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The Effect of Temperature, Time, and Methanol Concentration on Enhancing the Yield of Biodiesel Production of Castor and Rapeseed Seeds via the Transesterification Process

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In Loving Memory of Late Professor Doctor ""Mohamed Refaat Hussein Mahran""

Abstract
Increased global demand for energy and fossil fuels in particular as a result of the noticeable increase in industrialization and modernization in the world However, the energy derived from this source is considered a major source of pollution. This prompted the hunt for a different energy source that produced from renewable, non-polluting raw materials, such as biodiesel, as an alternative, clean, and non-polluting fuel. Castor and Rapeseed seeds oil are considered waste in Egypt because their production exceeds the requirements of agriculture and industry. Therefore, they are considered an important and available source to produce biodiesel from their oils, and therefore they are sustainable, abundant, and cost-effective. In this paper, three primary parameters were examined in relation to the synthesis of biodiesel from each oil at 1% catalyst concentration: reaction time, reaction temperature, and methanol concentration. To speed up the conversion of free fatty acids (FFA), methanol potassium hydroxide (KOH) was added to the transesterification process as a homogenous alkaline catalyst. Exists in the oil to fatty acid methyl ester (FAME). The highest biodiesel mass yield for castor seed oil was 87.96 at the optimum condition: 20 wt.% methanol 90°C for 60 minutes. In addition, the highest biodiesel mass yield for rapeseed oil was 89.405% at the optimum condition: 20 wt.% methanol, 90°C reaction temperature for 45 minutes. A comparison study was made between ASTM biodiesel standard, commercial.
Keywords: Alternative Fuel, Castor seed, Rapeseed, Transesterification, alkaline homogeneous catalyst, Biodiesel.

1. Introduction
The increasing global population and its corresponding energy demands necessitate a shift towards renewable and sustainable energy sources [1] [2]. Industrialization and urbanization, while improving living conditions, have placed significant pressure on natural resources, particularly fossil fuels, resulting in environmental degradation. Ensuring a sustainable future and addressing the problems provided by population expansion need the development of energy sources that are both environmentally friendly and economically feasible [3]. Investigating alternative fuels, including biodiesel, as a potential substitute for traditional diesel in internal combustion engines has gained more attention in recent years. Due to its renewability and similarities to diesel oil, biodiesel has a lot of potential [4]. It is distinguished as a renewable fuel since it is produced by transesterification of vegetable or animal fats and has characteristics that are comparable to or superior to those of conventional diesel fuel [5][6]. Extensive efforts have been undertaken to investigate viable feedstock options rich in oil/lipids that have the potential for economically efficient biodiesel production. Within these options, microalgae, oil seeds, and waste from animals and fats/oil have garnered significant interest. These resources hold considerable appeal because they do not compete with food resources, making them attractive candidates for sustainable biodiesel production [7].

The castor plant is widely distributed across tropical and sub-tropical regions, displaying resilience to diverse climate conditions and requiring minimal attention. However, certain countries, notably Brazil, China, and India, have experienced a surge in dedicated cultivation of castor plants. The oil extracted from castor bean seeds offers an extensive range of applications, encompassing sectors such as plastics, cosmetics, biodiesel production, lubricants, and medicine [8]. Notably, its

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high viscosity makes it a valuable additive for enhancing the lubricating properties of diesel fuel. India leads in both production and export of castor oil, accounting for around 70% of total exports, with China (15%), Brazil (8%), and Thailand (1%), in order of precedence. Japan, Russia, and the United States are important importers. Over half of the biofuel produced in Europe comes from rapeseed, the continent's main oil crop. 79% of the feedstock crops used in Europe to produce biodiesel were rapeseed in 2008. Nearly every nation in Europe cultivates it. To ascertain the full costs and advantages of biofuels, including their net energy production efficiency and the extent of related environmental and social implications, more research is necessary[9] [10].

According to Marta M. Conceição's 2007 research, 47–49% of castor seeds are made of oil. When compared to biodiesel made from other oils, castor oil biodiesel is more economical. An alkaline transesterification reaction was used to convert castor oil into biodiesel. Methanol was added at a mass ratio of 20%, and a potassium hydroxide catalyst was dissolved in alcohol at a concentration of 1%. For thirty minutes, the resultant mixture was mixed at room temperature [11]. Glycerin was extracted and biodiesel was purified using a 30-minute decantation process, resulting in a 98% conversion rate.[12]. Additionally, In the study conducted in 2007, Umer Rashid and Farooq Anwar developed a refined process for transesterification, an alkaline-catalyzed method of producing biodiesel from rapeseed oil. The methanol/oil molar ratio (which ranges from 3:1 to 21:1), catalyst concentration (0.25-1.50%), temperature (35-65 °C), mixing intensity (180-600 rpm), and catalyst type were among the reaction factors that the researchers investigated. By employing gas chromatography to examine the biodiesel (rapeseed oil fatty acid methyl esters) at various reaction periods, the transesterification process was assessed. A methanol/oil molar ratio of 6:1, a potassium hydroxide catalyst concentration of 1.0%, a mixing intensity of 600 rpm, and a reaction temperature of 65 °C were found to give the maximum yield and quality of biodiesel. In these ideal circumstances, the biodiesel output ranged from 95 to 96% [13].

Historically, rapeseed cultivation was not documented among Egyptian farmers primarily because it was excluded as an edible oilseed in the Egyptian code. This exclusion was based on the high presence of erucic acid (C22:1), a long-chain fatty acid found in the oil of conventional rapeseed cultivars. Erucic acid ingestion has been linked to a number of detrimental health impacts in people, including the emergence of heart disease. As a result, rather than being utilized for food, conventional rapeseed oil, which has significant quantities of erucic acid, is now mostly employed in industrial applications and to make biofuels [14]. In the case of castor seeds in Egypt, the oil content ranges from 40% to 55%, with seed kernels containing about 64–71% oil. The primary fatty acid present is ricinolein acid, constituting an average of 75% of the oil. Additionally, Linoleic acid (9.7%), oleic acid (7.7%), palmitic acid (2.5%), and stearic acid (2.7%), are the constituents of castor seed oil. Despite its toxic and fatal properties if ingested, castor oil finds suitability in the cosmetics industry and for biofuel production [15].

Considering the previously supplied data, the aim of this investigation was to evaluate the feasibility of producing biodiesel from leftover oils sourced from castor and rapeseed oil. Analyzing the effects of temperature, methanol concentration, and reaction time on the mass yield of biodiesel was part of the experiment. The characteristics of the biodiesel made from each waste oil were then assessed and contrasted with those of regular biodiesel and diesel samples.

2. Materials and methods

2.1. Materials

The National Research Center of Egypt's Cultivation and Production of Medicinal and Aromatic Plant Department supplied the castor and rape seeds used in this investigation. The National Research Center in Egypt's Mechanical Engineering Department uses oil extraction. We bought 99% of the potassium hydroxide and 99.8% of the methanol from Sigma Aldrich.

2.2. Experimental Method

Transesterification, as illustrated in Figure 1, is one of the most widely used techniques in the biodiesel industry to lower oil viscosity. It occurs between vegetable oil, animal fat, or seeds and an alcohol (methanol, ethanol, or butanol) either in the presence of a homogeneous or heterogeneous catalyst.

The FFA concentration (or acid value) of the non-edible oil plays a major role in determining the base or acid catalyst to use in one-step procedures. The conventional process for making biodiesel uses a homogeneous catalyst, but it needs more water to cleanse the end product [16] [17].

Compared to the homogeneous acid-catalyzed process, the homogeneous alkali-catalyzed transesterification reaction proceeds approximately 4000 times more quickly. Due to their affordability and accessibility, alkali catalysts sodium and potassium hydroxides are more often used and favored in the industrial manufacture of biodiesel. [18]

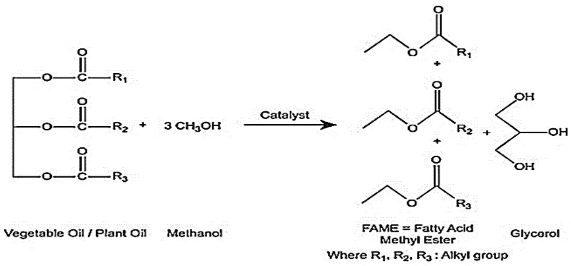

Figure 1: Transesterification reaction of triglycerides

The experiments were conducted within a 500 cm3 glass reactor placed on a magnetic stirrer featuring a hot plate, operating at standard atmospheric pressure. Initially, a 100 ml aliquot of rape and castor oils, which were then heated to a certain temperature. The heated oil was then mixed with methanol and the catalyst KOH. Various parameters were investigated to ascertain the optimal reaction conditions, encompassing reaction temperature, duration, catalyst 1% weight percentage, and methanol weight percentage.

After the reaction was finished, the mixture was separated into the glycerol and biodiesel phases using a separating funnel. Washings with hot water were applied successively to the biodiesel phase until the wash water became colorless. To eliminate residual water content, the biodiesel was desiccated on a hot plate set at 110 °C.

Equation (1) was used to determine the biodiesel yield. [19][20][21].

$$Yield, \% = \frac{mass\ of\ biodiesel, g}{mass\ of\ oi, g} \qquad \text{eq. (1)}$$

2.3. Characterization of the optimum rapeseed oil and castor biodiesel sample

The optimal biodiesel samples derived from rapeseed and castor oil underwent physicochemical property analyses. These analyses encompassed the assessment of density, pour point, flash point, and kinematic viscosity. The study was conducted to evaluate the viability of biodiesel as a potential substitute for conventional diesel fuel. A comprehensive comparison was made to contrast the properties of the biodiesel with those of standard diesel fuel.

3. Results and Discussion

3.1. G-C chromatography for caster and rapeseed oil

Gas chromatography is a widely used analytical technique that separates and analyzes compounds within a sample. It is particularly useful for determining the chemical composition for each oil. In this case, the castor and rapeseed oil samples were analyzed using gas chromatography to identify and quantify its various chemical components. This process helps in understanding the composition of the oils and is essential for optimizing the conditions required for studying temperature, reaction time, and the methanol concentration needed for the transesterification reaction in biodiesel production.

As depicted in Figure (2), the proportions of Palmitic acid, Stearic acid, Oleic acid, Linolenic acid, and α Linolenic acid were 1.26%, 1.76%, 0.77%, 1.35%, 2.61%, and 1.09% for castor oil. These results align with those obtained by Ogunniyi (2006) and Conceicao et al. (2007), who found that Castor oil contains minor amounts of Stearic acid (1%), Linoleic acid (4.2%), Linolenic acid (0.3%), Dihydroxystearic acid (0.7%), Oleic acid (3.0%), Palmitic acid (1%), and Eicosanoic acid (0.3%) (Ibrahim et al., 2020).

Figure (3) exhibits a GC chromatogram featuring known mixed GC-MS standards utilized to determine compound Retention Times (RT) and Retention Indices (RI). Additionally, (B) outlines the GC chromatograms of volatile compounds detected in the Egyptian rapeseed oil sample PRO1B1. Notably, standards such as 1-Butanol, dimethyl disulphide, butyl acetate, cyclohexane, and 2-phenyl-d5-ethanol were detected. Abundant compounds, including dimethyl disulphide, pentanal, hexanal, and 1-hexanol, were identified based on the RI of each compound.

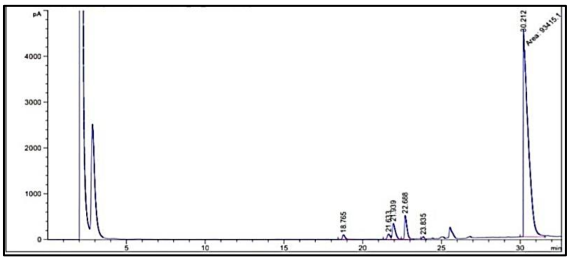

Figure (2) Gas chromatography analysis for Castor oil

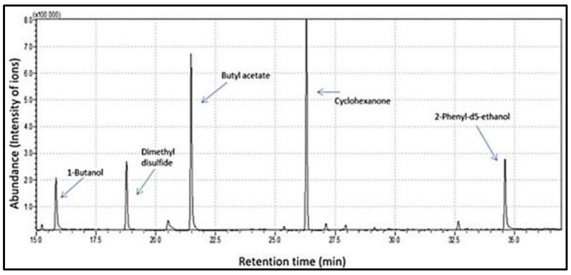

Figure (3) Gas chromatography analysis for rapeseed oil

3.2. The study of biodiesel production from Castor oil

3.2.1. Influence of temperature on biodiesel yield

Figure 4 shows how temperature affects the bulk output of biodiesel from castor oil. The study investigated various temperatures. (60, 70, 80, 90, 100, 110, and 120°C) while varying methanol concentrations (10%, 15%, 20%) during transesterification using a 1 wt.% KOH catalyst for 1 hour. Initially, the mass yield decreased at low temperatures due to the low kinetic energy of reaction molecules, then increased with rising temperature, and eventually declined. The elevation in temperature enhanced mass yield by increasing the energy of reacting molecules, improving the miscibility of polar alcohol with non-polar oil, and facilitating faster reactions[12].However, beyond 90 °C, the mass yield decreased, potentially attributed to alcohol evaporation at higher temperatures[22],[23]. Additionally, an observation was made that increasing methanol concentration augmented the biodiesel mass yield. At 90°C and 20% wt. methanol, the mass yield increased by 11.8% compared to 10% wt. methanol. The ideal mass yield was therefore found to be achieved at 90% wt. methanol and 90°C.

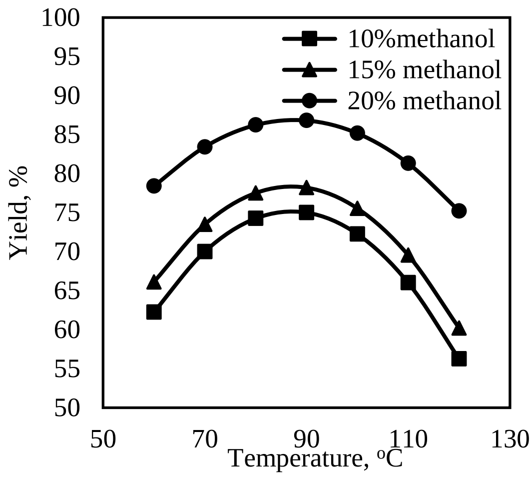

Figure 4: Temperature's effect on castor oil's mass output of biodiesel

3.2.2. Time's effect on biodiesel yield

The effect of reaction time on the mass yield of biodiesel produced from castor oil is shown in Figure 5. The study examined several time intervals (15, 30, 45, 60, and 75 minutes). while varying methanol concentrations (10%, 15%, 20%) during transesterification at the optimal temperature of 90 °C and 1% wt. KOH. The results revealed a rapid progression of the reaction, showing a significant rise in fatty acid esters' mass yield once the reaction initiated. Within the initial 60 minutes, the mass yield peaked. However, with prolonged reaction time, a decline in mass yield occurred, potentially attributed to alcohol evaporation and the reversible nature of the transesterification reaction, resulting in decreased FAME content [22]. Augmenting methanol quantity led to an increased mass yield, as more molecules engaged with oil particles, facilitating a complete reaction[12],[23],[23]. Specifically, at 20% wt. methanol and a 60-minute reaction time, the mass yield increased by 8.962% compared to 10% methanol. Consequently, the optimum mass yield was attained at 90°C with 20% wt. methanol over a 60-minute reaction period.

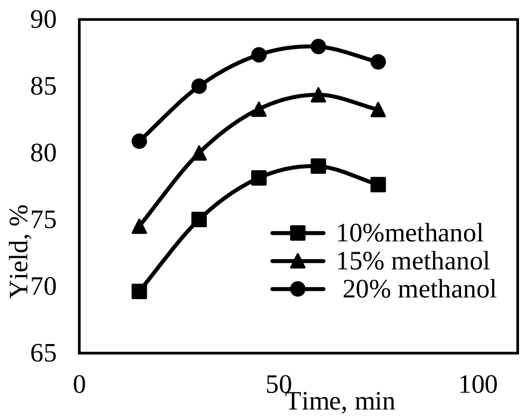

Figure 5: Time's effect on castor oil's biodiesel yield

3.3. The study of biodiesel production from Rapeseed oil

3.3.1. Influence of temperature on biodiesel yield

Figure 6 shows how temperature affects the bulk output of biodiesel from rape oil. The study investigated a range of temperatures (60, 70, 80, 90, 100, 110, and 120). °C) while varying methanol concentrations (10%, 15%, 20%) during transesterification using a 1 wt.% KOH catalyst for 1 hour. Initially, the mass yield decreased at low temperatures due to the low kinetic energy of reaction molecules, then increased with rising temperature, and eventually declined. The elevation in temperature enhanced mass yield by increasing the energy of reacting molecules, improving the miscibility of polar alcohol with non-polar oil, and facilitating faster reactions[24],[25][13]. However, beyond 90 °C, the mass yield decreased, potentially attributed to alcohol evaporation at higher temperatures[26],[27]. Additionally, an observation was made that increasing methanol concentration augmented the biodiesel mass yield. At 90°C and 20% wt. methanol, the mass yield increased by 15.9% compared to 10% wt. methanol. Consequently, the optimum mass yield was therefore found to be achieved at 90% wt. methanol and 90°C.

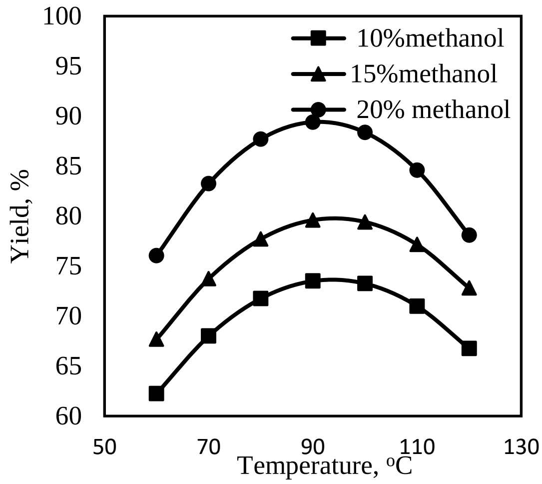

Figure 6: Temperature's effect on Rapeseed oil's biodiesel yield

3.3.2. Influence of time on biodiesel yield

The effect of reaction time on the mass yield of biodiesel produced from castor oil is shown in Figure 7. The study examined several time intervals (15, 30, 45, 60, and 75 minutes). while varying methanol concentrations (10%, 15%, 20%) during transesterification at the optimal temperature of 90 °C and 1% wt. KOH. The results revealed a rapid progression of the reaction, showing a significant rise in fatty acid esters' mass yield once the reaction initiated. Within the initial 45 minutes, the mass yield peaked. However, with prolonged reaction time, a decline in mass yield occurred, potentially attributed to alcohol evaporation and the reversible nature of the transesterification reaction, resulting in decreased FAME content[24],[25][13].Augmenting methanol quantity led to an increased mass yield, as more molecules engaged with oil particles, facilitating a complete reaction[28], [26],[27]. Specifically, at 20% wt. methanol and a 45-minute reaction time, the mass yield increased by 9.573% compared to 10% methanol. Consequently, the optimal mass yield was attained at 90°C with 20%.

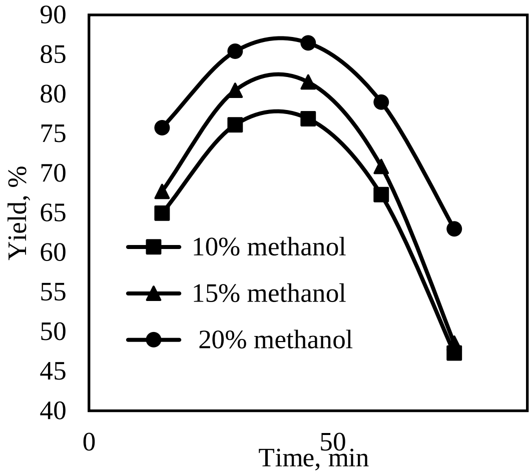

Figure 7: Time's effect on Rapeseed oil's biodiesel yield

3.3.3. Analysis of produced biodiesel

Pure biodiesel was produced at 90 °C under ideal circumstances using a catalyst consisting of 1 weight percent KOH and 20% weight methanol. For castor oil, the reaction time was set at 60 minutes, while for rape oil, it was 45 minutes. A comprehensive study of the resultant biodiesel is shown in Table 1.[29][30][22]. In compliance with the applicable ASTM standards, properties like density, kinematic viscosity, acid value, pour point, and flash point were carefully determined,[31],[32],[33].

Table 1:Comparison of physical properties between raw Castor oil, raw Rape oil, commercial diesel, produced biodiesel and its standard.

Parameter	Castor Oil	Rapeseed Oil	Commercial diesel fuel (D100)	Pure biodiesel prepared from Castor oil (B100)	Pure biodiesel prepared from Rape oil (B100)	ASTM Standards biodiesel D6751
Density at 15 °C g/ml	0.95	71.149	0.82	0.89	0.889	0.86-0.9
Viscosity at 20°C (mm^2 /s)	690	81.76	1.3-2.4	10.55	7.19	1.9-6
Pour point °C	1.8-2	-16	6	-39	-7	-5– 10
Acid value mg KOH/g	0.53	0.33	0.07	0.35	0.38	Max. 0.5
Flash point °C	162	218.2	90	161	166	Min. 100

These values were then juxtaposed with the properties of diesel fuel. Encouragingly, the results reveal that the produced biodiesel shares akin characteristics with commercial biodiesel, rendering it suitable for deployment in diesel engines. The acquired values demonstrate a commendable adherence to engine combustion standards, implying the potential of this biodiesel to fulfill the prerequisites for efficient engine performance.

4. Conclusion

This research investigated biodiesel's production from castor and rape seed oil as an alternate source of energy to limit negative effect of fossil fuel on economy and environment. Castor and Rape seeds are considered waste in Egypt as their production exceeds the requirements of agriculture and industry this makes it a promising source for biodiesel's production. The study examined the impact of temperature, duration, and alcohol percentage on the yield of FAME generated following trans-esterification. Experiments showed that for castor oil 87.96% yield was achieved at optimum conditions of: 20 wt.% methanol, 90°C for 60 minutes, and a yield of 89.405% at the optimum condition: 20 wt.% methanol, 90°C reaction temperature for 45 minutes for rape oil. Biodiesel's Physiochemical properties were evaluated and compared to those of ASTM standards. The results showed that produced biodiesel's properties achieved required properties for combustion engine.

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