



Research Article

Co-Solvent Free Electrochemical Synthesis of Biodiesel Using Graphite Electrode and Waste Concrete Heterogeneous Catalyst: Optimization of Biodiesel Yield

Wiyogo Prio Wicaksono^{1,2,*}, Sekar Asmara Jati^{1,2}, Ika Yanti¹, Prastika Krisma Jiwanti³

¹Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Islam Indonesia, Jl. Kaliurang KM 14.5, Yogyakarta, 55584, Indonesia.

²Research Center for Materials and Electrochemistry, Faculty of Mathematics and Natural Sciences, Universitas Islam Indonesia, Jl. Kaliurang KM 14.5, Yogyakarta, 55584, Indonesia.

³Nanotechnology Engineering, Faculty of Advanced Technology and Multidiscipline, Universitas Airlangga, Kampus C Universitas Airlangga, Surabaya, 60115, Indonesia.

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Abstract

This study optimized a co-solvent free electrochemical method for biodiesel synthesis using graphite electrode and waste concrete heterogeneous catalyst. Various parameters were evaluated, including: applied voltage (9.6, 14.4, 19.2 V), catalyst particle size uniformity (unfiltered and filtered with 150 mesh), and reaction time (15, 30, 120, 240 min). The results obtained 100% FAME content and 78.51% of biodiesel yield that were achieved at 14.4 V within 30 min using filtered catalyst and cooking oil feedstock. However, a slight decline was observed with the use of waste cooking oil. This optimized method offers a reliable and simple condition for mass biodiesel production.

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Keywords: Electrochemical Synthesis; Biodiesel; Waste Concrete; Heterogeneous Catalyst; Waste Cooking Oil

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1. Introduction

Biodiesel remains a significant eco-friendly and alternative renewable energy source [1], particularly in developing countries [2]. In meeting the huge national demand, a larger preference for the feedstock, including palm oil and the derivatives, e.g. waste cooking oil (WCO), are also considered. Previous studies

widely regarded WCO utilization as a very suitable raw material and energy resource, as well as a possible solution to waste problems [3–5].

Among the available biodiesel production methods, electrochemical synthesis (electrosynthesis / electrolysis) offers great benefits, termed ready conducted at ambient conditions, suitable for oil feedstock with high water content, less operational energy, no co-solvent required, and high possibility to enhance optimal performance by heterogeneous catalyst addition [3,6], including carbon@chitosan compo-

* Corresponding Author.

Email: wiyogo.prio@uii.ac.id (W.P. Wicaksono);

Telp: +62-74- 895920 ext 3012, Fax: +62-74- 896439

site [7], chitosan gel [8], CaO from eggshell [6], zeolite/chitosan composite [9]. Meanwhile, graphite electrodes were applied in this research due to cheap and excellent conductivity properties of carbon-based materials [10].

Our previous study reported the utilization of waste concrete could enhance the performance of biodiesel electrosynthesis by producing biodiesel with FAME content up to 98.37% using 0.5 wt.% of the catalyst at 9.6 V for 2 h [3]. This high result recognized the electrosynthesis as a very promising approach with the need for further development, and also with potentials to meet the minimum European and Indonesian standard (96.5%), without additional purification [11,12]. Furthermore, the study also reported that the amount of catalyst influenced the FAME content produced [3]. Unfortunately, to obtain optimized condition, the applied voltage, catalyst particle size uniformity, and reaction time were not comprehensively evaluated.

In this study, the effect of applied voltage, the uniformity of catalyst particle size, as well as the reaction time were investigated. The electric current flow in the electrolytic cell was also investigated for all experiments. All of the synthesized biodiesels were analyzed using gas chromatography-mass spectrometry (GC-MS) to calculate the FAME content as well as the biodiesel yield. Furthermore, the evaluation of physicochemical characteristic *i.e.*, water and free fatty acid (FFA) content in the cooking oil feedstock were also evaluated.

2. Materials and Methods

2.1 Materials

The vegetable (cooking) oil containing palm oil, was purchased from the mini market in Yogyakarta, while WCO was obtained from home industries around Universitas Islam Indonesia, Indonesia. NaOH (p.a.) and methanol (pa) were purchased from Merck, while NaCl (>99%) was purchased from PT UniChemCandi Indonesia. Waste concrete was supplied from building restoration around Universitas Islam Indonesia, Indonesia, without any pretreatment.

2.2 Oil Feedstock Characterization

In evaluating cooking oil and WCO qualities, water and FFA contents were analyzed using gravimetric and titration methods, respectively, based on the National Standardization of Indonesia (SNI) [3]. A brief water content

analysis was conducted, where a sample holder was dried for 30 min in the oven (130±1) °C and then cooled down (W_0). Subsequently, 2 g of oil was added to a vessel (W_1), followed by further drying at similar temperature, and also left to be cooled down (W_2). This procedure was repeated until W_2 achieved equilibrium. Furthermore, the water content (%) was calculated using the Eq. (1):

$$\text{water content \%} = \frac{W_1 - W_2}{W_1 - W_0} \times 100\% \quad (1)$$

with W_0 is the weight of blank sample holder (g), W_1 and W_2 are the weight of sample holder contains oil sample before and after drying (g), respectively.

Meanwhile, for FFA analysis, 10 g of oil was diluted in 50 mL of warm ethanol, followed by the addition of 5 drops of PP indicator. A 0.1M NaOH served as a titrant in the acid-base titration, where a color change to pink was observed at equilibrium. The FFA (as palmitic acid) was calculated using the Eq. (2):

$$\text{free fatty acid (FFA)\%} = \frac{25.6 \times V \times N}{W} \quad (2)$$

where, the value of the constant is 25.6, V and N represent the volume (mL) and normality (N) of NaOH (mL), while W refers to the oil sample weight (g).

2.3 Optimization of the Condition of Biodiesel Electrosynthesis

The biodiesel electrosynthesis was conducted using a constant DC voltage (Sanfix SP-305E) in line with previous report [3,13]. Briefly, a single chamber electrolytic cell equipped with two graphite electrodes (3.52 cm² of active surface area) were separated by 12 mm, with a 30 mL solution containing cooking oil and methanol (1:24 molar ratio), deionized water (0.1 wt% of entire mixture weight), and NaCl (0.56 wt% of oil weight) as the electrolyte. A 0.5 wt% of waste concrete (based on oil weight) was selected as the optimum heterogeneous catalyst weight, as reported in previous result [3]. The effects of applied voltage (9.6, 14.4, and 19.2 V), catalyst particle size uniformity (unfiltered and filtered by 150 mesh), and reaction time (15, 30, 120, and 240 min) were evaluated. Furthermore, the electric current flow in electrolytic cell was also monitored. The overall synthesis was conducted at ambient condition under constant stirring, without any co-solvent. Subsequently, the optimized condition was employed with WCO as feedstock.

2.4 Biodiesel Characterization

The biodiesel product (top) was immediately separated from the final mixture. In addition, GC-MS (Shimadzu QP 2010 equipped with Rtx-5MS column) and a flame ionization detector (FID) were employed to evaluate the chemical composition, with helium as the carrier gas. A 0.1 mL of oil sample was injected, as the split inlet temperature and flow were retained at 250 °C and 30.8 mL min⁻¹, respectively. The

column heat was operated at 80 °C, but subsequently climbed to 300 °C. This state was maintained for 5 min and the yield (%) was calculated using the following equation (Eq. (3)) [2,14]:

$$\text{yield (\% of biodiesel)} = \frac{\text{mass of biodiesel produced (g)}}{\text{mass of initial oil (g)}} \times \text{FAME content (\%)} \quad (3)$$

The FAME content (%) was determined by the sum of FAME % area from the product chromatogram.

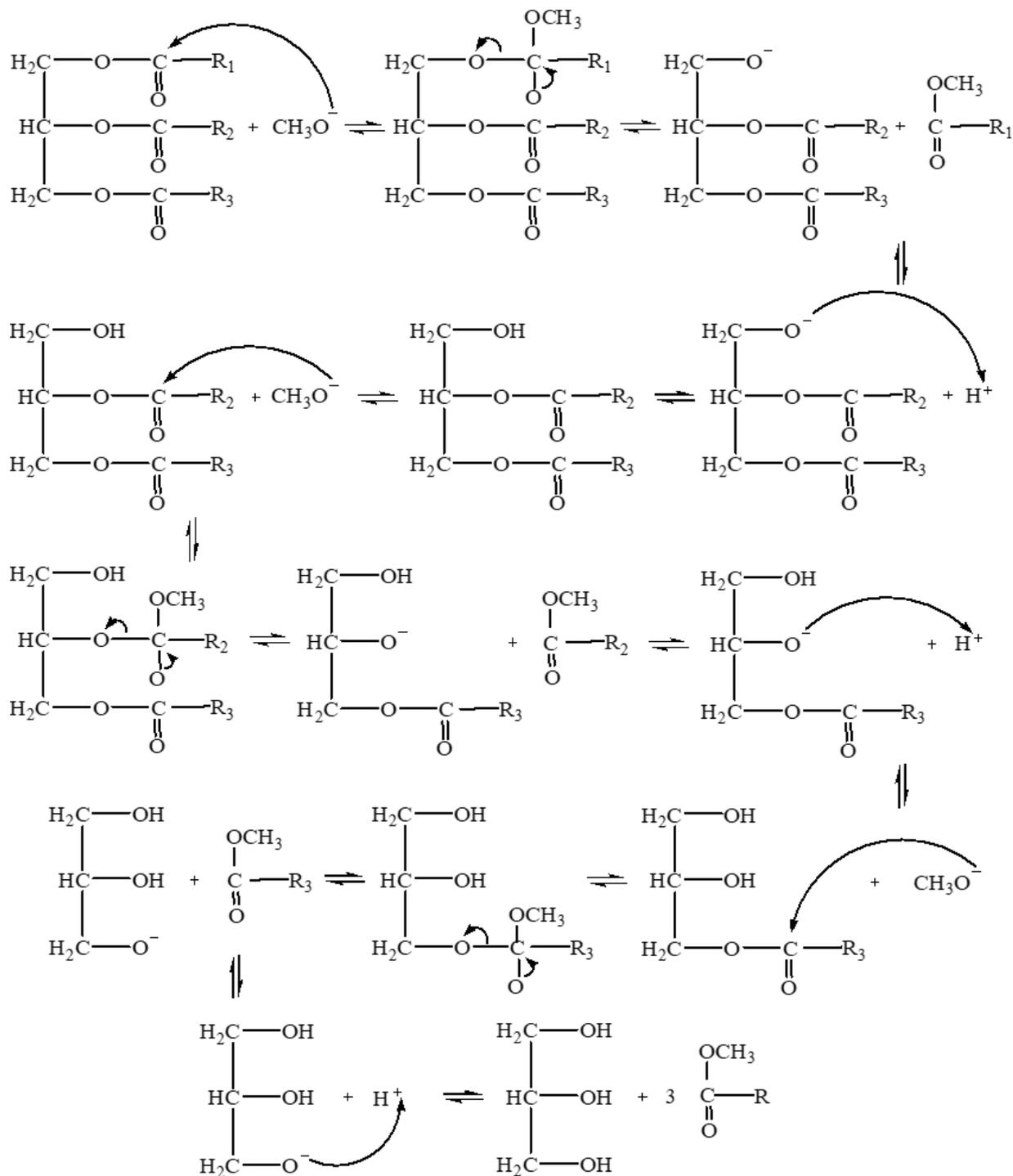
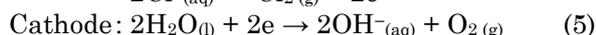
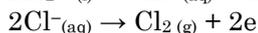
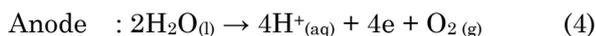


Figure 1. The transesterification reaction in the biodiesel synthesis.

3. Results and Discussion

3.1 Electrosynthesis of Biodiesel

The biodiesel synthesis was conducted in a single electrolytic cell containing oil, methanol, NaCl, water, and concrete waste rich-metal oxide ($-M-O-$), using graphite electrodes [3], due to low cost, inertness, good electrical conductivity, physical-chemical stability and durability [15]. The electrode provides comparable properties with other precious metals, such as platinum (Pt) and stainless steel (SS) that have been utilized in biodiesel synthesis [13,16]. The redox reaction on the electrodes are as follow (Eqs. (4,5)):



The water electrolysis generated H^+ and OH^- at anode and cathode that could act as acid and base catalyst for the esterification and transesterification reaction in the biodiesel synthesis, respectively. Meanwhile, the addition of concrete waste that could lead the protonation of water to produce surface OH^- that will en-

hance the catalytic activity (Eq. (6)) [17-19].



The OH^- from both water electrolysis and water protonation by waste concrete concurrently extracted the H^+ from methanol to produce methoxide (CH_3O^-) (Eq. (7)). This species was subsequently employed in the transesterification reaction by the following mechanism (Figure 1).



3.2 The Effect of Applied Voltage

Figure 2 shows the effects of applied voltage on FAME (%) and biodiesel yield (%), where the FAME content increased with the increasing of applied voltage and reached up to 100% at 19.2 V. The result was linear with the previous report [6]. The increase in applied voltage will improve the electric current as well as the conductivity in the electrolytic system which led to the more extensive OH^- generation in the cathode [13]. This trend was also projected for biodiesel yield (%) [6], but unfortunately, a decline to 68.77% was observed at high applied voltage of 19.2 V. It is due to the high voltage that decreased the electrode stability, led to the decrease of the conductivity as well as the electric current as shown in Figure 2C. Thus, the applied voltage of 14.4 V was employed for all further experiments.

3.3 The Effect of the Uniformity of Catalyst Particle Size

The effects of catalyst particle size uniformity of waste concrete on FAME content (%) and biodiesel yield (%) were evaluated. Figure 3A depicts high FAME up to 100% with filtered

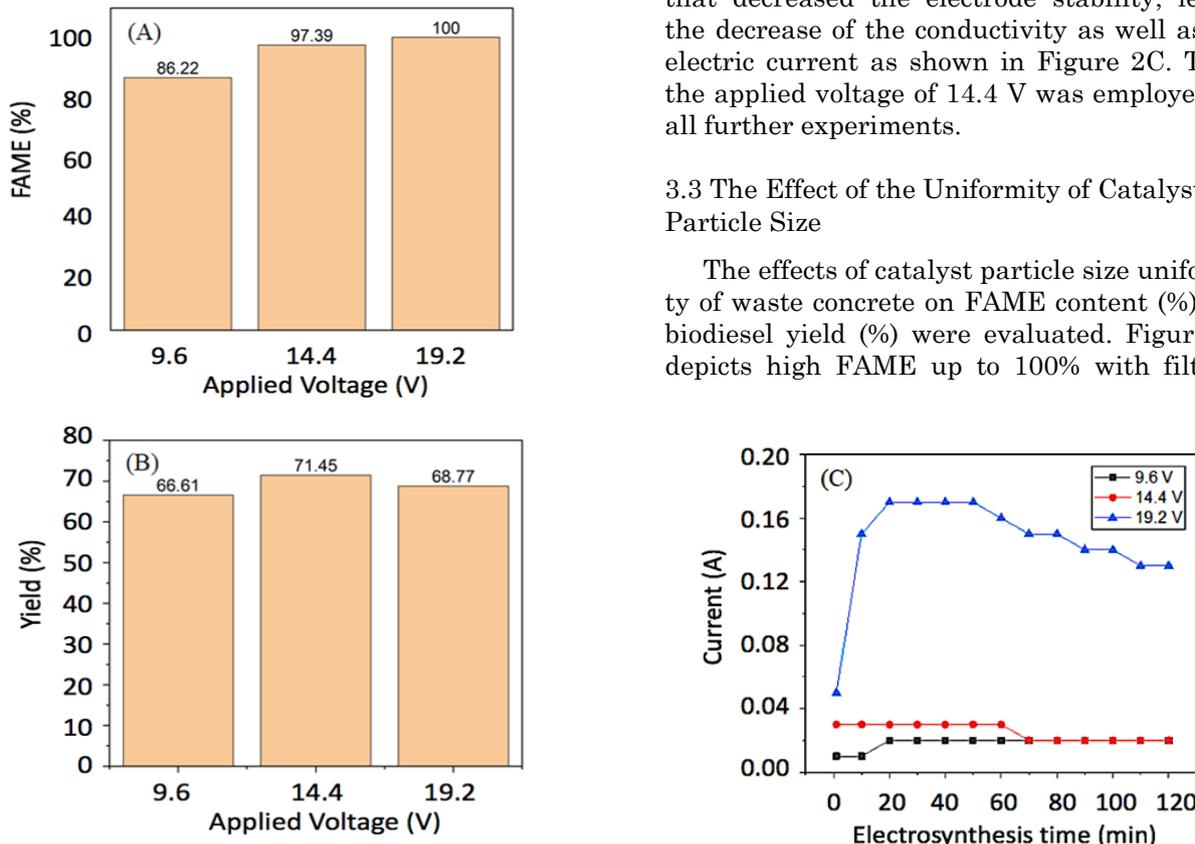


Figure 2. The FAME content (%) (A) and yield (%) (B) of biodiesel produced at various applied voltages (V); and the electric current recorded (C) for 2 h reaction.

catalyst, but with unfiltered catalyst, a minor decline to 97.39% was observed. The uniform size of the catalyst promotes the reactants to effectively adsorb on the catalyst surface then react with other reactants to generate products. The catalyst with uniform size provides a higher effectiveness factor in the catalytic process compared to the non-uniform size [19]. Furthermore, the non-uniform catalyst provided

various active sites and was not able to completely control selectivity, leading to various products [20]. As a consequence, these variations did not significantly influence the biodiesel yield and electric current (Figure 3B and C). It is due to the only slight difference in uniformity of the filtered and the unfiltered catalyst particle. Therefore, the filtering treatment using 150 mesh was employed for further analysis.

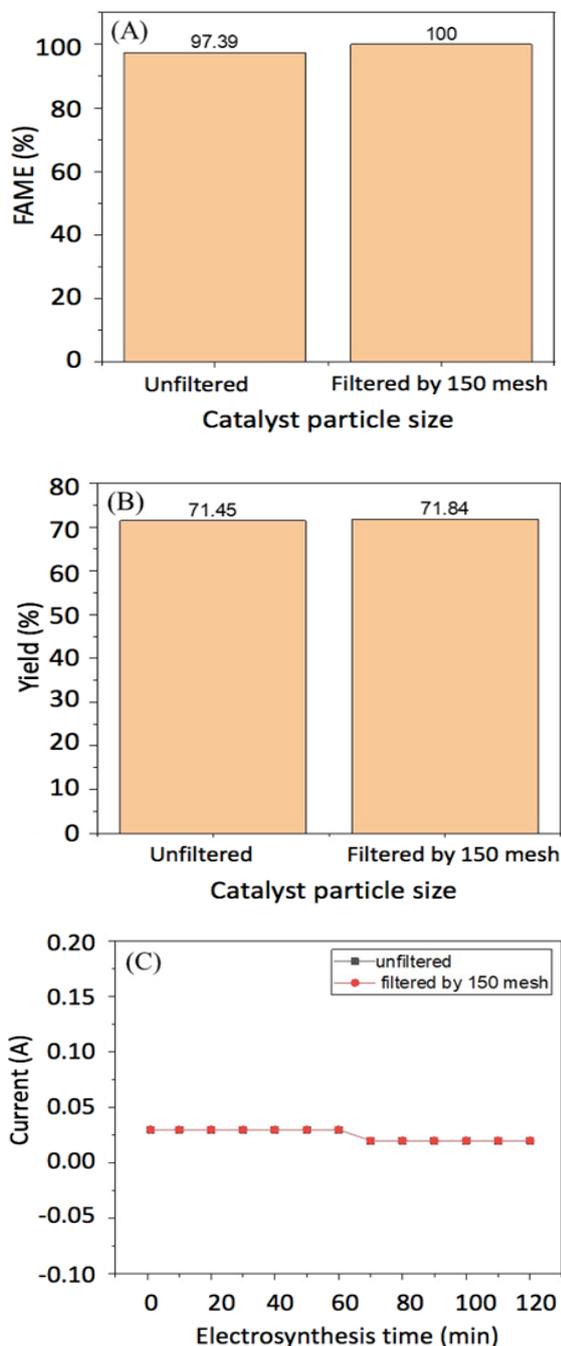


Figure 3. The FAME content (%) (A) and yield (%) (B) of biodiesel produced using unfiltered and filtered catalyst; and the electric current recorded (C) for 2 h reaction.

3.4 The Effect of Reaction Time

The reaction time plays a significant role in the optimization process. Generally, the longer

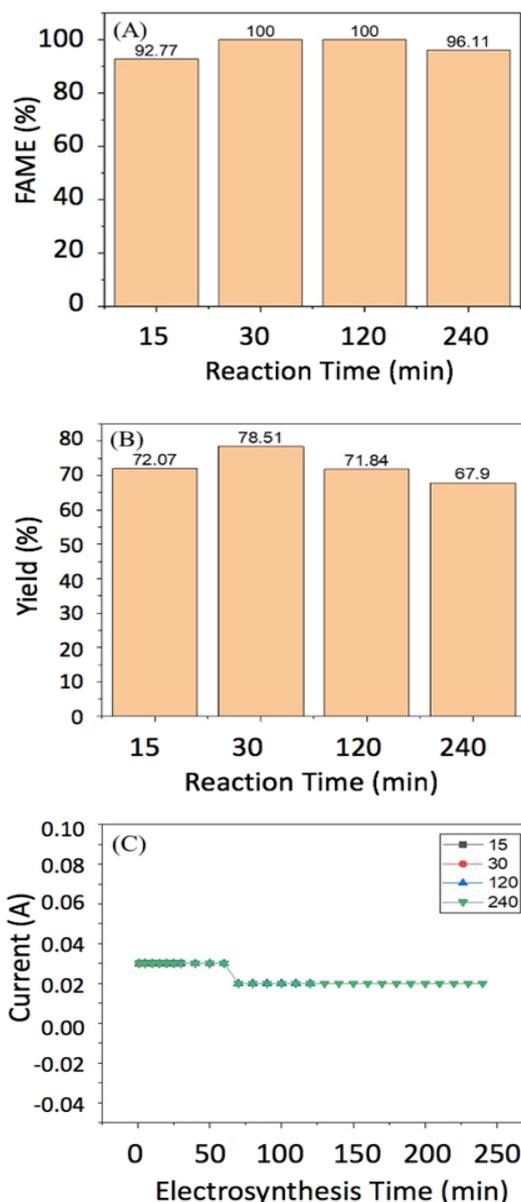


Figure 4. The FAME content (%) (A) and yield (%) (B) of biodiesel produced at various reaction time; and the electric current recorded (C).

reaction time provides the reactant to collide with each other resulting in more products. Also, FAME is expected to increase as the reaction time continues to reach the equilibrium [21]. Figure 4A shows the reaction time of FAME content at 15, 30, 120, and 240 min. The FAME content increased with the increase in reaction time and reached up to 100% at 30 and 120 min, then slightly decreased to 96.11% at 240 min. The decrease may be ascribed to the further conversion of FAME to unwanted prod-

ucts, due to a long electrosynthesis process. Figure 4B shows the highest yield (%) of biodiesel that was achieved at 30 min, followed by a decline. It may due to the decrease of FAME content as well as the backreaction in the transesterification [22]. Additionally, the decrease of electric current in the electrolytic cell after 30 min reaction may be contributed to the decrease in yield, as shown in Figure 4C. Therefore, reaction time of 30 min reaction time was selected for the further investigation.

3.5 Evaluation of the Optimized Biodiesel Electrosynthesis Method Using WCO

The evaluation of optimized biodiesel electrosynthesis method was conducted with WCO feedstock. An applied voltage of 14.4 V, filtered waste concrete catalyst, and 30 min reaction time were employed, based on the optimum previous condition. Figure 5A depicts FAME content up to 100% was achieved using both oil feedstocks, indicating that WCO has potential as feedstock for biodiesel synthesis. This result revealed that higher water content and FFA of WCO (Table 1) did not interfere the biodiesel electrosynthesis process, where commonly those high contents lead to a saponification reaction in the conventional biodiesel synthesis method [13]. A slight difference in FFA content between the cooking oil and the WCO indicates that the properties of WCO only slightly changed during the frying. This result was in agreement with the previous reports [23]. The FFA content generated by the hydrolysis reaction during the frying process primarily depends on the temperature, time, and mode of the frying process [23,24]. A higher frying temperature, longer frying time, as well as number of frying, resulted in a higher generation of FFA. Furthermore, the obtained yield (Figure 5B) was not significantly different for both feedstocks, whereas the slight decrease of the yield may be due to the more viscous character of WCO, leading the slower stirring process and the lower electric current (Figure 5C) [25]. Figure 6 and Table 2 represent the chromatograms of the synthesized biodiesel and chemical composition, respectively. Table 3 shows

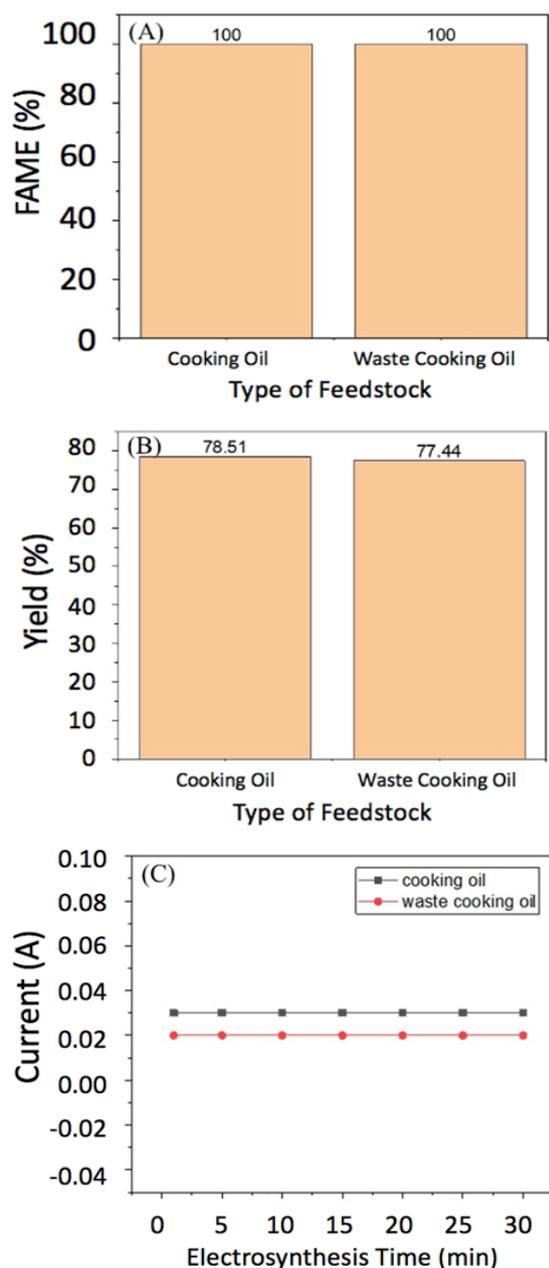


Figure 5. The FAME content (%) (A) and yield (%) (B) of biodiesel produced using waste cooking oil feedstock; and the electric current recorded (C).

Table 1. Physicochemical characteristic of biodiesel feed stocks.

Parameter	Cooking oil	Waste cooking oil
Water content (%)	0.238	0.263
FFA (%)	0.495	0.539

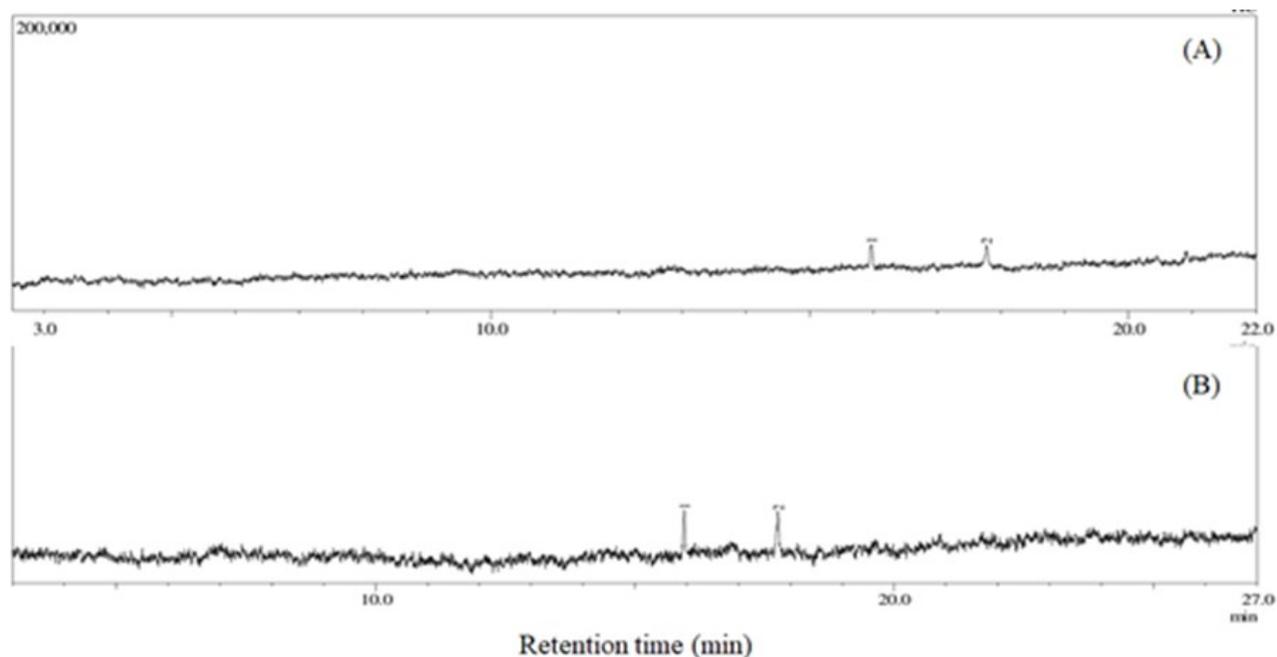


Figure 6. The chromatograms of the synthesized biodiesel with feedstock of: (A) cooking oil and (B) waste cooking oil.

Table 2. Chemical composition identification of the synthesized biodiesel using GC-MS.

Biodiesel feedstock	Peak	Retention time (min)	Chemical formula	Chemical compound	Composition (wt%)
Cooking oil	1	15.969	C ₁₇ H ₃₄ O ₂	Pentadecanoic acid, 14-methyl-, methyl ester	66.68
	2	17.771	C ₂₀ H ₃₈ O ₂	Cyclopropanepentanoic acid, 2-undecyl-, methyl ester, trans-	33.32
	FAME (%)				100
Waste cooking oil	1	15.954	C ₁₇ H ₃₄ O ₂	Pentadecanoic acid, 14-methyl-, methyl ester	61.20
	2	17.759	C ₂₀ H ₃₈ O ₂	Cyclopropanepentanoic acid, 2-undecyl-, methyl ester, trans-	38.80
	FAME (%)				100

Table 3. Comparison of several biodiesel production methods.

Method/ Electrode materials	Applied voltage (V)	Reaction time (min)	Additional catalyst	Uniformity catalyst treatment	FAME (%)	Yield (%)	Ref.
Electrosynthesis/ Graphite	18.2	60	CaO from egg shell	No	98.99	N/A	[6]
Microwave	-	60	SiO ₂ from bamboo leaves ash	No	N/A	57.50	[2]
Conventional	-	15	NaOH	No	99.93	61.79	[26]
Ultrasound irradiation	-	15	KOH	No	N/A	62.77	[28]
Conventional	-	240	CaO-clay	No	N/A	73	[27]
Electrosynthesis/ Graphite	14.4	30	Waste concrete	Yes	100	78.51	This work

the comparison of this method with other biodiesel production methods. This approach offered faster reaction time than microwave [2] and other biodiesel electrosynthesis method [6]. In addition, lower applied voltage was also observed [6]. In contrast to conventional [26,27] and ultrasound irradiation biodiesel synthesis [28], this research method showed the capacity to produce higher FAME content and biodiesel yield.

4. Conclusions

Optimizing conditions, including applied voltage, catalyst particle size uniformity, and reaction time of electrochemical biodiesel synthesis, have been successfully conducted in a single chamber electrolytic cell, using graphite electrodes and waste concrete catalyst. It was found that FAME content and the biodiesel yield increased with the increase of the applied voltage. Consequently, a decline was observed after achieving optimum voltage, due to electrode damage. The filtration phase offers the catalyst particle size uniformity known to influence the improvement of both FAME and biodiesel yield. The increase in reaction time up to 30 min could improve the FAME content and the yield, then declined for longer reaction. Moreover, the maximum FAME content and the biodiesel yield were achieved at applied voltage of 14.4 V in 30 min reaction time, using both cooking and waste cooking oils. It is suggested that high energy consumption and long reaction are not necessary to synthesize biodiesel by filtered waste concrete catalytic enhanced electrochemical method in ambient condition. Finally, an optimized process condition was achieved and is believed to be cost-effective for mass biodiesel production.

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