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Production and Purification of Biodiesel Produced from Used Frying Oil

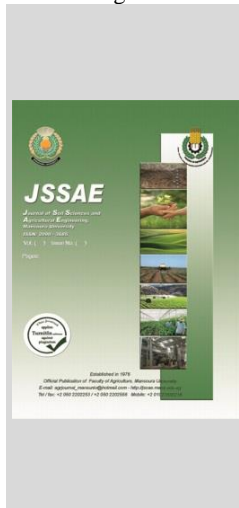
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ABSTRACT

This study aims to investigate the possibility of biodiesel production from used frying oil and purify the produced biodiesel using dry (activated charcoal - magnesium silicate) and wet (acid water) washing methods. A biodiesel production pilot unit was constructed and installed at the energy laboratory, Agric. Eng. Dept., Faculty of Agric, Mansoura Univ., Egypt. Methanol and potassium hydroxide were used as catalysts. Glycerol was the bottom layer that was deposited by gravity, and the upper layer was biodiesel. Biodiesel was purified using dry washing (5% activated charcoal and 1% magnesium silicate) (w/w) at 65 °C and stirring for 10, 20, and 30 minutes, and then microfiltrated to separate biodiesel from dry purifiers. The biodiesel produced was also purified using acid water. Impure biodiesel was washed with 10% acid water at 55 °C. A decrease in free fatty acids was observed during the 30-minute stirring period, from 2.26% before purification to levels of 0.23, 0.14 and 0.23% for the activated charcoal, magnesium silicate, and acid water, respectively. The physical properties of the produced biodiesel were estimated. Purification with the activated charcoal gave the best results compared to purification with magnesium silicate and acid water, as it was lower in viscosity (7.88–5.70%), higher in flash point (5.72–8.58%), and the least in biodiesel losses. During purification, it was less than magnesium silicate and acid water by (5% and 25%) respectively. The rest of the properties are among those listed according to ASTM D6751. It was also the cheapest material used for the purification process.

Keywords: Purification – Biodiesel – Dry washing – Wet washing – Properties.

INTRODUCTION

Many researchers are interested in creating substitute fuels for internal combustion (IC) engines. Vegetable oil, biogas, biomass, and alcohol are just a few examples of alternative fuels that are all naturally renewable. Vegetable oil stands out among them as a crucial diesel fuel substitute for internal combustion engines due to its accessibility, biodegradability, lack of toxicity, and environmental friendliness. In his diesel engine, Rudolf Diesel used peanut vegetable oil because of its availability. Several oils have been used as raw materials for biodiesel manufacturing in various nations. Surplus edible oils such as soybean oil, sunflower oil, and rapeseed oil are utilized as feedstock for biodiesel manufacturing in the United States and Europe (Shelke et al., 2018).

Using this strategy could aid in achieving the objectives of a greenhouse solution to reduce the earth's temperature. The plan can include switching to renewable energy (RE) systems. Renewable energy resources including solar, wind, bio, and hydro-energy resources are employed to supply all of the country's energy needs. One of the goals of the European Union (EU) is to increase the share of renewable up by 27% by 2030 (Mottaeva and Cetkovic., 2018).

Biodiesel produces no sulphur, no net carbon dioxide, less carbon monoxide, no particulate matter, no smoke, and no hydrocarbons as compared to diesel. Complete combustion and lower emissions are produced when there is more free oxygen. Compared to low-sulphur diesel fuels, biodiesel offers higher cetane ratings and lubricating characteristics. Its calorific value is roughly 37.27 MJ/kg, which is 9% less than that of ordinary petroleum and a flash

point of more than 423 K compared to 350 K for petroleum (Mishra and Goswami, 2018).

In recent years, biodiesel has been made from a variety of edible and non-edible sources, including animal fats, soap stock, recycled frying oils, macro and microalgae. Several sources with different compositions and levels of purity can be used to get the feedstock needed to make biodiesel. The selection of feedstock, which is the first step in the production of biodiesel, is impacted by elements like yield, composition, and cost. The availability and kind of feedstock source are the main determinants for categorizing biodiesel into edible, non-edible, and waste-based sources. The feedstock utilized to make biodiesel is influenced by location as well. Before choosing the feedstock, the accessibility and economic conditions of the country are primarily taken into consideration (Hamza et al., 2021).

Biodiesel is an alkyl ester of fatty acids produced by the transesterification of oils or fats from plants or animals with short-chain alcohols like methanol and ethanol in the presence of a catalyst. As a result, glycerin is a by-product of biodiesel manufacturing (Fatah et al., 2012). Berrios and Skelton, (2008) found that, in this reaction, triglycerides, as the main components of vegetable oils, react with an alcohol to produce fatty acid monoalkyl esters and glycerol, that is separated from biodiesel and then biodiesel is purified. As defined by the American Society for Testing and Materials (ASTM D 6751) biodiesel is a fuel composed of monoalkyl esters of long-chain fatty acids derived from renewable vegetable oils or animal fats.

Methyl esters cannot be classified as biodiesel until EN14214 or ASTM D6751 meet the standard specifications.

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Therefore, the purification phase is necessary. Unprocessed biodiesel contains several Impurities, such as free glycerin, soap, metal, methanol, Free Fatty Acid (FFA), catalyst, water, and glycerides. The presence of these impurities reduces the life of the engine. There are two ways to purify biodiesel: wet and dry Purification. The most traditional wet washing method is widely used to remove excess pollutants and chemical residues from biodiesel. However, wet purification has many drawbacks, including increased cost and production time, and highly polluted effluents are generated. This leads to significant product loss and the composition of emulsions when processing used cooking oils with a high FFA content (Kirubakaran and Selvan.,2018).

Dry purification replaces water with ion exchange resin, Amberlite BD10 Dry, Burolite PD 206 and silica or magnesium silicate powder to neutralize impurities. Dry washing methods are used in Industrial facilities. This method; is characterized by low losses, low cost, and achieves the desired results according to ASTM D6751 (Atadashi, 2015).

This study aims to investigate the possibility of biodiesel production from the used frying oil and purify the produced biodiesel using two different washing methods: dry (activated charcoal and magnesium silicate) and wet (acid water) washing methods.

MATERIALS AND METHODS

Biodiesel feedstock.

Used frying oil.

The main source of the experimental work is the frying oil used in restaurants. The required quantity was collected from different restaurants. This oil has different types and is used to fry different foods. The combined oil was mixed well to be homogeneous, while its fatty acid composition and some of its properties were evaluated. Data tabulated in Table (1) show the composition of fatty acids of the used frying oil. The analysis was performed with a GC device in the chromatographic laboratory at the National Research Center (NRC) in Cairo, Egypt.

Table 1. Composition of the used frying oil fatty acids.

Components of the used frying oil	Wt, %
Lauric acid	0.15
Myristic acid	0.71
Palmitic acid	26.66
Stearic acid	5.15
Oleic acid	34.35
Linoleic acid	30.48
Linolenic acid	1.76
Arachidic acid	0.28
Cis.11.Eicosenic	0.21
Behenic acid	0.18
EPA	0.07

Chemicals used to produce biodiesel.

The chemicals used to produce biodiesel during the transesterification reaction were as follows:

- Methanol alcohol at concentration of 99%.
- potassium hydroxide at concentration of 90%.

Chemicals used to purify biodiesel.

To study the effect of dry washing on the purification of the produced biodiesel, two different materials were used activated charcoal and magnesium silicate (magnesol).

The activated charcoal

Due to its high surface area and the presence of an oxygen group on its surface, the activated charcoal was used

as an adsorbent substance to remove the contaminants from the biodiesel. These features help in better interactions (physical or chemical) between the surface of the material used for the purification process and the produced biodiesel (Atadashi, 2015). Fig. (1) and Table (2) show the properties of the activated charcoal was used for experimental work according to Piochem Lab. Chemical (2021).



Fig. 1. The activated charcoal

Table 2. Physical properties of the activated charcoal.

Item	Specification
Appearance	Black light powder
Particle size, mm	0.3 – 0.5
Melting, °C	3550
Assay, wt.%	97 – 99
Ignition residue, wt.%	2

The magnesium silicate (Magnesol)

Magnesium silicate (60 µm particle size diameter) is the mineral that was utilized to clean the produced biodiesel in a dry washing (waterless) procedure. One normal brand name for the synthetic magnesium silicate is Magnesol. Likewise, with ion exchange resins, less waste is produced, equivalent to about 1% of the biodiesel weight. Fig. (2) and Table (3) show the properties of magnesium silicate (Magnesol) was used for the experimental work according to Alpha Chemika (2021).



Fig. 2. The magnesium silicate (Magnesol).

Table 3. Physical properties of the synthetic magnesium silicate (magnesol).

Component	wt. %
Assay	99.00
Chloride (Cl)	0.08
Sulphate (SO ₄)	0.004
Iron (Fe)	0.0001

Biodiesel production procedure.

Biodiesel was produced from the used frying oil using the transesterification process as recommended by many researchers (Sharma et al., 2008) (Atadashi, 2015). The transesterification procedure involves the following steps:

- 1- The used frying oil collected from restaurants was heated at 80 °C as recommended by (Conceicao et. al., 2007) for 10 minutes with constant stirring to get rid of any moisture

- content resulting from the cooking process of different foods.
- 2- The heated oil was filtered to get rid of any impurities or food residues. The filtering process was done after heating due to the high viscosity of the oil and the difficulty in filtering it before heating.
 - 3- A titration process was performed to determine the appropriate amount of catalyst (NaOH or KOH) needed to neutralize the fatty acids in the oil. Adding more or less quantity of the catalyst reduces the amount of the produced biodiesel. The amount of catalyst required can be calculated from the following steps:
 - Dissolve 1 gram of potassium hydroxide or sodium hydroxide with 1 liter of distilled water in a beaker.
 - Dissolve 1 ml of the used oil in 10 ml of pure isopropyl alcohol.

- Gently heat the beaker in a water bath, with constant stirring, until the oil dissolves in the alcohol.
- Add 3 drops of pH indicator (phenolphthalein) to the solution.
- The titration is done with 0.1% KOH or NaOH solution.
- Notice the color changing to a light pink, with stability for 30 seconds.
- The number of milliliters of titrant solution was recorded as shown in Table (4), while Table (5) shows the appropriate volume of different catalysts for the examined used oil.

Table 4. Catalyst amount determined by the titration process.

Titration No.	KOH, ml	NaOH, ml
Rep.1	4.9	2.2
Rep.2	5.2	2.3
Rep.3	4.5	2.4
Average	4.9	2.3

Table 5. Volume of catalyst for 1000 ml of used oil.

KOH, ml	NaOH, ml
Average= 4.9 ml	Average= 2.3 ml
Add 7 to the result	Add 5.5 to the result
Utah supplies biodiesel According to (2005)	Utah supplies biodiesel According to (2005)
In most cases, it will be difficult to find 100% pure KOH or NaOH.	Use NaOH which is 96% pure, then divide 5.5 by 0.96
Use KOH which is 90% pure, then divide 7 by 0.90	(5.5 / 0.96 = 5.73)
(7 / 0.90 = 7.78)	2.4+5.73= 8.13 grams/liter
4.9+7.78= 12.67 grams/liter	1000 ml oil need 8.13 g
1000 ml oil need 12.67 g	

The number of the used oil moles as stated by El-Wehishy et al., (2018) was estimated as the following steps:
 Mass of used oil, g = Batch volume, ml x oil density, g/ml
Number of oil moles = Mass of used oil, g / oil molecular weight, mole
Number of methanol moles = number of used oil moles x parts of methanol (6 in the applied molar ratio).
Mass of methanol = number of methanol moles x methanol molecular weight 32 g/mole
Volume of methanol = mass of methanol / methanol density (0.79 g/mole).

The required amount of catalyst KOH was estimated as 1% by weight of the above specified batch of oil.

Transesterification process.

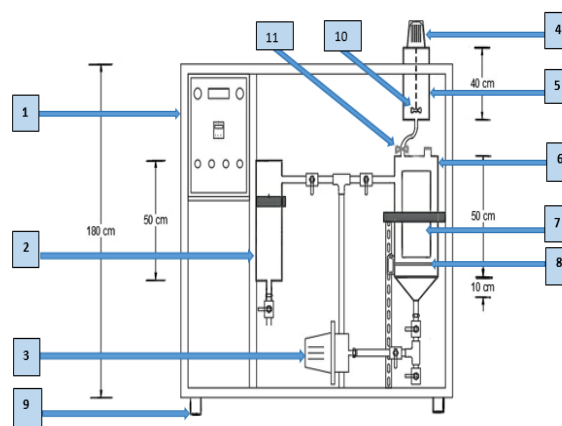
To produce biodiesel, (Alama et. al., 2016) stated that, the reaction is called transesterification. The alcohol is mixed with the catalyst for the reaction, typically a strong base such as NaOH or KOH. The alcohol and catalyst are then reacted with the fatty acid, so that the transesterification reaction occurs. The catalyst is prepared by mixing methanol with a strong base, such as sodium hydroxide or potassium hydroxide. During the preparation, the KOH breaks into ions of K⁺ and OH⁻. The OH⁻ abstracts the hydrogen from methanol to form water and leaves the CH₃O⁻ available for reaction. Methanol should be as dry as possible. When the OH⁻ ion reacts with the H⁺ ion, it reacts to form water.

In these experiments, the oil was heated at 65°C with hydraulic stirring for an hour. The mixture was left to separate by gravity for 24 hours to form two layers. The upper layer is the methyl ester, and the lower is glycerol. The glycerol was separated by opening the outlet valve. Methyl ester remains to begin the purification stage and is heated at 110 °C to evaporate methanol and water.

Biodiesel production laboratory unit.

A biodiesel production unit (Fig. 3) was constructed and installed in the Energy laboratory at the Agricultural Engineering Department – Faculty of Agriculture – Mansoura

University, to produce and purify biodiesel in batch experiments and it could be used also for continuous operation. The installed unit consists of three different units including a chemical mixing part, a reaction and separation part, and a purification part. The unit is equipped with four wheels for easy transportation and movement. Also, it contains an operating control unit.



1	Control panel	7	Observation window
2	Purification part	8	Electric heater
3	Stainless steel electric pump	9	Wheels
4	Electric motor	10	Stirrer
5	Chemical mixing part	11	Stainless steel valve
6	Reaction and separation part		

Fig. 3. Laboratory scale biodiesel production unit.

The chemical mixing part consists of a (stainless steel 316) cylinder (15 cm in diameter, 40 cm in height, and 7.1 liter volume). The chemicals are mixed inside the cylinder using a stainless steel mechanical stirrer, which is connected to a small electric motor installed in the top cover of the cylinder. The lower part is connected to the reaction and separation unit via a stainless steel valve.

The reaction and separation part consists of a stainless steel cylinder (25 cm in diameter, 60 cm in height). The bottom part of the cylinder is conical in shape with a height of 10 cm and the total volume of this unit is 26.3 liter. A transparent acrylic window was installed on the front surface of the cylinder to monitor the oil during the reaction process. Also, it contains an electric heater (3000 W) to heat the oil during the reaction. The raw material is fed from the upper part of the reaction cylinder and the oil is circulated during the hydraulic stirring cycle using an electric pump and valves that control the direction of the flow.

The purification part for the dry washing of the produced biodiesel consists of a stainless steel cylinder (15 cm in diameter, 50 cm in height, with a volume of 8.8 liters). It has a side crude biodiesel inlet, and there are also two filter holders installed inside the cylinder. The filters were operated according to microfiltration theory. Filters can contain many different sizes of pores ranging from 1 to 200 microns. The crude biodiesel passes through the filter after adjusting its path direction using the control valves, thus, the biodiesel is purified from impurities.

The Purification process of biodiesel.

Biodiesel Separation.

After the transesterification process the first step, often used to recover the produced biodiesel is the separation of crude biodiesel from the by-product (glycerol). The quick separation of glycerol from biodiesel is caused by large differences in their densities as well as differences in their polarity (Ogunwole., 2015).

Glycerol and biodiesel have densities of at least 1.05 g/cc and 0.88 g/cc, respectively (Alsultan et al., 2021). The proportions of water, catalyst, and methanol in glycerol determine its density. This density difference allows for the separation of glycerol phase from the biodiesel phase using a straightforward gravity separation approach. Settling times could be as short as a few minutes or as long as 24 hours according to Gómez-Hernández et al. (2021).

Biodiesel washing.

In this study, purification of the produced biodiesel was carried out by dry activated charcoal and magnesium silicate (magnesol) and wet (acid water) washing methods.

Dry washing:

The crude biodiesel was purified using the dry washing method (5% activated charcoal or 1% magnesium silicate) by weight of the produced biodiesel, heating at 65 °C, and the mixture stirred for 10, 20 and 30 minutes respectively. The final stage is filtering the mixture to get rid of any sediment.

Wet washing:

The impure biodiesel was washed with 10% acid water at 55 °C. Acid water was prepared by adding 2% (v/v) phosphoric acid to the distilled water. The acid water neutralizes the catalyst and decomposes the soap formation (Faccini et al., 2011). This washing step was performed in the same esterification unit, stirring gently so that an emulsion would not form. After different stirring times (10, 20, and 30 minutes) the mixture was transferred to the separation funnel which settled into two layers. The top layer is a washed biodiesel, while the bottom layer is a contaminated water, then disposed of the water. This process was repeated (four times) on the biodiesel until the water becomes completely colorless. The washed biodiesel was heated at 110 °C to get

rid of any water. It can be seen from Fig. (4) the color change of water resulting from the purification process during the four stages. Fig. (5) illustrate the final form of purification for each studied method.

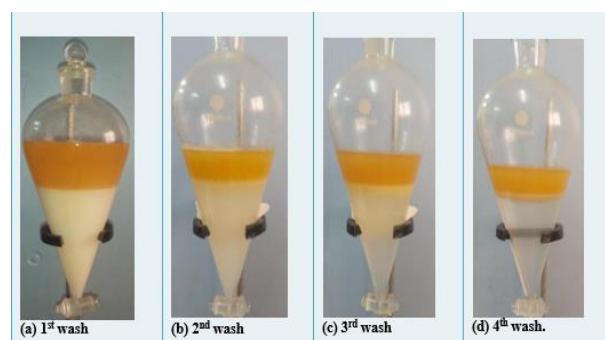


Fig. 4. Biodiesel and wastewater layers.

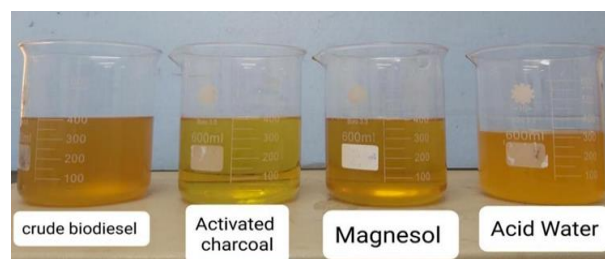


Fig. 5. The final form of each purification method.

Evaluation of the produced biodiesel.

Free fatty acids and acid number.

The acid number (AN) or neutralization number is a measure of the amount of free fatty acids present in the fresh fuel sample and the free fatty acids and acids resulting from decomposition in the old samples. This test is used to determine acidic components in the produced biodiesel fuel. It is expressed in mg KOH required to neutralize 1 gram of fatty acids of methyl esters (FAME). The sample is dissolved in a mixture of toluene and propanol containing a small amount of water and titrated potentiometrically with alcoholic KOH. Endpoints are noted and readings of titrant volumes are taken and used in a formula to calculate the total acid number in the biodiesel sample.

Fatty acid peaks were identified by comparison to the retention times of reference standards. For the determination of free fatty acid content, 0.5 g of biodiesel, 10 ml of ethanol and 1–2 drops of phenolphthalein were used as indicator. The mixture was titrated against with 0.1 N NaOH and change in color was monitored. The FFA content was expressed g/100 g as oleic acid and calculated as shown in Eq. (1). The acid value was

calculated using relation shown in Eq. (2). as mentioned by Ogunwole., (2015).

$$\text{FFA content \%} = V \times N \times \frac{28.2}{W} \quad (1)$$

$$\text{Acid value (mg KOH/g fat)} = \frac{V \times N \times 56.1}{W} \quad (2)$$

V = Volume of KOH titrant used, (ml)

W = Weight of oil, (g)

N = Normality of KOH

Biodiesel impurities.

Saponification value of the produced biodiesel sample.

For the determination of the saponification value of the produced biodiesel, 0.5 g biodiesel and 20 ml of 0.5 N alcoholic (ethanol) KOH were mixed. The mixture was

placed in a round bottom flask, refluxed and heated at 40 °C until the appearance of clear solution, as indicator of the saponification reaction. After cooling the contents, phenolphthalein was added as indicator and the mixture was titrated against with 0.5 N HCl until the pink color disappeared. The saponification value was determined using the relation shown in Eq.(3) as mentioned by Ogunwole.,(2015).

$$\text{Saponification value} = \frac{28.05 \times (B-A)}{W} \quad (3)$$

Where:

B= ml of HCL used in blank titration

A= ml of 0.5N HCL slution used for titration

W= weight of sample

Total glycerin and methanol.

The GC 7890B model from Agilent Technologies is flame-equipped Ionization detector in the network of central laboratories at the National Research Center Cairo Egypt, was used to determine the total glycerin and methanol of the produced biodiesel. Injector and detector (FID) was held at 280°C and 300°C respectively. Reagent gases: nitrogen gas 20 ml/min, hydrogen 35 ml/min, air 280 ml /min.

Physical and chemical properties of biodiesel.

The physical properties of the produced biodiesel were measured at the National Research Centre. These properties were determined according to ASTM D6751 procedures for petroleum products, as shown in the Table (6).

Table 6. Tests for fuel properties according to the international standard (EN 14214) and (ASTM D6751).

Type of test	Methods	Ref.
Density (Kg/L) at 15 °C	EN-ISO 3675/12185	EN test method
Calorific value (MJ/L)	D-240	
Kinematic viscosity mm ² /sec at 40 °C	D-445	
Cetane number	D-613	ASTM test method
Pour point (°C)	D-97	
Cloud point (°C)	D-2500	
Flash piont (°C)	D-93	

Biodiesel losses during the purification process.

The lost percentage of biodiesel during the purification process was calculated using the equation (4) as mentioned by Ogunwole., (2015).

$$\text{Lost percentage} = \frac{W_1 - W_2}{W_1} \times 100 \quad (4)$$

Where:

W₁=The volume of biodiesel before filtration, ml.

W₂= the volume of biodiesel after filtration, ml.

RESULTS AND DISCUSSIONS

Experimental variables parameters.

Biodiesel production

A comparison was made between the use of sodium hydroxide and potassium hydroxide as catalysts during the transesterification reaction.

Biodiesel purification

In order to obtain good quality of biodiesel, it is important to choose the best way to get rid of chemicals, glycerin, and unreacted oil in the fuel. In this work, the dry washing method (activated charcoal - magnesium silicate (magnesol)) and the wet washing method (acid water) are the two methods used for biodiesel purification.

Biodiesel purification time

Three stirring times of 10, 20, and 30 minutes of purification of the produced biodiesel were used to determine

the effect of purification time on the reduction of acid number and free fatty acids.

Effect of using sodium hydroxide and potassium hydroxide as a catalyst for the transesterification reaction.

Two experiments were conducted to determine the appropriate catalyst for producing biodiesel from used frying oil. The data showed that it is better to use a potassium hydroxide catalyst for producing biodiesel as it has a conversion efficiency of 92%, in contrast to sodium hydroxide, which produces a very small amount of biodiesel and large amounts of glycerin and soap.

Effect of purification time on Free Fatty Acids (FFA) and acid value.

High content of free fatty acids in the used frying oils, reduces the efficiency of biodiesel production and increases soap formation. Free fatty acids could be reduced through the washing process. FFA percentages and acid values of the produced biodiesel samples purified by the three examined methods at three stirring times of 10, 20, and 30 minutes were shown in Table (7) and Fig.(6). The free fatty acids of the used oil are estimated at 3.56 %, then it became 2.26 % in the biodiesel before purification. As shown in Table (7), the FFA% decreased from a level of 2.26 % for the produced biodiesel to the lowest values of 0.23%, 0.14% and 0.23% for the purification methods of activated charcoal, magnesium silicate and acid water at a purification time of 30 min, respectively.

The corresponding acid value for the purified biodiesel were 0.46,0.27 and 0.46 mgKOH/g respectively. In general, it could be concluded that, the purification time of 30 min is considered the most proper for the purification process, These results were within the permissible range according to ASTM D6751.

Table 7. Effect of stirring time on free fatty acids content.

No.	Purification method	purification time (min)	FFA%
1		10	0.45
2	Activated charcoal	20	0.30
3		30	0.23
4		10	0.16
5	magnesium silicate (Magnesol)	20	0.15
6		30	0.14
7		10	0.28
8	Acid water	20	0.25
9		30	0.23

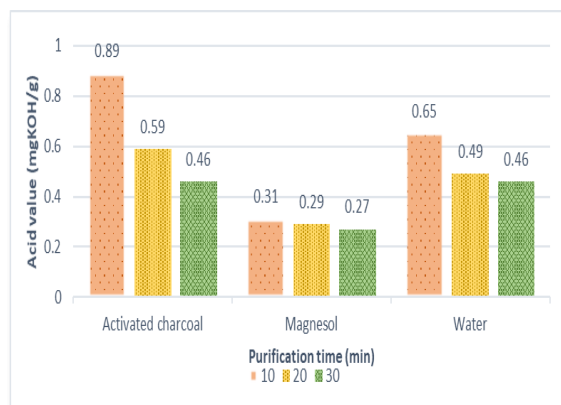


Fig. 6. Effect of reaction time on acid value.

Effect of purification methods on biodiesel impurities.

Table (8) shows the levels of impurities (Methanol - Total glycerine - Soap - Water content) present in biodiesel before and after purification by dry washing (activated charcoal- magnesium silicate) and wet washing (Acid water).

Table 8. Effect of purification methods on biodiesel impurities .

Impurities	Before Purification	After purification			Biodiesel standard ASTM D6751
		Activated charcoal	Magnesium silicate (Magnesol)	Acid water	
Water content, %	0.0088	0.0043	0.0035	0.0124	0.005 vol%
Total glycerin, %	0.4160	0.0050	0.0150	0.0300	0.240
Methanol, %	4.4840	0.1300	0.0700	0.1100	0.200
Soap, ppm	7.6000	1.5000	6.7000	3.0000	-

The results showed that, water content in the produced biodiesel before purification was (0.0088 %) and became 0.0043, 0.0035 and 0.0124% with purification by activated charcoal, magnesol, and acid water respectively. The water content was decreased by 51.14 and 60.23% through purification with activated charcoal and magnesol respectively. In contrast, the water content increased by 40.91 % in the case of purification with acid water. These results range within the permissible levels of the standard biodiesel specifications ASTM D6751.

The most effective method to reduce the amount of total glycerin is activated charcoal, which was more effective than magnesol and acid water by 2.41 and 6.01% respectively. These results are within the permissible range of the standard biodiesel specifications (ASTM). The results also show that,

the magnesol washing method was the most efficient in removing methanol, as it was 1.34 % and 0.89 % more efficient than the activated charcoal and acid water respectively. These results are also within the permissible range of the standard biodiesel specifications ASTM and EN 12412. Meanwhile, the activated charcoal is considered the most efficient in removing the formed soap, as it was more efficient in removing soap using magnesol and acid water by 68.42% and 19.73%, respectively.

Effect of purification method on physical and chemical properties of biodiesel.

Data tabulated in Table (9) shows the physical and chemical properties of the produced biodiesel under different purification methods.

Table 9. Physical and chemical properties of the produced biodiesel.

Properties	Before purification	After purification			biodiesel Standard
		Activated charcoal	Magnesol	Acid Water	
FFA%	0.92	0.23	0.14	0.23	-
Acid value (mgKOH/g)	1.84	0.46	0.27	0.46	0.50
Density at 15°C, g/ml	0.8861	0.8845	0.8856	0.8859	0.800-0.900
Calorific value, MJ/Kg	44.8000	44.8800	44.9300	44.9400	-
Viscosity at 40 °C, mm ² /s	6.4270	4.6610	5.1670	5.0270	1.9: 6.0
Flash point, °C	70	134	130	128	< 93
Cloud point, °C	5	3.5	4	3	-3 : 12
Pour point, °C	-4	-5	-5.8	-6	-15 : 5

It is clear that, through different purification methods, the produced biodiesel viscosity was reduced by 27.48%, 19.6%, and 21.78% for biodiesel purified by activated charcoal, magnesol, and acid water, respectively. The lowest value of viscosity was achieved through the purification method of activated charcoal, where the viscosity was reduced by 7.88,5.70%, for the methods of magnesol, and acid water, respectively. Therefore, the activated charcoal is considered one of the most commonly used methods in this research to reduce viscosity. It could be concluded that all results are consistent with the standard specified by ASTM D6751.

According to ASTM D6751 and EN 14214, the density should be between 0.86 – 0.90 g /ml for biodiesel standard. The results in the previous table present the density values before and after purification using the activated charcoal, magnesol and acid water which were 0.8861, 0.8845, 0.8856 and 0.8859 g /ml respectively. After purification it was noticed a decrease in density compared to before purification, where the percentage of decrease were 0.1805, 0.056 and 0.0225 % for the activated charcoal, magnesol and acid water respectively. It can be seen that the density of all biodiesel fuels was consistent with the range specified by the biodiesel standards.

The results of biodiesel calorific values before and after purification with activated charcoal, magnesol, and acid water, were 44.80, 44.88, 44.93 and 44.94 kJ/kg respectively. The calorific value was increased after purification by 0.18, 0.29 and 0.31% using the activated charcoal, magnesol, and

acid water, respectively. The highest increase in calorific value was achieved through purification with the acid water, so the acid water was 0.13 and 0.02% more efficient compared to the activated charcoal and magnesol, respectively. These results are also agree with that found by (Demirbas.,2009) who reported that the calorific value of biodiesel is relatively high (39 – 41 MJ/kg) but slightly less than gasoline (46 MJ/kg) and diesel gasoline (43 MJ/kg).

It is evident that, the flash points before and after purification with the activated charcoal, magnesol, and acid water were 70, 134, 130 and 128 °C, respectively. Through the examined purification methods, the flash point was increased by 91.43, 85.71, and 82.85% with the activated charcoal, magnesol, and acid water, respectively. The highest degree of flash point was achieved through the activated charcoal purification method, as it was greater by 5.72 and 8.58% than the magnesol and acid water, respectively. These results indicate that all the biodiesel produced meets the flash point specifications of ASTM D6751. Also, it is clear that, the cloud point of the produced biodiesel is in the order of 5, 3.5, 4, and 3 °C before purification and after purification with activated charcoal, magnesol, and acid water, respectively. The Cloud Point temperature was decreased by 30, 20, and 40 %, respectively, by using the activated charcoal, magnesol, and acid water. The lowest temperature was obtained during purification with acid water, as it was lower by 10 and 20% than that of activated charcoal and magnesol, respectively.

The pour point of biodiesel before purification and after purification with activated charcoal, magnesol, and acid

water, was - 4, - 5, -5.8 and - 6 °C respectively. The pour point was decreased by 50, 45, and 25% for the purification methods of activated charcoal, magnesol, and acid water, respectively. It has been observed that most of the intake points and pour points comply consistence with biodiesel standards.

It can be seen that the acid value after purification with activated charcoal, magnesol, and acid water was 1.84, 0.46, 0.27 and 0.46 mgKOH/g respectively. Through purification, the acid number was decreased by 75, 85.33, and 75% for activated charcoal, magnesol, and acid water treatments, respectively. The lowest acid number was obtained through magnesol purification, which was 10.33% lower than both

acid water and activated charcoal treatments. This may be due to the acid water and activated charcoal are activated by acid, they contain a percentage of it that causes a slight increase in magnesol corresponds to the maximum specified in ASTM D6751 and EN 14214 standards below 0.5 mg KOH/g oil.

Biodiesel losses during various purification methods.

During the purification of biodiesel, part of it is exposed to loss during the washing process with water when separated from biodiesel, where it is difficult to control that. The largest percentage of losses approached 30% for acid water. For dry washing, the percentage of losses with activated charcoal and magnesol, were 5 and 10% respectively (Fig. 7).

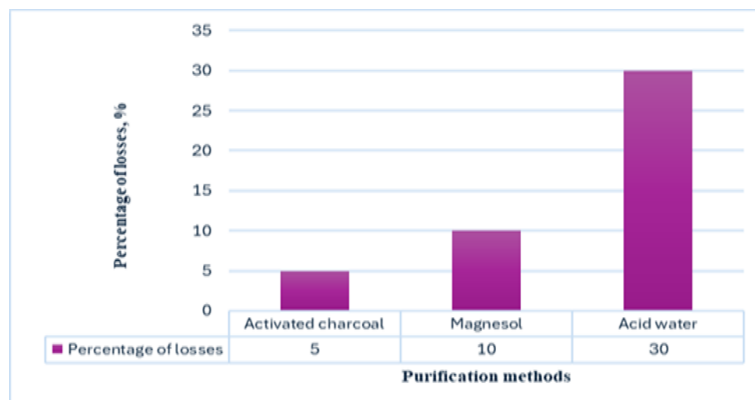


Fig. 7. Biodiesel losses through different purification methods.

CONCLUSION

- During the production of biodiesel using two types of catalysts, sodium hydroxide and potassium hydroxide, potassium hydroxide was more efficient in producing biodiesel. In contrast, sodium hydroxide did not produce a suitable amount of biodiesel, as a large amount of soap was formed when used.

- Dry purification methods with activated charcoal and magnesium silicate as an alternative for wet purification method using wet washing to overcome the problem of the required large amount of water disposed in the drain and the corresponding high operation cost of the process.

- Purification time of 30 min is recommended for conducting the dry purification process with high efficiency.

- Dry purification with activated charcoal has achieved the required results for the produced biodiesel, which has the lowest viscosity, the highest flash point, the lowest pour point, and the lowest percentage of produced biodiesel losses.

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إنتاج و تنقية الوقود الحيوي الناتج من زيت الطهي المستعمل

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المخلص

يهدف هذا البحث إلى دراسة إمكانية إنتاج الوقود الحيوي (Biodiesel) من زيت الطهي المستعمل والذي تم تجميعه من العديد من المطاعم وخطه جيدا ليكون متجانساً في مكوناته. تم إضافة كحول الميثانول و هيدروكسيد البوتاسيوم كمحفزات للتفاعل وذلك باستخدام التقليل الهيدروليكي لمدة ساعة على درجة حرارة 65 درجة مئوية بوحدة معملية لإنتاج الوقود الحيوي بمعمل الطاقة بقسم الهندسة الزراعية - كلية الزراعة - جامعة المنصورة. تم فصل الجليسرين والشوائب الناتجة من التفاعل عن الوقود الحيوي خلال 24 ساعة حيث تتكون في الطبقة السفلى وذلك لزيادة كثافة الجليسرين مقارنة بالوقود الحيوي الناتج والذي يشكل الطبقة العليا داخل وحدة التفاعل. تم إجراء مقارنة خلال مرحلة إنتاج الوقود الحيوي باستخدام نوعين من المحفزات القلوية (هيدروكسيد البوتاسيوم - هيدروكسيد الصوديوم) للحصول على أكبر نسبة من الوقود الحيوي. تم إجراء مقارنة بين ثلاث طرق للتنقية اشتملت على التنقية الجافة باستخدام (5%) فحم نشط و 1% سليكات المغنسيوم الامامية) والتنقية الرطبة باستخدام الماء المحمض (تم تحضيره بإضافة 2% (حجم/حجم) من حمض الفوسفوريك المركز والماء المقطر، وهنا يساعد الماء المحمض في معادلة المحفز المتبقي من التفاعل وتحلل الصابون المتكون) عند ثلاث أزمنة مختلفة (10، 20، 30 دقيقة) ثم الترشيح الدقيق لفصل الفحم النشط وسليكات المغنسيوم عن الوقود الحيوي (Biodiesel). أوضحت النتائج المتحصل عليها، كان هيدروكسيد البوتاسيوم أكثر كفاءة كمحفز للتفاعل أثناء إنتاج البيوديزل مقارنة بهيدروكسيد الصوديوم الذي شكل كمية كبيرة من المواد الصابونية مما أدى إلى انخفاض كمية البيوديزل الناتج ولذلك تم استخدام هيدروكسيد البوتاسيوم كمحفز أثناء التفاعل في هذه الدراسة. إنخفاض الأحماض الدهنية الحرة خلال زمن تقليب 30 دقيقة حيث إنخفضت من 2.26 % للوقود الحيوي قبل التنقية إلى 0.23، 0.14، 0.23 % للفحم النشط وسليكات المغنسيوم والماء المحمض على التوالي. عند تقدير الخواص الفيزيائية للوقود الناتج بعد تنقيته أوضحت النتائج أن التنقية باستخدام الفحم النشط كانت الأفضل مقارنة بالتنقية باستخدام سليكات المغنسيوم أو الماء المحمض حيث كان الوقود الناتج الأقل لزوجة (7.88، 5.70 %) والأعلى في نقطة الوميض (5.72، 8.58 %) والأقل أيضاً في نسبة الفواقد (5، 25%) على التوالي والأقل تكلفة في طرق الغسيل المستخدمة، كما كانت باقي الخواص الفيزيائية للوقود الحيوي الناتج ضمن الحدود الموصى بها من الجمعية الأمريكية للإختبارات والمواد (ASTM D6751).