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## Energy-saving and rapid transesterification of jatropha oil using a microwave heating system with ionic liquid catalyst

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## ABSTRACT

The use of inedible oils to produce biodiesel, which lowers engine emissions, has been studied extensively. Jatropha oil is an ideal feedstock for biodiesel production because of its high oil content. This study focuses on applications to improve jatropha oil biodiesel yields with a microwave heating system with the ionic liquid catalyst. Although there were multiple research projects that have focused on the use of microwave heating systems to improve methyl ester yields, the topic to raise yields from microwave heating systems and IL catalyst have yet to be thoroughly studied. Current experimental results indicate that the best methyl ester yield is 98.5% with an  $I_1N_{0.75}$  catalyst (1 wt% IL + 0.75 wt% NaOH), at a methanol-to-oil molar ratio of 9, microwave temperature of 70 °C, and 6 min of reaction time. A microwave heating system offers a rapid route to biodiesel production that is also more energy-efficient and cost-effective.

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### 1. Introduction

Increasingly stringent regulations have been introduced worldwide to tackle the issue of pollution with diesel engines being identified as a major source. Technological evolution for use of biodiesel in diesel engines helps reduce engine emissions. Meanwhile, edible oils such as rapeseed oil, sunflower oil, palm oil, and soybean oil are popular choices as raw materials for biodiesel production [1]. An expansion for sources of biodiesel for production is essential for long-term sustenance of widespread biodiesel usage. The oil content in jatropha seed is 25–40 wt%, and 45–60 wt% in the kernel. Jatropha trees [2], a potential biodiesel crop, are easily cultivated in China and India [3–5]. Kumar et al. [6] improved jatropha oil biodiesel yields and reduced reaction time with the use of Na/SiO<sub>2</sub> as a catalyst and ultrasonication. They found the best yield of jatropha oil biodiesel was 98.5% with a methanol to oil molar ratio of 9, catalyst concentration of 3 wt% of oil, and 15 min reaction time. Deng et al. [7] found the best yield of jatropha oil biodiesel was 95.2% when the methanol to oil molar ratio is 4 to 1, 1.0 wt% catalyst concentration, with reaction temperature set at 318 K, ultrasonic power of 210 W, and 1.5 h of reaction time. Taufiq-Yap et al. [8] found the jatropha biodiesel yield of more than 80% was achieved from the use of CaMgO and CaZnO

catalysts at 338 K for the transesterification process, 4 wt% catalyst concentration, methanol to oil molar ratio of 15, and a reaction time of 6 h. In another study conducted by the same authors [9], it was reported that various calcium based mixed oxide catalysts with different Ca/Mg atomic ratio, reaction time of 3 h, methanol-to-oil molar ratio of 25 to 1, 3 wt% catalyst, and reaction temperature of 120 °C resulted in a jatropha biodiesel yield between 75% and 90%. Endalew et al. [10] found that a CaO:Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> weight ratio of 3:1 was tested and a yield of jatropha oil biodiesel was 93.4%. While using LieCaO as a catalyst, the product yield was further increased to 96%. Olutoye and Hameed [11] used aluminum modified heterogeneous basic oxide (Mg–Zn) as catalyst in biodiesel production from crude jatropha oil. The optimal reaction conditions were 3.32 wt% catalyst, 11:1 molar ratio of methanol to oil, 182 °C temperature, and 6 h reaction time with 94% yield of jatropha biodiesel.

Ionic liquids (ILs), defined as liquid-state salts at temperatures lower than 100 °C, are widely used industrially. The prospects of ILs as green solvents in various chemical processes have received more focus in recent research. ILs have been successfully used as environmental solvents/catalysts in various chemical reactions because of lower corrosion effects, separable, recyclable, direct, and continuous processing, reduced wastewater formation. ILs, due to their significant environmental benefits, process improvements, and their dual roles as solvents and catalysts, have attracted significant attention for their use in biofuel production [12–28]. Much light is being shed on ILs in other research with the prospect to develop

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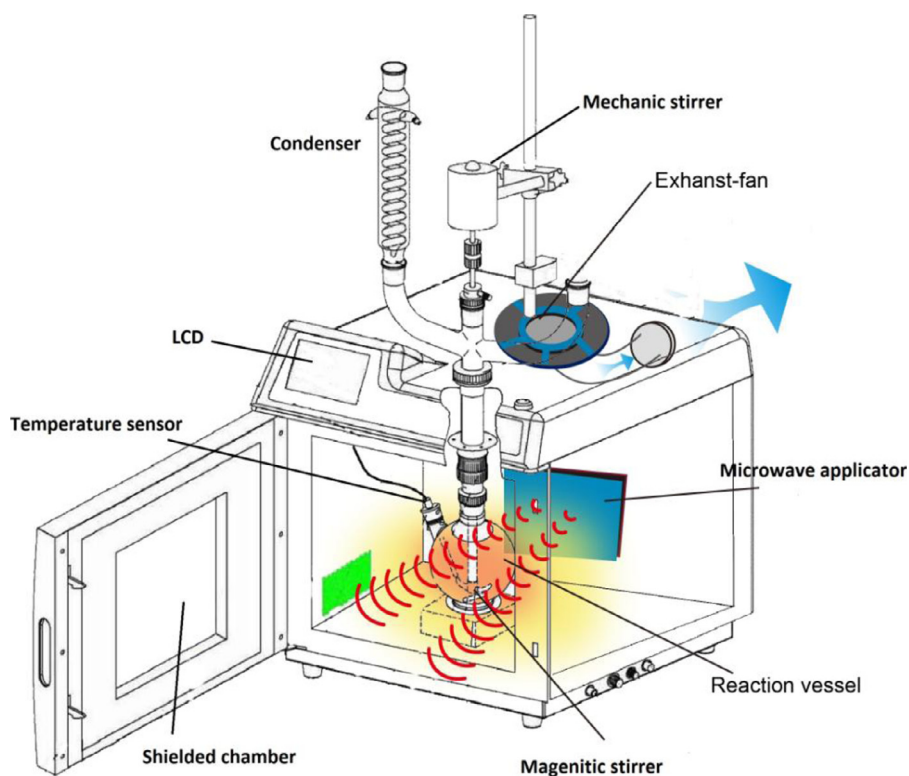


Fig. 1. The experimental setup.

an environmental friendly, while economically feasible industrial process for biodiesel production. It has been shown that ILs catalysis reduces the number of reactions and purification steps in the preparation and separation of biodiesel. This results for more competitive processing while yielding highly purified esters [29–31]. The performance of recycled ILs is impressive. ILs can maintain their catalytic activity based on the yield and selectivity of product after proper separation and purification steps [19,21]. Li et al. [19] investigated transesterifications of jatropha oil with ILs. They concluded that the best yield of jatropha oil biodiesel was 92.0% at a temperature of 70 °C, molar ratio of oil/alcohol/[BSPy]CF<sub>3</sub>SO<sub>3</sub> 1:10:0.12, and reaction time of 5 h. Guo et al. [20] reported a maximum biodiesel yield of 99.7% at 120 °C when FeCl<sub>3</sub> was added to [BMIm][CH<sub>3</sub>SO<sub>3</sub>] (1-butyl-3-methylimidazolium tosylate). Multiple studies have shown the advantages of microwave-assisted chemical reactions with faster reaction rates, better product yields, and higher purities of biodiesels [32–35]. It was also shown that the microwave heating method is more energy-efficient than conventional heating in transesterification [36,37]. It is notable that the input energy used for conventional heating can be two to twenty-three times higher than for microwave heating [36,37]. These results suggest that appropriate use of microwaves reduces the overall energy consumptions in biodiesel production.

Despite previous research on microwave heating systems and ILs for the yields of biodiesel, the use of microwave heating systems and IL 4-allyl-4-methylmorpholin-4-ium bromine ([MorMeA][Br]) as a catalyst for the yield of biodiesel production has yet to be addressed. Morpholinium-based ionic liquids are widely used due to their competitive properties and low cost. This study investigates the yields of biodiesel from jatropha oil with a microwave heating system and I<sub>x</sub>N<sub>y</sub> catalysts (defined as *x* wt% IL + *y* wt% NaOH). Additionally, the effects of catalyst type and amount, reaction time, methanol to oil molar ratio, and reaction temperature are investigated and discussed.

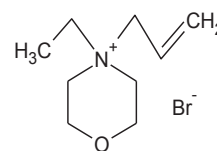


Fig. 2. The chemical structure of 4-allyl-4-methylmorpholin-4-ium bromine ionic liquid ([MorMeA][Br]) catalyst.

## 2. Experimental section

### 2.1. Transesterification procedures

The jatropha oil sample used in this study was supplied by CPC in Taiwan. The acidic value of the sample was 2.8 mg KOH/g. Methanol and sodium hydroxide (NaOH) employed in the experiments were those of high-performance liquid chromatography (HPLC) grade. The experimental setup is shown in Fig. 1. A microwave synthesis reactor (PreeKem APEX, PreeKem Scientific Instruments Co. Ltd., China), equipped with a mechanical stirrer and a condenser (LC-10, Hi-point Co. Ltd, Taiwan) was used for microwave reactions. For the energy analysis, the reactors are the same. The stirrer operated at 600 rpm with a magnetic nucleus. The temperature detector of microwave heating system is resistance temperature detector (RTD) used to measure temperature by correlating the resistance of the RTD element, platinum (Pt), with temperature. The Pt has a predictable change in resistance as the temperature changes. It is this predictable change used to determine temperature. For conventional heating, the same vessel with samples was put in a water-bath. The ionic liquid used in this study is shown in Fig. 2. A solution of 360 mmol allyl bromide was added to a solution of 360 mmol *N*-methylmorpholine. The mixture was refluxed for 24 h at 50 °C in argon atmosphere. Upon cooling of the mixture, the solid that was formed was filtered. The crude

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