaves heat quickly, they present mass transfer limitations, and while ultrasonics can produce effective mechanical mixing via cavitation, the technology suffers from limited heat transfer (Martinez-Guerra & Gude, 2015; Milano et al., 2018). These problems can be addressed by combining heat and mass transfer into a single device, making the process of biodiesel production more energy efficient.

This study aimed to investigate the effects of ultrasonic mixing time and microwave irradiation time on biodiesel yields from crude tung oil. This approach greatly improved process efficiency, increasing biodiesel yield, lowering energy consumption, improving biodiesel quality, and accelerating reaction times. A parametric investigation was conducted, examining catalyst concentration, microwave irradiation time, ultrasonic mixing time, and the methanol-to-oil molar ratio.

2. MATERIALS AND METHODS

2.1 Materials

Crude tung oil containing 0.98–1.03% w/w free fatty acids (FFAs), with an acid value of 1.47–3.38 mg KOH/g and a molecular weight of 278.73 g/mol, was obtained from tung seed using a screw press extraction machine. The crude oil FFAs, such as palmitic, linoleic, petroselinic, stearic, α-eleostearic, and gadoleic acids, were analyzed using gas chromatography (GC) standards, as shown in Table 1. The properties of crude tung oil are presented in Table 2. All chemicals used in the study, including 99.99% methanol and 85% potassium hydroxide, were of analytical reagent (AR) grade.

2.2 Experimental procedure

This study produced biodiesel in a laboratory-scale batch reactor using ultrasonic mixing and microwave heating. The reactor consisted of a closed 500 mL plastic beaker, electrically mixed using an ultrasonic cleaner set (28 kHz, 50 W) and heated to 60 ± 5°C using a commercial home microwave device (SAMSUNG R-267, 2.45 GHz, max. 900 W) fitted with a temperature control system. The schematic of biodiesel production is shown in Figure 1. Triglycerides were converted into methyl esters using an alkali catalyst in methanol. The transesterification process comprised two steps: (1) crude tung oil, catalyst, and methanol were loaded into the reactor and mixed with ultrasonic mixing at ambient temperature (30–32°C); and (2) the reaction was assisted by closed microwave irradiation at a temperature set and controlled with a thermocouple inserted into the plastic beaker.

The effects on biodiesel yield of ultrasonic mixing time and microwave heating time were investigated at various methanol-to-oil molar ratios and catalyst concentrations. After separation, waste residues were removed from the ester mixture using distilled water. A schematic diagram of the batch reactor system is shown in Figure 1.

Table 1. Free fatty acid composition of crude tung oil

Free fatty acid	Chemical structure	Composition (% w/w)
Palmitic acid	C17:0	2.79
Linoleic acid	C19:2	9.17
Petroselinic acid	C19:1	8.52
Stearic acid	C19:0	2.47
α-Eleostearic acid	C19:3	74.97
Gadoleic acid	C21:1	0.91

Table 2. Properties of crude tung oil

Parameters	Crude tung oil	Methods
Acid value (mg KOH/g)	2.06	ASTM D664
Viscosity at 40°C (cSt)	87.06	ASTM D445
Density at 15°C	938.70	ASTM D4052
Pour point (°C)	< 29	ASTM D97-04
Flash point (°C)	> 120	ASTM D93-02
Higher heating value (MJ/kg)	38.89	ASTM D240-92

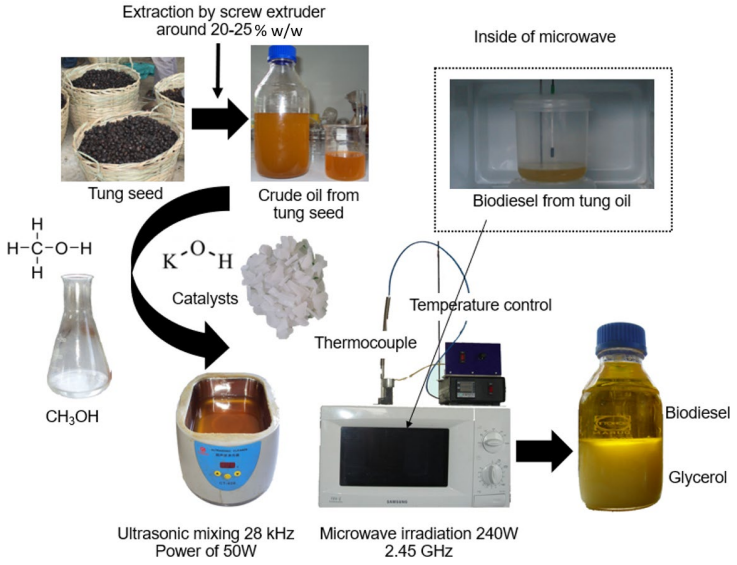

Figure 1. Biodiesel production setup using ultrasonic mixing and microwave heating-assisted transesterification of tung oil

2.3 FAME analysis by GC-MS (FID) and other parameters

The acid value was determined using an American Oil Chemists' Society (AOCS) standard titrimetric method. The kinematic viscosity was measured using a viscosity bath according to the ASTM D5002 standard (Phukan et al., 2013). The oil density was calculated using a specific gravity hydrometer. The flash and fire points were determined using the Cleveland open-cup method according to ASTM D93 (Phukan et al., 2013). The higher heating value was measured with a bomb calorimeter following ASTM D240. The composition of fatty acid methyl esters was analyzed using gas chromatography with an Agilent DB-23 column (60 m x 0.25 mm, 0.15 µm, J&W 122-2361, Agilent, USA). The injector and detector temperatures were set to 250 and 280°C, respectively. A sample volume of 1 µL was injected with a split ratio of 1:50, at a gas flow rate of 40 mL/min for hydrogen, 450 mL/min for air, and 30 mL/min for helium as the carrier gas. Peak areas were integrated using GC software and identified using a standard mixture of 37 FAME compounds. The internal standard for biodiesel was methyl heptadecanoate, and a FAME GLC-90 standard mixture (Merck Ltd., Germany) was used for analysis. The ester content (C), expressed as a mass fraction in percentage, was calculated using Equation 1 (Duvekot, 2020).

$$Ester\ content(C) = \left[\left(\frac{\sum_A - AEI}{AEI}\right) \times \left(\frac{CEI - VEI}{m}\right)\right] \times 100 \quad (1)$$

where C is the total of ester content (% w/w), $\sum_A$ is total FAME peak area (C14:0 to C24:1), AEI is the methyl heptadecanoate peak area, CEI is the methyl heptadecanoate solution concentration (mg/mL), VEI is the methyl heptadecanoate solution volume (mL), and m is the mass of the sample (mg).

The transesterification yield (%), expressed as a mass of biodiesel relative to tung crude oil, was calculated using Equation 2.

$$Transesterification\ yield\ (\%) = \left(\frac{M_{biodiesel}}{M_{crude\ oil\ of\ tung\ seed}}\right) \times 100 \quad (2)$$

where $M_{biodiesel}$ is mass of biodiesel (g), and $M_{crude\ oil\ of\ tung\ seed}$ is the mass of crude tung seed oil (g).

3. RESULTS AND DISCUSSION

3.1 Effect of methanol-to-oil molar ratio on transesterification yield

The effect of methanol-to-oil molar ratio (3:1, 4:1, 5:1, 6:1, 7:1, 8:1, and 9:1) was studied at a KOH concentration of 1.25% w/w. The mixture underwent ultrasonic mixing for 10 min and was then heated by microwave radiation to 60 ± 5°C for 5 min. The transesterification yield increased gradually with the methanol-to-oil ratio

from 3:1 to 5:1, and then decreased at a ratio of 7:1 (Figure 2). The decreased yield at a higher methanol-to-oil molar ratio can be attributed to byproduct formation and the reverse reaction (Hsiao et al., 2010). However, the maximum biodiesel yield was 88.21 ± 1.23% at a methanol-to-oil molar ratio of 8:1.

3.2 Effect of catalyst concentration on transesterification yield

The effect of catalyst concentration (0.5, 1.0, 1.25, and 1.50% w/w) on transesterification yield was studied at a methanol-to-oil ratio of 8:1, with 10 min of ultrasonic mixing followed by 5 min of microwave heating. Increasing the catalyst concentration led to a higher crude tung oil conversion rate in the transesterification reaction and a maximum average transesterification yield of 88.21 ± 1.23% at a catalyst concentration of 1.25% w/w. However, catalyst concentrations higher than 1.25% w/w resulted in lower conversion rates to biodiesel (Figure 3), suggesting the possibility of saponification (Hsiao et al., 2010).

3.3 Effect of ultrasonic mixing and microwave heating times on transesterification yield

The effect of ultrasonic mixing time (5, 10, 15, and 20 min) on transesterification yield was studied at a methanol-to-oil ratio of 8:1, with 1.25% w/w catalyst, and with microwave heating for 1 min. The transesterification yield increased with ultrasonic mixing time, reaching a maximum yield of 91.55 ± 1.13% with 15 min of mixing. The high yield observed under ultrasonic mixing conditions may be attributable to the rapid mass transfer between the methanol and free fatty acid, and the formation of microemulsions caused by the ultrasonic cavitation phenomena, as shown in Figure 4 (Teixeira et al., 2009).

The effect of microwave heating time (0.5–3 min) on biodiesel yield was studied at a methanol-to-oil ratio of 8:1, with 1.25% w/w catalyst, and ultrasonic mixing for 15 min. The transesterification yield increased slightly as microwave heating duration increased from 0.5 to 2 min, but decreased slightly thereafter to reach equilibrium. Increased heating time positively affects the conversion and biodiesel yield. However, the yield decreases close to the boiling point of methanol due to saponification (Tippayawong & Sittisun, 2012). The maximum transesterification yield was 93.68 ± 1.79% at a microwave irradiation time of 2 min, as shown in Figure 5.

3.4 Properties of biodiesel from different seed oils

Table 3 presents the properties of biodiesel produced from jatropha, palm, and tung crude seed oils. The parameters for tung oil biodiesel were determined according to Thailand's 2019 standards for the characteristics and quality of methyl esters from fatty acids. The concentration of methyl esters is higher in Thailand standard (Boonmee et al., 2010; El-Araby et al., 2018; Kongkasawan & Capareda, 2012) than those reported by Manh et al. (2012) and Chen et al. (2010). The results of the chemical analysis showed an acid value of 0.43 mg KOH/g, a density of 885 kg/m^3, a viscosity of 7.86 cSt, a flash point of 196°C, and a FAME content of 98.83% w/w.

3.5 Biodiesel process comparison

The conventional method, microwave heating, ultrasonic mixing, and a combination of microwave heating and ultrasonic mixing were compared for biodiesel production from tung oil. The transesterification yields for each method were 91.19, 93.11, 88.29, and 93.68%, respectively. The maximum average transesterification yield of 93.68 ± 1.79% was achieved by combining ultrasonic mixing and microwave heating, indicating that the mixing and heating time of the transesterification process is optimal, as shown in Figure 6.

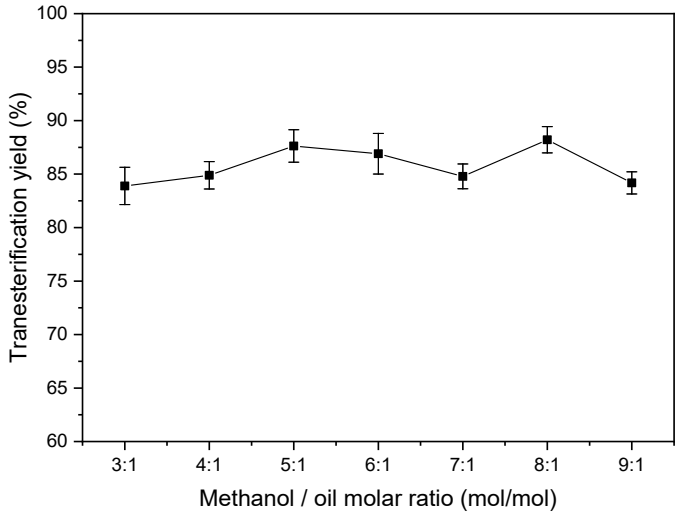

Figure 2. Effect of methanol-to-oil molar ratio on transesterification yield: reaction conditions were 10 min ultrasonic mixing, 1.25% w/w catalyst, and 5 min closed microwave heating to 60 ± 5°C

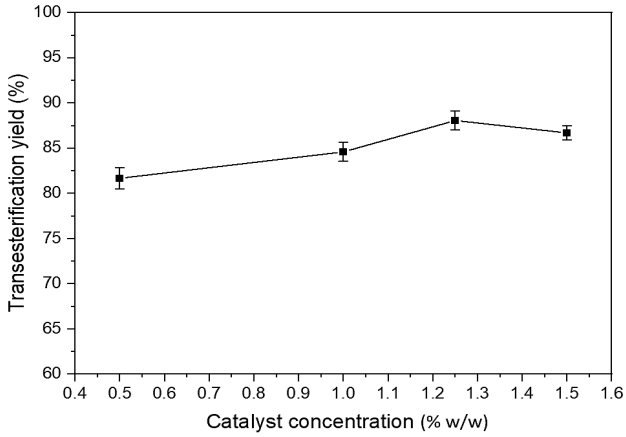

Figure 3. Effect of catalyst concentration on yield: Reaction conditions were methanol-to-oil 8:1, 10 min ultrasonic mixing, and 5 min microwave heating to 60 ± 5°C

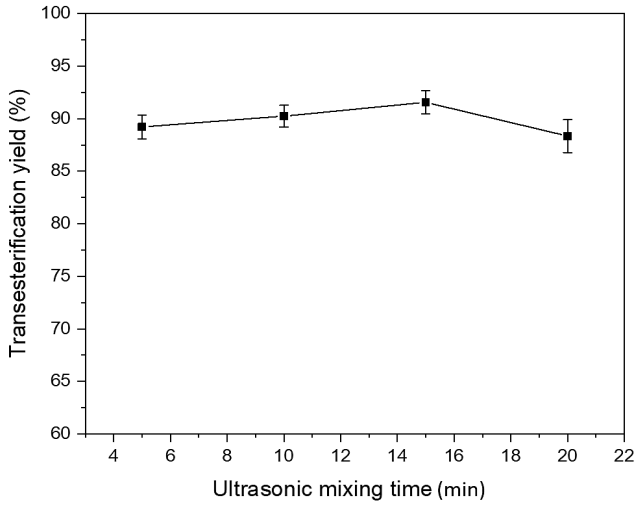

Figure 4. Effect of ultrasonic mixing time on transesterification yield: Reaction conditions were methanol-to-oil 8:1, 1.25% w/w catalyst, and microwave heating for 1 min

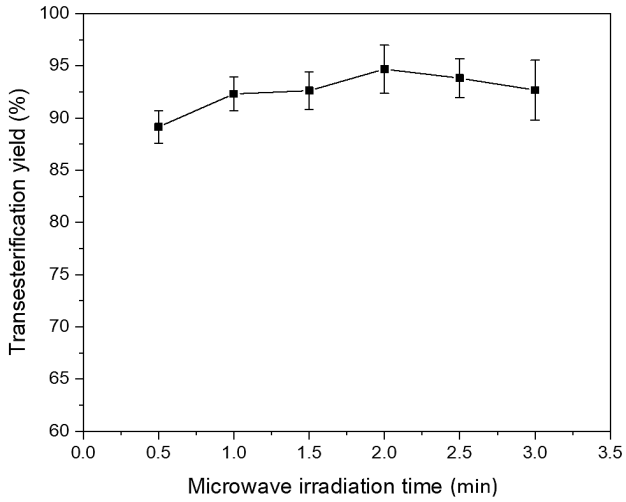

Figure 5. Effect of microwave irradiation time on transesterification yield: Reaction conditions were methanol-to-oil 8:1, 1.25% w/w catalyst, and ultrasonic mixing for 15 min

Table 3. Biodiesel properties of tung oil, jatropha oil, and palm oil

Parameter	Jatropha oil	Palm oil	Tung oil			Thailand standard
			This work	(Manh et al., 2012)	(Chen et al., 2010)	
Acid value (mg KOH/g)	0.120	0.129	0.43	0.11	0.124	< 0.50
Density at 15°C (kg/m³)	881	875	885	905	903	860–900
Viscosity at 40°C (cSt)	4.33	4.43	7.86	9.18	7.84	3.5–5.0
Flash point (°C)	177	172	196	-	-	> 120
Pour point (°C)	4.90	4.00	−15.00	−16.00	−11.00	< 10
FAME (% w/w)	99.40	99.80	98.83	91.15	94.90	> 96.50
Higher heating value (MJ/kg)	39.5	40.58	42.33	-	-	-

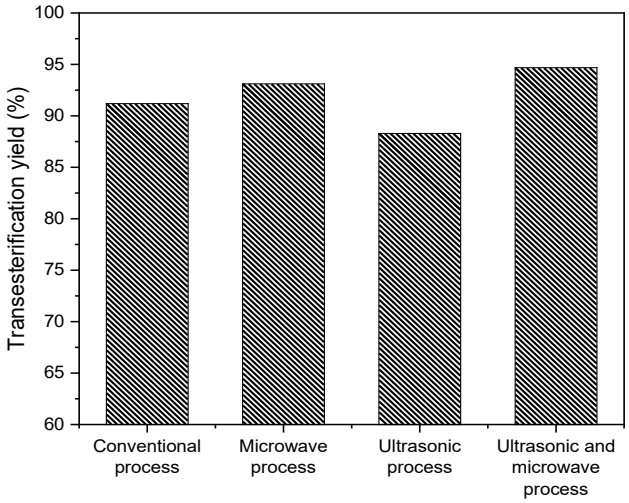

Figure 6. Transesterification yield of tung oil under different process conditions

Table 4 summarizes the optimum transesterification yields of tung oil and other seed oils. Conventional biodiesel production from jatropha oil yielded between 90 and 98% using NaOH as a catalyst (Berchmans & Hirata, 2008; Chitra et al., 2005), and 99% with a 4:1 methanol-to-oil molar ratio and 0.6% w/w KOH catalyst (Tiwari et al., 2007). The transesterification procedure was completed within 90–120 min using NaOH and KOH as catalysts. Soybean and maize seed oils gave transesterification yields of 98% in 1–5 min using microwave-assisted procedures and KOH (Leadbeater & Stencel, 2006) or NaOH (Ozturk et al., 2010).

Table 4. Comparison of optimal conditions and resulting biodiesel yields

Crude oil	Mixing & heating	Catalyst and concentration (% w/w)	Methanol-to-oil molar ratio	Reaction time (min)	Biodiesel yields (%)	References
Jatropha oil	HS	NaOH 1.0	6:1	90	98.00	(Talebian-Kiakalaieh et al., 2013)
	HS	NaOH 1.4	7:1	120	90.00	(Teixeira et al., 2009)
	HS	KOH 0.6	4:1	24	99.00	(Tippayawong & Sittisun, 2012)
Soybean oil	M & MW	KOH 5.0	10:1	1	98.00	(Tiwari et al., 2007)
Tung oil	M & MW	KOH 1.25	8:1	120	91.20	This work
	UL & HS	KOH 1.25	8:1	45	93.10	This work
	HS & MW	KOH 1.25	8:1	5	88.30	This work
	UL & MW	KOH 1.25	8:1	17	93.68	This work

Note: HS hotplate stirrer, M magnetic stirrer, MW microwave heating, UL ultrasonic mixing

4. CONCLUSION

The transesterification procedure to produce tung oil methyl esters was enhanced using ultrasonic mixing and microwave heating. This new process results in shorter production times and higher yields compared to conventional methods. The results demonstrate that 15 min of ultrasonic mixing, 2 min of microwave heating, an 8:1 methanol-to-oil molar ratio, and 1.25% w/w KOH catalyst are the optimal conditions for biodiesel production in the laboratory setup employed. Under these conditions, the transesterification yield was found to be 93.68 ± 1.79%. This study shows that biodiesel production under these new conditions can be practical and efficient, accelerating the chemical transformation and decreasing the energy requirement to produce

biodiesel. Thus, ultrasonic mixing and microwave heating are effective methods for rapidly converting tung oil to its methyl ester.

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