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Optimization Process for Biodiesel Production from Waste Cooking of Vegetable Oil by Microwave Irradiation

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HIGHLIGHTS

- The using of microwave oven technology with a multiple power source range in the process of producing biodiesel from waste cooking vegetable oil.
- One of the most important findings of a study is use of ethanol which is a renewable source of production instead of methanol as in other studies.
- Biodiesel was produced from waste cooking vegetable oil that can be used as an alternative to regular fuel and thus it is considered clean renewable energy, subject to recycling processes and environmentally friendly.

ABSTRACT

Biodiesel is the main alternative to diesel fuel today. The waste cooking vegetable oil can be used as a basic material in its preparation. Microwave irradiation is used in its production and it is considered one of the effective methods that do not consume much power. In this study, biodiesel is produce and determine the factors affecting the quality and quantity of production also investigated using experimental design technique. The effecting of operating variables power, time, and oil-to-ethanol molar ratio were studied, as well as the effect of interaction between operating variables on the quality of production. The optimum conditions that have been found are; the power that the microwave operates is 480 watts, the reaction time is 3.5 minutes, and the oil-to-ethanol molar ratio is 1:5. The maximum production is 91.63% at these optimum conditions. Also, the biodiesel production is testing which include physical properties specific gravity, kinematic viscosity, water content, cloud point, pour point, and flash point and it was in good agreement with standard diesel fuel.

ARTICLE INFO

1. Introduction

The demand for energy continues to rise, due to rapid manufacturing and regional growth. The main energy supplies are petroleum, coal and natural gas, but because of their non-renewable existence, these fossil fuels are shrinking day by day [1]. On the other hand, the misuse of these conventional energy products is also a cause of global warming, which must be resolved through the introduction of alternative energy sources [2]. Both energy and environmental degradation are a major problems that could be avoided by implementing renewable energy sources such as the production of biofuels from renewable sources and the adoption of safe and environmentally friendly technologies for biodiesel generation [3]. The phrase "waste vegetable oil" relates to vegetable oil that was used in the cooking food and is no longer viable for its intended use. Waste vegetable oil comes from a variety of sources including domestic, commercial and industrial sources [4]. Biodiesel can be made through a transesterification reaction from animal fats or vegetable oils with methanol or ethanol as a catalyst. Biodiesel used as a renewable fuel can minimize hydrocarbon, CO, SO, polycyclic aromatic hydrocarbons PAH and polychlorinated dibenzo-p-dioxin/2 dibenzofurans emissions [5-6]. There are many advantages for using biodiesel as:

The waste cooking oil that throws in water source to raise organic contamination; also it is effect on the pipes drain as well as wear and tear in pipes which leads to different diseases such as cholera, and high water treatment cost [7]. Simple to use: no modification of the vehicle or any fueling equipment required. Energy, efficiency and economy: proven power generation, performance and cost-effectiveness have transformed biodiesel into a useful fuel. Environmental impact: Biodiesel helps to reduce pollution and improve health by lowering CO_2 emissions, decrease the effects of global warming. Biodiesel is better to treat because it is less toxic than gasoline and easier to store [8]. There are many types of alcohols that are used in biodiesel

production. The most widely used short-chain types of alcohol are ethanol, methanol, and butanol. Although the usage of various alcohols poses certain variations with respect to the kinetics of reaction, the ultimate yield of esters is more or less unchangeable. Methanol dominated in most reviewed literature. Methyl ester was developed rather than ethyl, as the prevalent consumer products are methyl esters, methanol is usually cheaper than ethanol, and the downstream processing of reactants alcohol is far simpler [9]. Ethanol holds an edge in its potential to be sustainable. It can be generated from sustainable agricultural resources, thereby achieving full independence towards petroleum-based alcohols [10-11]. The bulk of methanol worldwide is manufactured utilizing natural gas as a raw material. However, there is increasing interest in the capacity to generate methanol from sustainable biomass resources [12].In addition to the entirely agricultural nature of the ethanol, the extra carbon atom brought by the ethanol molecule slightly increases the heat content and the cetane number. As well as the ethanol 's purely agricultural origin, the extra carbon atom provided by the ethanol molecule marginally raises the specific heat and the amount of cetane. Ethanol is therefore superior to methanol as an extraction agent, owing to the significantly higher dissolving capacity for oils. Another significant benefit of ethanol use is that the ethyl esters have lower pour points and cloud than the methyl esters [11]. Nevertheless, the usage of ethanol often has risks. Compared with the development of methyl esters the base-catalyzed formulation of ethyl ester is complicated. Ethanol reaction rate is not fast like methanol, which is related to the more complex ethoxide anion formation [12]. Formation of emulsion is often an issue at ethanolysis. Methanol and ethanol are not miscible at room temperature with triglycerides, so the reaction combination is typically mixed manually to improve mass transfer. Emulsions are typically produced during the reaction. During the instance of methanolysis, these emulsions rapidly and readily break down to create a loaded layer of glycerol at down, and rich methyl ester at top surface [11-13]. Sodium hydroxide (NaOH) and potassium hydroxide (KOH) particles are one of the most widely utilized alkaline catalysts in the biodiesel production, which are cheap and easy to manage in transport and storage [14]. In several studies biodiesel with the strongest properties has been obtained employing KOH as just a catalyst [15-16]. In addition, several other studies have obtained best outcomes using NaOH [17-18]. [Encinar, J. M.; Juan, F.; Gonzalez, J. F.; Rodriguez-Reinares, A.,][11] Studied the effect of the catalyst form on the development of esters produce over time and noting that the potassium catalysts had the strongest actions and also noticed that the hydroxides yielded greater percentage than the related methoxides as shown in Figure (1).These findings are somewhat close to those seen in the literature on procedures of cooking oil with methanol and other solvents used during transesterification [16-19-20]

There are many researches on biodiesel yield. In study [21] try to develop testing and optimizing system for a batch microwave using waste cooking vegetable oil (WCVO) that was used as biodiesel feedstock. Two catalysts, potassium hydroxide (KOH) and sodium hydroxide (NaOH) were tested in this study. Transesterification reactions between oil and methanol were carried out in presence of microwaves. It was predestined by utilization of microwaves, the reaction times were drastically reduced. As high as 99.5% conversions could be achieved for 0.5% KOH concentration. The optimum condition of 1:6 oil/-methanol molar ratio, 0.5% KOH catalyst and 6 minutes reaction time at 400W microwave power were analyzed concerning some significant specifications as fuel in diesel engine. The study [22] found the optimum conditions were using commercial SrO, 40 to 180 seconds reaction time,around 80ºC reaction temperature, 6 methanol to oil ratio, and 1,000W microwave power output. 99% and 93 % biodiesel conversion efficiency for cooking oil and waste cooking oil were reached within in these conditions.

The optimal conditions for synthesis [23] of biodiesel were 0.50 wt% KOH, 8:1 methanol to oil molar ratio, 32 °C reaction temperature, 30 min of reaction, and 600 rpm rate of stirring. Under these conditions, biodiesel with a yield of 95.20 ± 2.5% w/w was obtained. In the study [24], the best conversion of RO–AKO blend to MBD (96.12 ± 1.25%) and MEBD (94.23 ± 2.22) was attained at KOH concentration, 0.75% w/w of oil, alcohol/oil molar ratio, 6/1, mixing intensity, 600 rpm, reaction temperature, 60°C, and 45-min reaction period, whereas the best conditions that produced the highest yield of EBD (95.19 ± 2.0 %) were KOH concentration, 1.0 KOH % w/w of oil, ethanol/oil molar ratio, 8/1, mixing intensity, 600 rpm, reaction temperature, 65°C, and 75-min reaction period.

The main objective of this study is to produce biodiesel, which is a type of renewable energy source because it depends in the method of its production on waste cooking vegetable oils. The production method is economical because the production process was done using a microwave device, which helped reduce the reaction time from an hour, as in research No [25] to 3.5 minutes, thus saving a large part of the energy consumed in the production process.
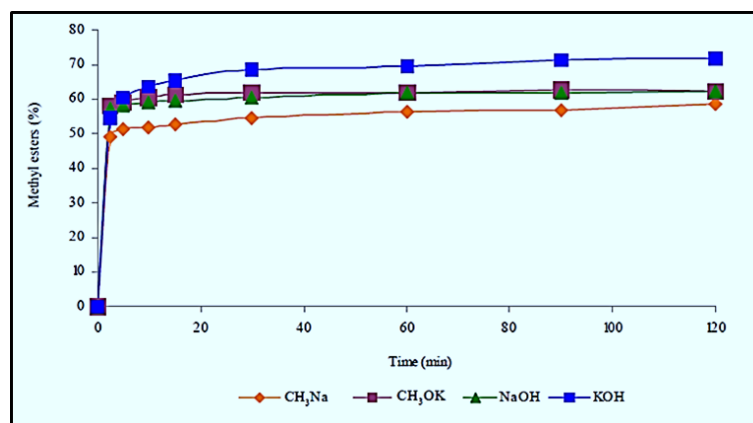

Figure 1: The effect of catalyst type on ester production [11]

2. Optimization Technique

2.1 Response Surface Method

Response Surface Methodology (RSM) comes from statistical and mathematical techniques. This technique can be used to investigate the effects of many variables at different levels and their effects on each other. The 3-factor, 5-level, a frequently used type of RSM, was chosen to define significant parameters influencing parameter interactions [26].

2.2 Design of experiments (DOE)

It is necessary to know the parameters that have a considerable effect on the system behavior, or the factors that influence on the objective function of the system before studying any process. So must do several experiments to include all effect of each parameter and also the interactions, between parameters if they are not independent. The systematic way which satisfies the these function with minimum digit of experimental is called "Experimental Design". The implementation of the experimental design to schedule the experiments requires to test the system, knowledge of the information from pre-existing data by utilizing a statistical method to explain the results in the normal form with the minimum digit of observations [27].

The technique of experimental design consists of two parts [27]:

1) Designing the experiments according to a specified design, with noting the description of the parameters values in the designs by a coded form.
2) Achieving the retraction analysis for the specific set of runs in the design, with noting the coded form of the parameters and the results of the objectives function regarding each experiment in the set.

2.3 Optimum conditions

WinQSB version (1.0) is the software used to find the optimum conditions for obtaining the maximum yield. The optimum condition can be obtained by constructing a set of 20 constrains, the constraints are defined as [28].

$a11x1 + a12x2 + \ldots \ldots a1nxn \ (\leq, \geq, =) \ b1$

$a21x1 + a22x2 + \ldots \ldots a2nxn \ (\leq, \geq, =) \ b2$

$am1x1 + am2x2 + \ldots \ldots amnxn \ (\leq, \geq, =) \ bm$

$xi \geq 0 \ (i = 1, 2, \ldots.n)$, n= number of variables, m= number of constraints,

$j = 1, 2 \ldots n, \quad i = 1, 2 \ldots m.$

The principle of the program's work in this case is to search for maximum value of an objective function (biodiesel production).

3. Materials and Devices

This study includes many materials and devices used in the experimental work in addition to experimental procedure. In the table (1), shows the materials, and table (2), shows the devices that used in the experimental work.

Table 1: Materials used in experimental work

No.	Materials	Specifications	Value
1.		Unsaponification	2.5 Max
		Free fatty acid	0.1 % Max.
		Peroxide value	1 meg/kg Max
	Pure oil	Colour	2 Red max
		Moisture	0.1% Max
		Appearance	Clear and bright
		Odour and flavour	Odourless and bland
2.		Categories	Laboratory
		Brand	Alpha chemika
		CAS Number	64-17-5
		Molecular Formula	C_2H_6O
	Ethanol	Purity	99.5%
		Molecular Weight	46.07
		Packaging Size	1 LTR
		Form of Chemicals	Liquid
3.	Silica Gel	60 mesh PH of 10% slurry about 7, supplied from Alpha chemika company	
4.		Serial no.	AL3705
		Brand	Alpha chemika
		Wight	500gm
		Cl	Max 0.004%
		SO_4	Max 0.003%
		PO_4	Max 0.002%
	Hydroxide potassium (KOH)	SiO_2	Max 0.01%
		N	Max 0.0005%
		Pb	Max 0.001%
		Fe	Max 0.001%
		As	Max 0.0004%
		Ca	Max 0.002%
		Na	Max 0.5%

Table 2: Devices used in experimental work

No.	Equipment's	Specifications	Value
1.	Microwave	Model	CM-320
		Origin	China
		Capacity	20L
		Voltage	230V~ , 50Hz
		Input power	1150 W
		Output power	700 W
		frequency	2450 MHz
2.	Variable Voltage Transformer	Origin	Germany
		Capacity	5KVA
		Output Current	12A
		Output	0-250V AC
		Frequency	50/60Hz
		Input	220V AC
		Insulation Resistance	>5MΩ
		No. of Phases	Single-phase
3.	Digital AC multi-meter	Origin	China
		Model	SK85-2SL(5035AV)
		Input voltage	(100 – 300)V AC
		Input current	(0 – 100) A AC
		Current transformer (CT)	100A/100m A
4.	Electronic Scale	Origin	Germany
		Accurse	±0.001 gm
		Capacity	500 gm
5.	Hot Plate and Stirrer	Model	78-1 Magnetic Stirrer.
		Origin	China
		Power Supply	AC 220V/110V 50Hz/60Hz
		Speed	0-2400rpm
		Motor powers	25W
		Heating power	200W
		Temperature control range	Room temperature to 100 Celsius
		Timer	0-120min
		Two-way rotation	Yes
6.	Infrared thermometer	Model Number	HIT015501
		Origin	China
		Operating Temperature	-30 °C to 550 °C
		Response Time	Less Than 0.5 Sec
7.	Filter paper	Type is medium (102), supplied from Alpha chemika company.	

4. Experimental work

In this study, a waste cooking vegetable oil is collected from domestic consumption, and then filtered using silica gel at first to absorb the moisture present in the oil, then the oil is filtered using filter paper. The type of catalyst used was hydroxide potassium (KOH) at 0.5wt% . Ethanol is mixed with KOH first, by using a mixing device, then the purified oil is added and the mixture is re-mixed, noting that the materials are mixed according to their weight in each experiment. The transesterification reaction occurs at variance molar ratio of oil-to-ethanol varying from 1:3, 1:4, 1:5, 1:6, and 1:7 at different power 160, 320, 480, 640 and 800 (watt). Also the reaction times several 2, 4, 6, 8 and 10 (min). Samples are placed in the microwave oven after adjusting it according to the power value in each experiment. After transesterification reaction is done, the product is removed from the microwave oven, and sets for 24 hours. Two layers are formed, the top layer is the biodiesel, and the bottom is glycerin. Then, it is transferred to a separation processes by using filter paper and funnel. The product was analyzed in the laboratory and its specifications were measured and conformed to the standard specifications as shown in paragraph (5.2.1). Figure (2) illustrates biodiesel production steps. Table (3), explain transesterification reaction schedule according (CCRD)
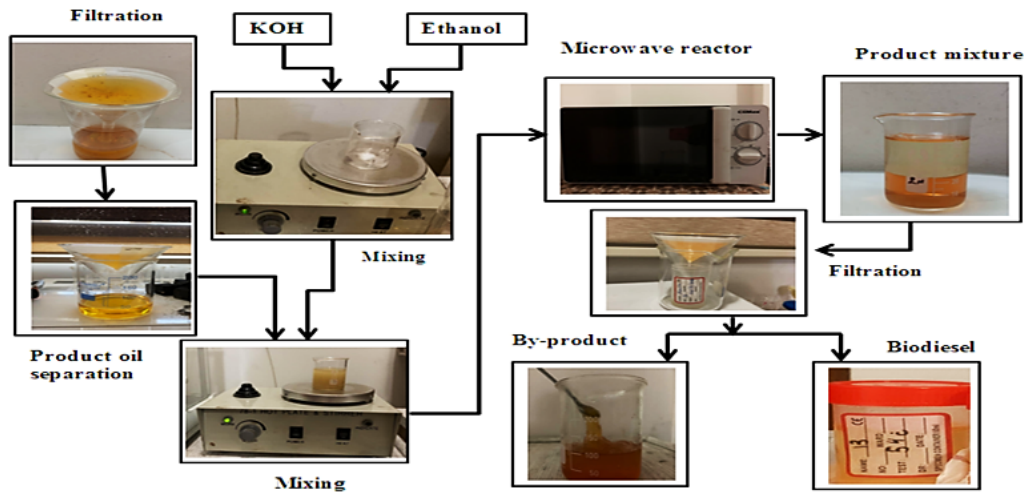
Figure 2: Biodiesel production steps

Table 3: Transesterification reaction schedule

No. of exp.	Oil/ethanol (X1) (molar ratio)	Power (X2) (watt)	Time (X3) (min)	Yield % (biodiesel)
1	1:4	320	4	70
2	1:6	320	4	75
3	1:4	640	4	35.48
4	1:4	320	8	62
5	1:6	320	8	65
6	1:4	640	8	30
7	1:6	640	4	40.2
8	1:6	640	8	35
9	1:3	480	6	45.87
10	1:7	480	6	83
11	1:5	160	6	10
12	1:5	800	6	2
13	1:5	480	2	87
14	1:5	480	10	50
15	1:5	480	6	60
16	1:5	480	6	60
17	1:5	480	6	60
18	1:5	480	6	60
19	1:5	480	6	60
20	1:5	480	6	60

5. Results and Discussion

5.1 Calculations

The amount of biodiesel production was calculated in all experiments from the following expression:

$$yield\ of\ biodiesel\ (\%) = \frac{weight\ of\ biodiesel\ yield}{weight\ of\ oil\ taken\ for\ reaction} \times 100$$

The weight of the oil after filtration process, and it is constant in all experiments (30 gm).

5.2 Optimum Condition

A program work twenty constrains (practical experiments), using a nonlinear programming technique (NLP) in solution, to obtain optimum conditions for the three variables and within the limits of the range specified in the experiments. Table (4), indicate the optimum condition to get maximum biodiesel production yield.

The properties obtained when testing the biodiesel production are representative of the physical properties shown in the Table (5).The results were within the limits of the biodiesel specification standards. The test of the biodiesel includes viscosity, density, flash point, Specific gravity, pour point, water content.

5.3 The effect of operating variables on the production of biodiesel

5.3.1 The impact of (oil/ethanol) molar ratio:

In Figure (3), explain the production plan is divided into three main areas. The first area, the curve is slowly increasing, because the amount of alcohol (ethanol) in the reaction mixture is insufficient to complete the reaction as the amount of oil in it is more. The second area is the optimum point, where the maximum biodiesel production yield was obtained d. The third area, productivity decrease in this region, due to, the amount of alcohol (ethanol) is much more than oil.

Table 4:　Optimum condition result

X1	X2 (watt)	X3(min)	Y %
1:5	480	3.5	91.63

Table 5:　Physical properties of biodiesel

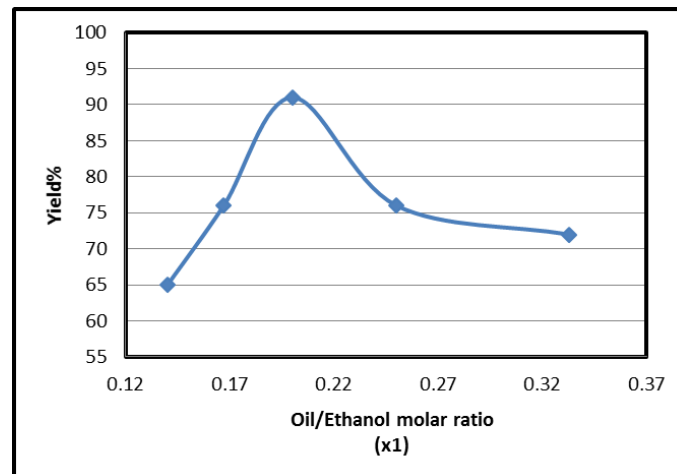| Physical properties	Produced biodiesel [present study]	Biodiesel standard [29]	Diesel standard [30]
Specific gravity at 25 °C	0.86	0.88 at 15.5°C	0.88 at 15.5°C
Kinematic viscosity (mm^2/s) at 40 °C	3.95	1.9-6	1.3-4.1
Water content max. (wt. %)	0.05	0.05max.	0.161
Cloud point	4.5	-3 to 12	-15 to 5
Pour point °C	1	-15 to 10	-35 to -15
Flash point min °C	100	100-170	80 to 60

Figure 3: The effect of (Oil/Eth.) molar ratio on biodiesel production yield at optimum condition

5.3.2 The impact of microwave oven power supply

Figure (4) illustrates the production process under optimum conditions. The curve is divided into three regions. The first area, the increase is gradual, because the rate of caloric content that affects the mixture is not sufficient to break down all the materials bonds and turn them into biodiesel. The second region has the highest production value, as most of the mixture is converted into biodiesel. The third region has a production process downturn due to the intensity of the heat rate to which the reaction mixture components are exposed. In the study [21], reported similar effect of power supply on the biodiesel production process.

5.3.3 The impact of reaction time

Figure (5), shows the highest production yield is obtained when the reaction time is equal 3.5 (min). The reaction time was sufficient to complete the reaction process and convert most of the reaction mixture into biodiesel. Then the production value decreases, due to its effect of the oil-to-ethanol molar ratio and the power supplied to the microwave oven.

The optimum conduction to get maximum biodiesel production yield (91.63) was oil/ethanol molar ratio (1:5), the power supply to microwave oven 480 (watt), the reaction time 3.5 (min) and the catalyst 0.5% wt (KOH).

5.4 The interaction effect between operating variables

5.4.1 Microwave oven power and time

If the electric power supplied to a microwave oven is high, the reaction need lower time to complete the reaction, and vice versa. Whereas, the reaction take a long time when the power supply was a little. Due to the radiation intensity of microwave oven was a little. Therefore the heat rate for reaction components need more time to arrive to optimum temperature to complete the reaction. Increasing power with increasing reaction time causes a decrease in the amount of yield due to the transformation most of the reaction mixture into a by- product. Omar and Mamdouh [31] reported similar phenomena biodiesel yield in the reaction that occurs in longer period than 24 minutes has been observed to decrease.
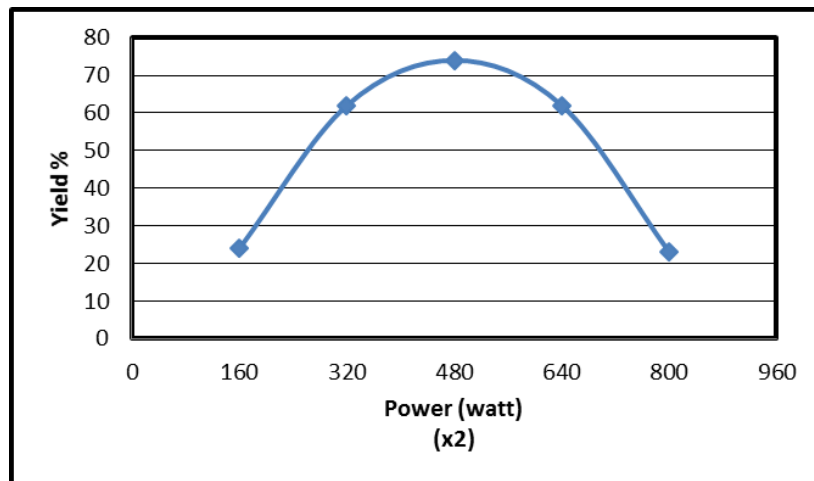
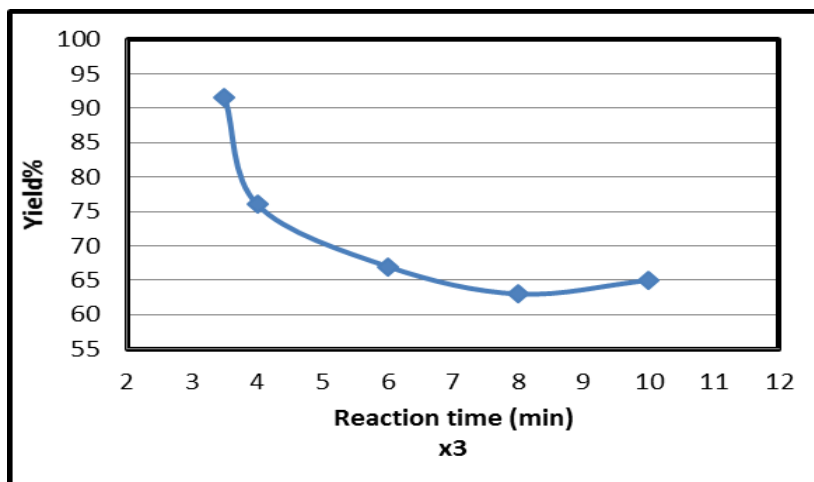**Figure 4:** The effect of power x2 (watt) on biodiesel production yield at optimum condition

Figure 5: The effect of reaction time x3 (min) on biodiesel production yield at optimum condition

5.4.2 Oil/Ethanol and microwave oven power

When the oil/ethanol molar ratio is high, it needs high microwave power to complete the reaction. The gradual increase in the oil-to-ethanol molar ratio and the microwave power supply will cause an increase in the amount of biodiesel yield. Moreover, reaching to the critical temperature of the components (ethanol), then the production of biodiesel decreases due to the evaporation of alcohol and the formation of by-products. Omar and Mamdouh [31] reported similar phenomena at high excess of methanol lowers the critical temperature of the reaction products as methanol has lower critical condition compared to the reaction mixture components. Lowering the critical temperature of the product enhance fatty acid methyl esters (FAME) decompositions and hence reducing biodiesel yield.

5.4.3 Oil/Ethanol molar ratio and reaction time

The increase in the oil/ethanol molar ratio with an increase in the reaction time is inversely proportional to the amount of biodiesel yield. The production decreases with the increase in time and the increase in the molar ratio due to the decrease in the percentage of ethanol in the sample, which evaporates as a result of the rise in the heat rate to which the sample is exposed for a long time.

5.5 The effect of one of operating variable on other

5.5.1 Oil/Ethanol with power and time reaction

In Figure (6), the power and time values were fixed at the maximum value with changing the molar ratio of oil-to-ethanol, to find the relationship between the amount of biodiesel production and the molar ratio. It is clear from the figure that the quantity of production is fluctuating, starting up at the beginning of the curve and then reaching the highest value and then decreasing, this is due to, in the beginning (0.33=1:3), the amount of the reaction mixture is small, and does not need this high power and high reaction time to complete the reaction, which leads to the transformation all most of the mixture into by-products. In point (0.2 = 1: 5), maximize production is under these conditions. At two points (0.14=1:7, 0.16=1:6) the productivity was not high due to the abundance of alcohol (ethanol) in the reaction mixture in addition to the influence of the high power and time.

Figure (7), has drawn the relationship between biodiesel production with the oil-to-ethanol molar ratio when the power and reaction time are at the lowest value with changing the molar ratio of oil-to-ethanol. The power and reaction time are so small that they are insufficient to convert all of the mixture into biodiesel, so the curve is oscillating. The highest production is at (0.2=1:5) the lowest at (0.14=1:7), because the amount of the mixture is large and requires more power and longer time.

5.5.2 Power with oil/ethanol and time reaction

In Figure (8), the relationship between the power supplied to the microwave oven with the amount of production yields a molar ratio of oil-to-ethanol and the reaction time are maximum value. At the beginning of the curve, there is a gradual increase, and this is normal because the value of the supplied power is low and therefore the heat rate is middle. The center of the curve represents the peak of production under these conditions, where the power provide to convert most of the mixture to biodiesel is sufficient to some extent, but the long reaction time affects negatively. The third part of the curve is in a slow decline due to the exposure of the mixture to higher power and a long reaction time that causes the alcohol to boil and evaporate. Figure (9), shows the effect of changing the power supplied on the amount of biodiesel production, when the molar ratio and reaction time are a minimum value. Production increases gradually at the beginning of the curve to reach the maximum. Then, it decreases as a result of increased power, which leads to an increase in the by-product and a decrease in the amount of biodiesel.
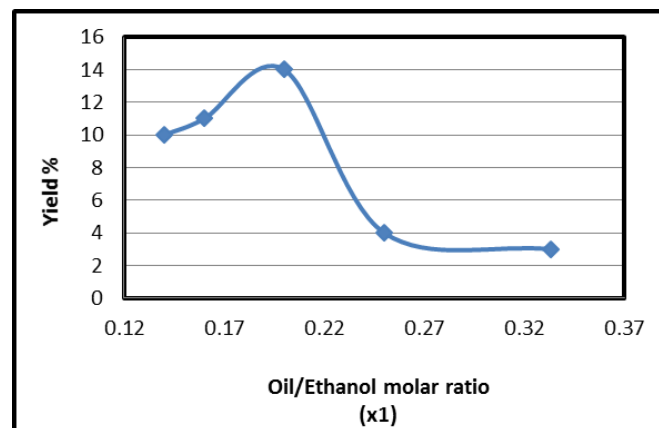

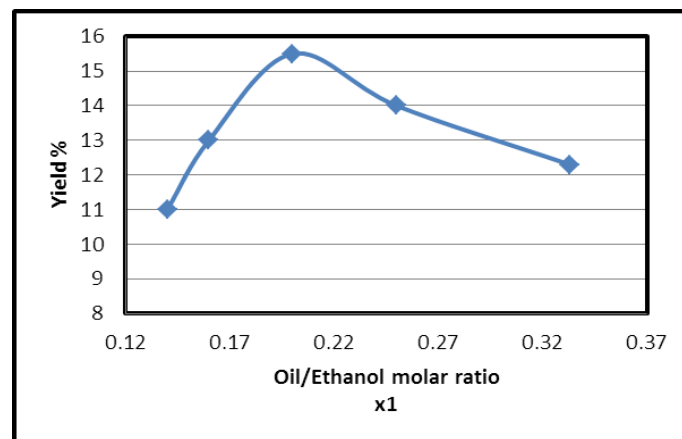**Figure 6:** The effect of (Oil/Eth.) molar ratio on biodiesel production yield at maximum value at x2=800 and x3=10

Figure 7: The effect of (Oil/Eth.) molar ratio on biodiesel production yield at minimum value at x2=160 watt and x3=2

5.5.3 Time reaction with oil/ethanol and power

Figure (10), shows the effect of changing reaction time on the amount of biodiesel production, when power and molar ratio are at the greatest value. The yield of the biodiesel decreases with the increase in the reaction time, due to the power and molarity rate are high, that causes the reaction in the reverse direction to form glycerin. In the last curve at 10 (min), the production increased due to the three reaction agents at their greatest values.

Figure (11), showing the effect of changing the reaction time on the amount of biodiesel production when the power and molar ratio are the lowest. The yield of the biodiesel decreases as the reaction time increases, due to the small amount of the mixture and the evaporation of ethanol, causing the formation of an emulsifier.
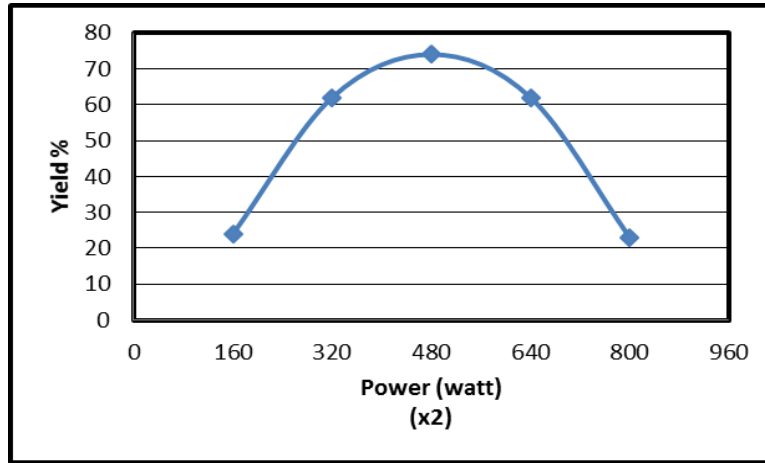

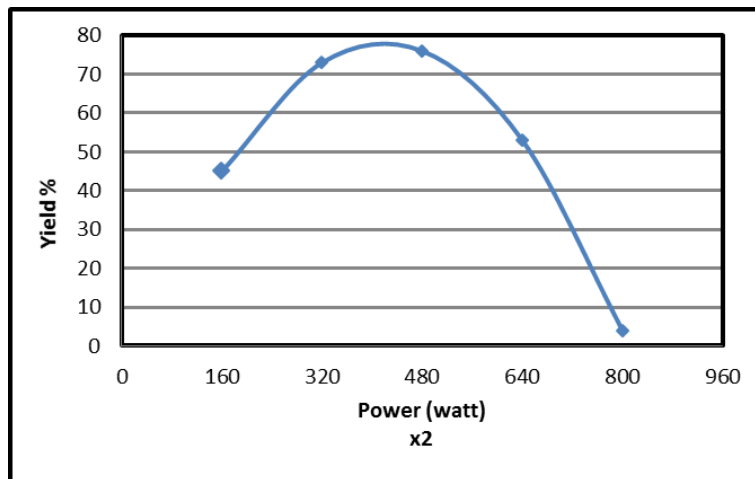**Figure 8:** Effect of power x2 on biodiesel production yield at maximum value x1=1:7 molar ratio and x3=10

Figure 9: The effect of power x2 on biodiesel production yield at minimum value x1=1:3 molar ratio and x3=2
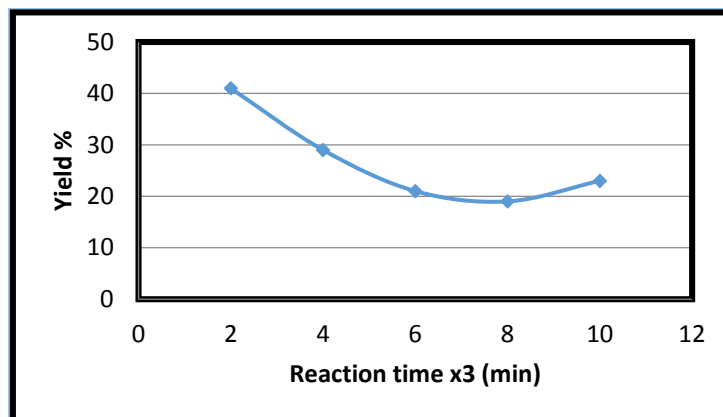

Figure 10: Effect of reaction time x3 on biodiesel production yield at maximum value of x1=1:7 molar ratio and x2=800
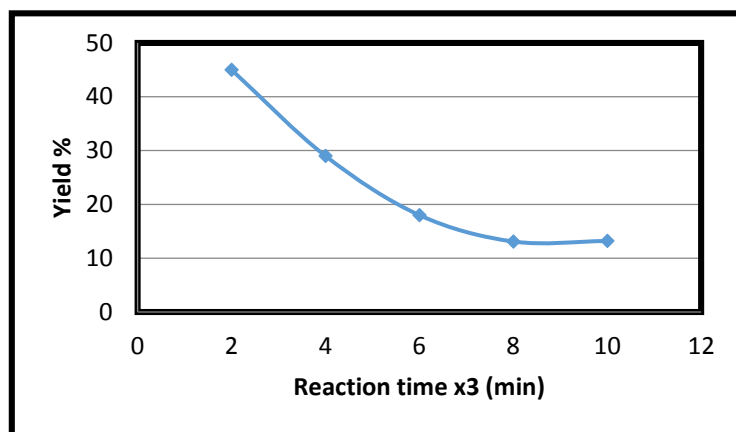
Figure 11: The effect of reaction time on biodiesel production yield at minimum value of x1=1:3 molar ratio and x2=160

6. Conclusion

Using of microwave oven technology with a multiple power source range in the process of producing biodiesel from waste cooking vegetable oil. The specifications of the resulting biodiesel are within the specifications and characteristics of diesel fuel and the standard limits. One of the most important findings of a study is, its use of ethanol, which is a renewable source of production instead of methanol, as in other studies. Biodiesel was produced from waste cooking vegetable oil that can be used as an alternative to regular fuel and thus it is considered clean renewable energy, subject to recycling processes and environmentally friendly.

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Data availability statement
The data that support the findings of this study are available on request from the corresponding author.
Conflicts of interest
The authors declare that there is no conflict of interest.

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