

# Removal of impurities from waste oil using eggshell and its active carbon

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## ABSTRACT

Waste oil is an inexpensive source for biodiesel production, but the high amount of impurities present (free fatty acids, oxidation products) impedes its practical application. The aim of this work is to assess the purification efficiency of the adsorption process with eggshell and its active carbon. Carbonization of eggshell was done at 200 °C for 4 hours. For activation of the resultant carbon, sulfuric acid was used, and finally, thermal activation (600 °C, 1 hour) was performed. The quality parameters of waste oil (free fatty acid, peroxide value, color indices, viscosity, and density) were determined before and after the adsorption process. The results showed an improvement pattern in the measured parameters after the adsorption. Reduction in peroxide and acid value were 71.7% and 51.72%, respectively. Also, produced biodiesel from treated waste oil has better quality indices including acidity, viscosity and specific gravity. Biodiesel yield also increased up to 40 percent.

**Keywords:** Waste oil, Eggshell, Active carbon, Biodiesel

## Introduction

Continuous global use of fossil fuels and their environmental impacts are major recent concerns of human society.<sup>1</sup> These problems are imperative issue for researchers to look for new sources of energy. Among the renewable energy resources, biodiesel is noteworthy due to its suitable properties<sup>2</sup> that include non-toxicity, biodegradability, good combustion efficiency and environment friendliness.<sup>3</sup> Biodiesel production is performed through transesterification process.<sup>4</sup> A major obstacle for biodiesel production in commercial scale is the high price of vegetable oil.<sup>5</sup> Optimization of the use of low-price waste oil for biodiesel production is thus a popular research aim.<sup>1, 6</sup> Low price, huge amount<sup>7,8</sup> and availability are the main advantages of waste oil for biodiesel production but impurities in waste oil are major problem for this approach. Free fatty acids<sup>9</sup> and peroxides<sup>10</sup> are the main impurities in waste oil that resist the formation of methyl ester.<sup>11</sup> Adsorption is an approach for elimination of

some impurities.<sup>12</sup> Different studies showed effectiveness of active carbon for adsorption of impurities.<sup>13,14</sup> Agricultural wastes, industrial by-products and wastes and natural substances have been studied as adsorbents.<sup>15-17</sup> Puspa Asri et al. investigated purification of waste oil by a combination of active carbon and earth clay and reported that by adsorption, biodiesel yield of waste oils rise up to 90%.<sup>18</sup> Eggshell is a by-product of baking industry and confectionaries. Chicken's eggshell typically consists of three parts (ceramic materials present in the outer cuticle, a spongy (calcareous) layer and inner lamellar layer). The outer two layers contain numerous circular pores. Eggshell has been estimated to contain between 7000-17000 pores.<sup>19</sup> Several studies have been focused on determination the suitability of the eggshell and membrane as adsorbents for iron,<sup>20</sup> cadmium chromium,<sup>21</sup> Cu (II),<sup>22</sup> reactive dyes,<sup>23</sup> Pb (II),<sup>24</sup> Cyanide,<sup>25</sup> H<sub>2</sub>S,<sup>26</sup> Ni, CO.<sup>27</sup> Its adsorption ability has so far been attributed to several mechanisms such as, chemical adsorption, complexation, coordination, ion exchange, chelation, physical adsorption and microprecipitation.<sup>28</sup> Samsudin reported the effectiveness of eggshell active carbon in removal of surfactants from wastewater.<sup>18</sup> To date, no research has been performed on

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adsorption ability of eggshell and its active carbon for removal of impurities from waste oils. The present work is hence focused on application of eggshell and its active carbon for purification of waste oil which is used as feedstock for biodiesel production.

### Materials and Methods

In this work, waste oil was obtained from a local restaurant. Eggshell was collected from the confectionary. All chemicals (sulfuric acid, potassium hydroxide, potassium iodide, sodium thiosulfate, ethanol, chloroform, hexane, methanol) were purchased from Merck. The viscosity of oil was determined by Brookfield viscometer modelLDV-II-Pro. Biodiesel viscosity measurement was done by Canon-Fenske viscometer. Gas Chromatography-Mass Spectrometry of waste oil was performed by GC-MS in an Agilent Technologies 7890A GC system. HunterLab Colorflex was used for measuring color.

### Preparation of active carbon

The raw materials (eggshell and its membrane) have been collected from local confectionary. After washing and drying, 250 g of weighed and crushed (blender BOSCH, MKM 6003 model) material was placed in furnace for 4 hours at 200 °C. The reason of this step is to remove non-carbonaceous compounds. Active carbon was prepared by pouring 60 g of the carbonized eggshell in a beaker. 50 mL of H<sub>2</sub>SO<sub>4</sub> was added and left on stirrer for 24 hours. Thereafter, neutralization was done by washing with deionized water. Thermal activation carried out in muffle furnace (600 °C, 1 hour).<sup>2</sup> According to several studies,<sup>29, 30</sup> milling of egg shell may cause lower specific surface area. Non-milled eggshell was applied as adsorbent.

### Active carbon characterization

Specific surface was determined according to methylene blue absorption method.<sup>31</sup> To determine true density, 1–5 g of adsorbent material was poured in a fixed volume container and weighed, then the container was filled with

water to half of its total volume, boiled for one hour, and cooled. Water was added to the container to reach the initial volume. The volume of added water is equal to true density.<sup>15</sup>

Apparent density was measured by pouring active carbon on a screen and placing it into molten paraffin. After removing, it was weighed and placed into distilled water. Apparent density of active carbon was calculated by the volume of displaced water.<sup>32</sup>

### Determination of optimum ratio of mixed egg shell and active Carbon

For finding the best ratio of active carbon and eggshell, different ratios of these adsorbents were tested and the best ratio was selected based on its capability of the maximum acid value lowering in waste oil (data not shown). Accordingly, a ratio of 5:1 eggshell to active carbon was selected and used in further experiments.

### Characteristics of waste cooking oil

#### Determination physical properties of oil

Standard methods used for analysis of oil quality include the American Oil Chemists' Society (AOCS) official method Cd 8–53 for peroxide value<sup>33</sup> and AOCS official method Cd 3d–63 for acid value<sup>34</sup> determination. Oil density and viscosity were measured according to ASTM D5002–99<sup>35</sup> and ASTM D2983,<sup>36</sup> respectively.

Analysis of oil color before and after the adsorption process was done using a colorimeter (Hunter Lab, Model Color Quest XT, United State), after standardization with Hunter Lab Color standards. In this color system, L\* represents the lightness, and a\* and b\* are the color coordinates, where +a represents the red coordinate, –a is the green coordinate, +b is the yellow coordinate, and –b is the blue coordinate.<sup>37</sup>

### Gas Chromatography-Mass Spectrometry

For precise assessment of oil quality, Gas

Chromatography-Mass Spectrometry was performed. Waste oil was analyzed by GC-MS in an Agilent Technologies 7890A GC system along with a 5975CVLMSD mass spectrometer and a series 7683B injector. Used column characteristics include: Agilent (9091) 413 °C HP-5 column, 320 µm diameter, 30 m long and film thickness equal to 0.25 µm. Helium was used as carrier gas with a flow rate of 3.35 mL/min is used. The injector temperature was 250 °C.<sup>38</sup>

### **Transesterification reaction and biodiesel production**

Transesterification is the reaction for biodiesel production from oils.<sup>1</sup> Several procedures were introduced for transesterification.<sup>39</sup> Selection of transesterification method is dependent on the amount of impurity in oil. In the case of low acid content of oils (<3%), one step acid transesterification is a suitable approach for biodiesel production. In this study, acidity is lower critical amount (3% acidity),<sup>40</sup> so, one-step acid transesterification was chosen.

After filtering the waste oil, transesterification was carried out in a 250 mL flask with reflux condenser and continuous stirring. Reaction condition was: methanol to waste oil ratio:1:6, three percent sulfuric acid, 96 h reaction time and 60 °C reaction temperature.<sup>41</sup>

The percentage of biodiesel yield was calculated following the European regulated procedure EN14103 as shown below:<sup>42</sup>

$$\% \text{ Biodiesel yield} = \frac{\text{Amount of biodiesel (g)}}{\text{Amount of waste oil (g)}} \times 100 \quad (1)$$

### **Determination biodiesel properties**

The biodiesels obtained from treated and untreated waste oil were further analyzed in accordance to ASTM test methods. ASTM D4052,<sup>43</sup> ASTM D445,<sup>44</sup> and ASTM D664<sup>45</sup> for density, kinematic viscosity at 40 °C, and acidity measurement, respectively.

### **Statistical analysis**

Data were reported as the mean ± standard deviation (SD) for triplicate determinations. Non-parametric Mann–Whitney tests were employed to identify differences in means. Statistical analysis was performed using SPSS for Windows (version release 10.0.5, SPSS, Inc., Chicago, IL). Statistical significance was declared at  $P < 0.05$ .

## **Results and Discussion**

### **Identification of active carbon properties**

Active carbon prepared from egg shell has true density and apparent density equal to 1.5 and 0.45 g/cm<sup>3</sup>, respectively. Specific surface of resulted active carbon was measured as 150 m<sup>2</sup>/g.

### **Characterization of oil quality**

According to the non-parametric Mann–Whitney test, in all target variables of waste oil (viscosity, peroxide, acidity and density), P-value is lower than 0.05 and there was significant difference between untreated and treated waste oil (Table 1).

Table 1. Descriptive statistics of waste oil properties (A1: waste oil, A2: waste oil after adsorption process)

Target Variables	Groups	Descriptive statistic				
		Sample size (n)	Mean ( $\bar{x}_i$ )	Standard deviation ( $S_i$ )	Mann-Whitney U	P-value
Viscosity (mm <sup>2</sup> /s) at 40 °C	A1	3	33.185	0.166	0.000	0.049
	A2	3	32.430	0.336		
Peroxide (meq/kg)	A1	3	19.67	0.577	0.000	0.043
	A2	3	5.67	0.577		
Acidity (mg KOH/g)	A1	3	0.2907	0.009	0.000	0.046
	A2	3	0.1403	0.0006		
Density (kg/m <sup>3</sup> )	A1	3	0.9123	0.0006	0.000	0.043
	A2	3	0.9107	0.0006		

### ***Effect of adsorbents on peroxide value of waste oil***

Peroxide value is an indicator of oxidation rate in oils. In waste oils, high peroxide value is a result of the application of too high temperatures.<sup>46</sup> Due to the adverse effect of peroxide value on transesterification reaction, peroxide value of waste oil was measured and reported as meq/kg in this study. According to the experimental results, peroxide value of untreated and treated waste oils were 19.67 and 5.67 meq/kg, respectively. These results confirmed that selected adsorbents were able to remove peroxide from waste oil. Peroxide elimination by biosorbents was reported by other researchers. Wannahari et al. showed that

application of 7.5 g bagasse adsorbent could reduce peroxide value of waste oil to 21%.<sup>10</sup> Khan et al. indicated activated charcoal could reduce peroxide value of waste oil to standard limit and cause 32.8% reduction of peroxide value.<sup>47</sup>

### ***Effect of adsorbents on color of waste oil***

Color is one measure of oil quality. Waste oil usually has dark color because heating causes formation of polymeric compounds that influence oil color.<sup>46</sup> Color measurement in Hunter lab system is showed with three indices that include lightness (L\*), redness (a\*) and yellowness (b\*). Color changes before and after adsorption process is depicted in Fig. 1.

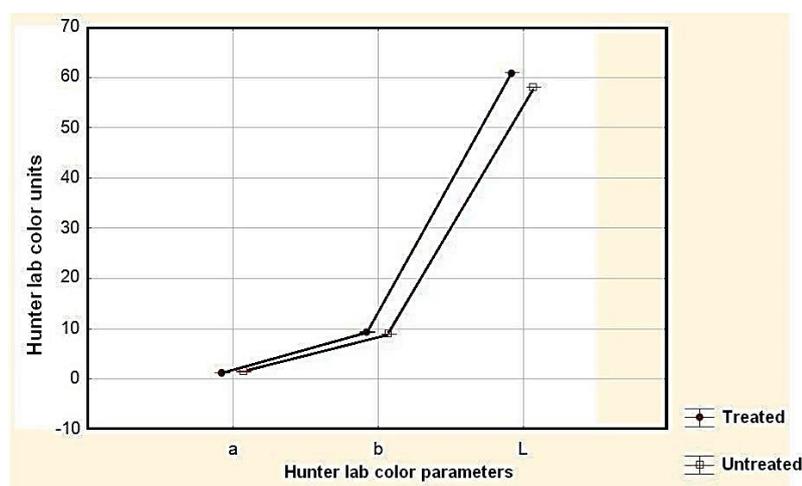


Fig. 1. Effect of adsorbent on color of waste oil. Lightness or L\*, redness-greenness or a\* and yellowness-blueness or b\*. Bars represent the standard deviation (n=5).

All color indices (a, b, L) are influenced by adsorption process. Redness (a\*) of waste oil was 1.5 and reaches to 1.3 after adsorption process, yellowness (b\*) of waste oil was 9 and 9.3 before and after adsorption, respectively. Lightness (L\*) was 58 prior to adsorption and reaches 61 after treatment. The bleaching ability of active carbon was reported by others. Nandini et al. confirmed that activated carbon possesses bleaching capacity as shown on sunflower waste oil. According to this study, increasing the mass of adsorbent and contact time could enhance bleaching capacity.<sup>48</sup> Similarly, groundnut oil has been easily decolorized by activated carbon from coconut shells.<sup>28</sup>

### ***Effect of adsorbents on acid number of waste oil***

Results approved the effect on adsorbents on acid value of waste oil (Table 1). Acidity of

untreated waste oil was 0.29 and in treated one, it reduces to 0.14 mg KOH/g. free fatty acids interfere with transesterification reaction and have negative effects on biodiesel production.<sup>11</sup> Ability of active carbon to remove free fatty acids is stated by researchers. Khan et al. indicated effectiveness of charcoal active carbon on lowering acidity of fried oil.<sup>47</sup>

### ***GC-MASS analysis of waste oil***

To confirm the effect of adsorbent on waste oil quality, GC-Mass spectrometry of waste oil (before and after adsorption treatment) was performed. Figure 2 shows the relative abundance of the peak area related to components which are found in untreated and treated waste oil. The extracted data are depicted in Table 2. According to the peaks identified

in untreated waste oil, 2,4 decadienal, palmitic acid, linoleic acid and oleic acid are the main component in waste oil. In the case of treated waste oil, the main component being recognized include, 2,4 decadienal and oleic acid. As shown in Fig. 2, the peaks of palmitic and linoleic acid completely disappeared in treated waste oil. 2,4 decadienal is also reduced (15.3%). These results indicated considerable effect of adsorption on reducing the impurities in waste oil. During oxidation of lipids, hydroperoxides form and break down; and doing so, produce secondary oxidation products such as aldehydes and ketones.<sup>46, 49</sup> In this work, 2,4 decadienal detected in waste oil in both conditions (before and after adsorption process) but a reduction magnitude of 15.3% observed in treated waste

oil. Existence of 2,4 decadienal in waste oil is reported by other studies.<sup>50</sup>

Table 2. Identification chemical composition of untreated waste oil

Peak No.	RT (min)	m/z	Possible structure
1	19.866	81.10	2,4-Decadienal
2	35.394	57.10	Hexadecanoic acid
3	38.844	43.10	9,12-Octadecenoic acid
4	46.597	55.10	9-Octadecadienoic acid

Table 3. Identification chemical composition of treated waste oil

Peak No.	RT (min)	m/z	Possible structure
1	19.851	81.10	2,4-Decadienal
2	19.898	81.10	Trans- trans, 2,4-Decadienal
3	46.267	69.10	9-Octadecadienoic acid

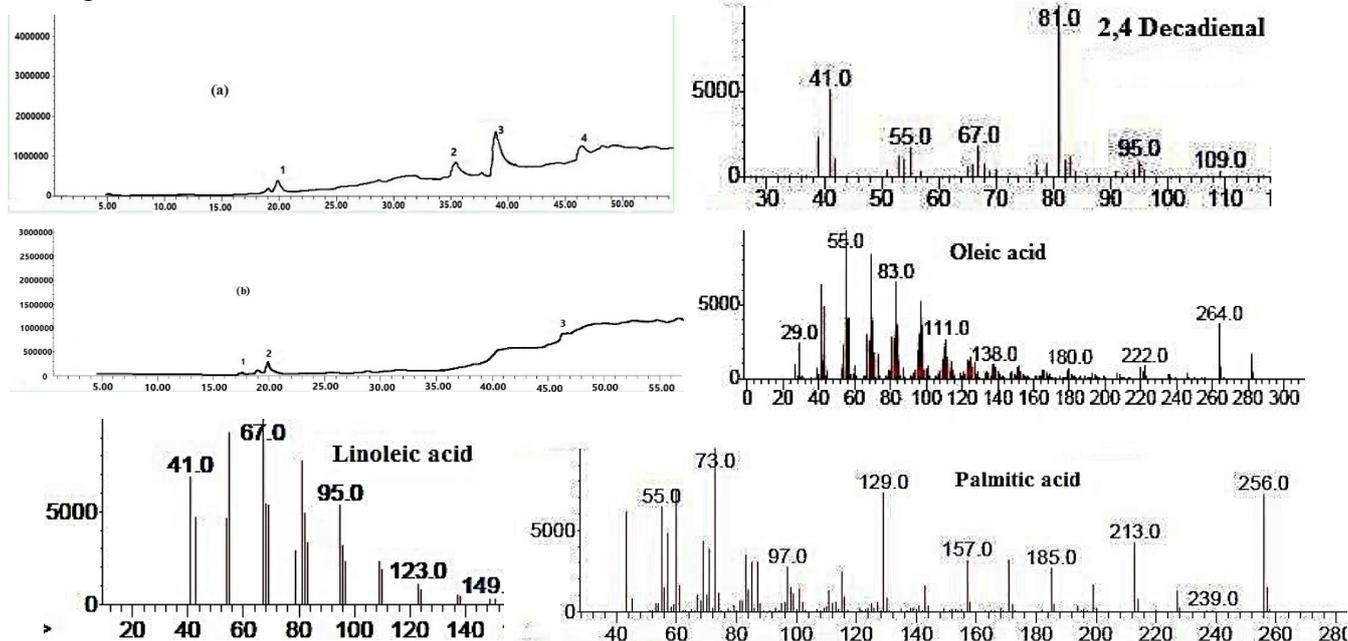


Fig. 2. Chromatogram of untreated (a) and treated oil (b).

**Biodiesel Properties**

According to non-parametric Mann–Whitney test, in all target variables of biodiesel (biodiesel yield, acidity, specific gravity, and

viscosity), P-value < 0.05 and there was a significant difference between biodiesel quality of untreated and treated waste oil (Table 4).

Table 4. Descriptive statistic of adsorption process on biodiesel quality. (B1: Biodiesel from waste oil; B2: biodiesel from treated waste oil)

Target Variables	Groups	Descriptive statistic				Mann-Whitney U	P-value
		Sample size (n)	Mean ( $\bar{X}_i$ )	Standard deviation ( $S_i$ )			
Biodiesel yield (%)	B1	3	43.67	0.577	0.000	0.046	
	B2	3	70.00	1.000			
Acidity (mg KOH/g)	B1	3	0.199	0.002	0.000	0.049	
	B2	3	0.09167	0.008021			
Specific gravity at 15.5 °C	B1	3	0.88633	0.001528	0.000	0.049	
	B2	3	0.88	0.001000			
Viscosity (mm <sup>2</sup> /s) at 40 °C	B1	3	5.2333	0.05774	0.000	0.043	
	B2	3	4.2333	0.05774			

### ***Biodiesel yield (%)***

Biodiesel yield (%) is an indicator of effectiveness of transesterification reaction and is calculated according to Formula 1. The biodiesel yield of treated and untreated waste oil were 70% and 43.67% (SD=1 and 0.577, respectively). It appears that the biodiesel yield of untreated waste oil is influenced by the amount of impurities it contains. Waste oil with high free fatty acid produced more amount of soap during transesterification that alleviate the final yield. The effect of purification of waste oils prior to biodiesel production were outlined by other reports. Puspa Asri et al. showed that biodiesel yield of waste oil reaches to 90% after pretreatment of waste oil with combination of active carbon and bleaching earth.<sup>18</sup>

### ***Biodiesel viscosity***

High viscosity is an obstacle for direct use of oils in diesel engines. Biodiesel production from oils could reduce viscosity to suitable level.<sup>51</sup> The viscosities of obtained biodiesels were determined by using a Cannon-Fenske viscometer. Triplicate samples were used for the experiments. It was observed that the average viscosity of both biodiesels was in the recommended range of ASTM (1.9–6.0 mm<sup>2</sup>/s at 40 °C).<sup>52</sup>

### ***Specific Gravity of biodiesel***

There was a little difference between specific gravity of two tested biodiesels (Table 4). Specific gravity of both produced biodiesels, were fitted with standard limits (0.88).<sup>52</sup>

### ***Acidity of biodiesel***

Biodiesel comes from oils and might include free fatty acids. ASTM D 664 is the standard reference method for determining the acid number of biodiesels.<sup>53</sup> Experiment results indicated that acid number of biodiesel made from treated waste oil is lower than untreated one. (Table 4). Acid number of both biodiesel is meet the standard limits defined for biodiesels by ASTM (<0.5%).<sup>52</sup> According to all tested properties of two different biodiesel, it could be concluded that adsorption process with the aid of egg shell and its active carbon, cause notable

improvement in biodiesel production from waste oil. These improvements consist of increasing biodiesel yield up to 70% and lowering acid number of biodiesel. Resultant biodiesel has standard viscosity and specific gravity value as well.

### **Conclusion**

Owing to the interfering effect of impurities in transesterification reaction, elimination of free fatty acids and peroxides could promote biodiesel yield and quality. A mixture of eggshell and its active carbon could reduce acidity and peroxide value up to 71.17 and 51.72%, respectively. Biodiesel coming from treated waste oil also show better properties compared to untreated one especially in terms of biodiesel yield (37.61% increase), so the application of the eggshell adsorbent offers a convenient and suitable method for waste oil purification.

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