

PALM OIL, COTTON OIL ESTERIFICATION, A KINETIC STUDY

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ABSTRACT

Elevated economic and environmental concerns have spurred the exploration of vegetable oils for biodiesel production, offering a greener alternative to conventional fossil diesel. With inherent fatty acid content vital for yield enhancement, this research delves into the kinetics of fatty acid esterification within a blend of (palm/cotton) oil. The investigation encompasses varied methanol molar ratios at different temperatures, Employing sulfuric acid as a catalyst with concentrations precisely designated at 5 wt% and 10 wt%. Optimal conditions include a methanol-to-oil molar ratio of 3:1, a 10% catalyst concentration, and a temperature of 60°C, resulting in a more efficient reaction time. Furthermore, This study rigorously evaluated physical and chemical properties, including acid value, FFA%, viscosity, and density, for both the oil blend and each of its components. A proposed kinetic model, employing the initial rates method, determined a rate constant of (2.3×10^{-3}) Lit/mol.min for the direct esterification reaction. Exploration of temperature's impact on the rate constant, utilizing the Arrhenius equation, revealed an activation energy (E_a) of 95.7357 kJ/mol. These comprehensive findings not only optimize the biodiesel production process but also underscore the importance of sustainable practices in shaping a cleaner and more efficient energy landscape.

KEYWORDS: Esterification, palm oil, cotton oil, kinetics, biodiesel.

1. INTRODUCTION

To satisfy its escalating energy needs, the world relies on a diverse range of sources. According to statistics, oil takes the lead, contributing 35% to global energy production, trailed by coal at 29%, and natural gas at 24%. Hydroelectric power and nuclear energy contribute 6% and 5%, respectively, while the remaining 1% is allocated to alternative energy sources. Although currently the primary global energy source, oil is non-renewable with finite reserves. Based on our present consumption rates, it is anticipated that the supplies of oil, natural gas, and coal will last roughly 45, 60, and 120 years, consecutively.^[1]

In recent years, there has been a notable increase in global fuel consumption, with the transportation sector claiming a significant 32% share of the world's fossil fuel usage.^[2] This surge has exacerbated the critical issue of global warming, primarily due to the greenhouse gas emissions originating from transportation sources. The escalating concerns surrounding the growing demand for energy resources and the resulting environmental challenges have prompted a genuine exploration of alternative fuel sources.^[3] Bio-diesel, derived from organic and renewable sources^[4], has emerged as a

promising short-term solution, especially in light of the relentless upward trend in oil prices. Bio-diesel offers numerous advantages, including cost-effectiveness, renewability, and substantially reducing pollutant emissions.^[5,6] Its compatibility with various engines and eco-friendly features can be linked to its chemical and physical characteristics^[7], closely resembling those of conventional petroleum-based fuels. This favorable alignment has led to a noticeable increase in its adoption recently. Notably, Indonesia has taken the lead as the foremost global biodiesel producer, with close contenders including the United States, Brazil, Germany, France, and Argentina. These nations employ various raw materials for biofuel production, with the US utilizing soybeans and corn, Europe relying on rapeseed, and Asia predominantly using palm oil.^[8,9]

Palm oil, abundant in saturated fatty acids, is an exceptional raw material for biodiesel production due to its superior oxidative stability.^[10] Nonetheless, a significant disadvantage is its low amount of unsaturated fatty acids, which might cause solidification at lower temperatures and impede the flow of palm oil-derived biofuels.^[7] To overcome this challenge, scientists have explored blending palm oil-based biofuels with synthetic

antioxidants to improve their characteristics.^[11] Conversely, cottonseed oil stands out due to its high unsaturated fatty acids, particularly linoleic acid. This composition enhances the low-temperature flow properties of biodiesel, making them a more advantageous option for transportation fuels than palm oil. Nevertheless, its susceptibility to oxidation limits its utilization in biodiesel production.^[12] In this context, it is evident that the flow properties of biodiesel originating from cottonseed oil outperform those derived from palm oil biodiesel. Conversely, biodiesel generated from palm oil demonstrates superior oxidation resistance compared to that obtained from cottonseed oil. Therefore, the research aimed to formulate biofuel with desired specifications by blending palm oil and cottonseed oil. This method involved adjusting the ratio of saturated fatty acids in the new oil blend, employing various parameter ratios to improve the biodiesel's characteristics promptly.

Biodiesel production involves transesterifying triglycerides or directly esterifying free fatty acids using acidic or alkaline catalysts or enzymes.^[13] Oils rich in free fatty acids are typically treated with acid catalysts, enabling direct esterification and transesterification to co-occur.^[14] The importance of direct esterification becomes apparent in enhancing biodiesel production. Conversely, alkaline catalysts are avoided when producing biodiesel from acidic oils to prevent the significant formation of soap as a byproduct alongside biodiesel production.^[15] Syria has gained recognition for its extensive cotton cultivation, particularly in the northeastern regions. With an increase in cultivated land, the country holds the potential to meet its demand for cottonseed oil, leveraging its status as a developing nation. This abundant resource can be tapped for biodiesel production. Consequently, the research focuses on utilizing cottonseed oil modified with palm oil to generate biodiesel under optimal conditions. This involves examining the reaction kinetics of biodiesel synthesis and varying parameters such as the alcohol-to-oil ratio, catalyst quantity, and temperature. The objective is to understand their respective impacts on the reaction rate, ultimately improving the overall process.

This research investigated the direct esterification kinetics of fatty acids with triglycerides in a blend of oils, specifically a blend comprising 50% palm oil and 50% cottonseed oil. Various quantities of methanol were applied, along with different proportions of sulfuric acid serving as a catalyst, and the reactions were conducted for varying durations. As highlighted in prior studies, sulfuric acid displayed notable catalytic effectiveness in direct esterification reactions, distinguishing itself from numerous organic and inorganic acids.^[16]

2. EXPERIMENTAL

2.1. Materials And Apparatus

Cottonseed and palm oil were purchased from the local market in Homs, Syria. Methanol, characterized by a 99%

purity, was obtained from MERCK, while sulfuric acid (H₂SO₄), functioning as a catalyst with a purity of 97%, was sourced from CARLO ERBA. EKA supplied potassium hydroxide with an 85.5% purity level, and the titration indicator phenolphthalein, with a purity of 99%, was acquired from Titan.

2.2. Preparation of biodiesel by Fatty Acid Esterification in a Blend of Palm and Cottonseed Oils

A homogeneous mixture of palm and cotton oils was prepared by combining them in a 50:50 volumetric ratio. The blend underwent stirring with a magnetic stirrer for two hours while being heated to 100°C to eliminate water content. Following this process, the oil blend's (cotton-palm) composition was determined using gas chromatography.^[17]

The direct esterification reaction occurred in a 250 ml double-necked flask, with one neck connected to a reflux condenser and the other left open to add chemicals and sample withdrawal. The flask containing only the oil was positioned in a water bath and heated to the desired temperature. The mixture of methanol and the catalyst was then introduced. Due to the limited miscibility of methanol and oil, magnetic stirring was applied at a constant speed of 600 rpm throughout the reaction period to ensure thorough mixing.^[18] Samples measuring 2 ml were collected at various intervals until the total reaction time reached 120 mins. After each sampling, the portion was rinsed with water to stop the reaction and aid in separation. The samples were left to settle for approximately 30 minutes to facilitate separation. Following this, a small sample from the upper layer (the oil layer) was collected for titration. Prior to titration, the sample is diluted by adding it to a mixture of ethyl ether and ethanol (50% volume). The phenolphthalein indicator is then introduced into the mixture for titration with a solution of KOH at a concentration of 0.02 N. The Acid Value (AV) is titrated with a 0.02 N KOH solution following the standard methods (AOCS Ca 5a-40). The volume of KOH required for the appearance of a pink color in the sample is recorded. The Acid Value (AV) represents the number of milligrams of potassium hydroxide necessary to eliminate the free fatty acids in 1 gram of the fat substance.^[19]

$$A_v = \frac{56.1 \times M \times (V_s - V_b)}{w} \quad (1)$$

Where: VS: The volume of KOH required for sample titration (ml).

VB: The volume of KOH required for blank titration (ml).

M: Molarity of the KOH solution. w: Weight of the sample (g).

56.1: The molar mass of potassium hydroxide (g/mol).

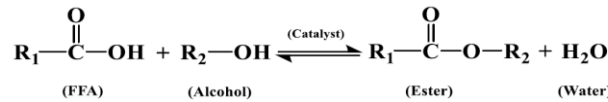
The percentage of free fatty acids is determined according to the equation

$$\text{FFA \%} = \frac{N.V}{w} \times 28.2 \quad (2)$$

The conversion ratio from the equation is calculated as

$$P\% = \frac{A_{V_0} - A_v}{A_{V_0}} \times 100 \quad (3)$$

The reaction was conducted using varying quantities of methanol, different amounts of sulfuric acid serving as a catalyst, and at different temperature levels.^[15,20]



The kinetic model for the examined direct esterification reaction was established based on the following assumptions

1. The kinetics of non-catalytic reactions are negligible compared to catalytic reactions.
2. Direct esterification reactions are considered irreversible heterogeneous processes, with the applied experimental conditions controlling the reaction kinetics.
3. Chemical reactions take place within the oil phase.^[22]

The relationship can express the kinetic equation

$$-\frac{dC_F}{dt} = k_1 C_F C_m - k_2 C_E C_w \quad (4)$$

Where C_F : represents the concentration of free fatty acids (FFA). C_E : The concentration of formed esters. C_w : The concentration of water formed during the reaction. C_m : The concentration of methanol. k_1, k_2 : The reaction rates for both the forward and reverse processes.

$C_F = C_{F_0} - x$ (Where x : the concentration of consumed free fatty acids, C_F : The concentration of free fatty acids (FFA) at a specific time (t). C_{F_0} : The initial concentration of free fatty acids).

$C_m = C_{m_0} - x$ (Where C_m : The concentration of methanol. C_{m_0} : The initial concentration of methanol).

$$M = \frac{C_{m_0}}{C_{F_0}} \rightarrow C_{m_0} = M C_{F_0},$$

$$\frac{dx}{dt} = k_1 (C_{F_0} - x)(C_{m_0} - x) - k_2 x^2$$

2.3. Kinetic Model

A kinetic model has been proposed for direct and reverse reactions to elucidate the kinetics involved in the direct esterification of free fatty acids within an oil blend. This process employs concentrated sulfuric acid as a catalyst in the investigated reaction system. According to the following equation, the direct acidic esterification reaction transforms free fatty acids.^[21]

$$\frac{dx}{dt} = k_1 (C_{F_0} - x) - (M C_{F_0} - x) - k_2 x^2 \quad (5)$$

Conversion ratio: $X_F = \frac{C_{F_0} - C_F}{C_{F_0}}$, where X_F : The conversion ratio of free fatty acids (FFA).

$$C_{F_0} X_F = C_{F_0} - C_F = x \quad (6)$$

And then, by differentiation

$$C_{F_0} dX_F = -dC_F = dx \quad (7)$$

And by substituting this into equation (5)

$$C_{F_0} \frac{dX_F}{dt} = k_1 (C_{F_0} - X_F C_{F_0})(M C_{F_0} - X_F C_{F_0}) - k_2 C_{F_0}^2 X_F^2$$

$$C_{F_0} \frac{dX_F}{dt} = k_1 C_{F_0} (1 - X_F) C_{F_0} (M - X_F) - k_2 C_{F_0}^2 X_F^2$$

$$C_{F_0} \frac{dX_F}{dt} = k_1 C_{F_0}^2 (1 - X_F)(M - X_F) - k_2 C_{F_0}^2 X_F^2 \quad (8)$$

3. RESULTS AND DISCUSSION

3.1. Studying Some Physical and Chemical Properties of the Prepared Oil blend

Accurate density and viscosity measurements were conducted for palm oil (PO) and cottonseed oil (CSO), followed by a meticulous comparison of the density and viscosity characteristics of the oil blend derived from these sources.^[23] Notably, the oil blend exhibited superior viscosity properties compared to each oil. Furthermore, a comprehensive analysis was undertaken to determine the acid value and the percentage of free fatty acids in palm oil, cottonseed oil, and the oil blend. Table (1) demonstrates the notable increase in the percentage of free fatty acids within the oil blend.

Table 1: Some physical and chemical properties of palm oil, cottonseed oil, and oil blend.

Sample	A_v mgKOH/g	FFA%	Density (gr/cm ³)	Viscosity (St)
CSO	0.554	0.277	0.910	0.4921
PO	0.592	0.296	0.884	0.5532
Mix	1.142	0.5741	0.897	0.6671

As Table (2) illustrates, the fatty acid content of the oil blend was determined using a Gas Chromatography analysis.

Table 2: Fatty Acid Composition of the Oil Blend.

Fatty Acid Name	Structure	Percent %
Myristic Acid	(C14 :0)	0.6
Palmitic Acid	(C16 :0)	24.34
Stearic Acid	(C18 :0)	10.03
Oleic Acid	(C18 :1)	45.12
Linoleic Acid	(C18 :2)	17.37
Other components	---	2.54

3.2. The Impact of Different Parameters on Reaction Rate

The study examined the impact of different concentrations of a sulfuric acid catalyst (5% and 10%), varying temperatures (50-55-60°C), and different oil-to-

methanol molar ratios of 1:3, 1:4, 1:5, as well as 1:6, affecting the reaction of esterifying oil blend with methanol. The experiment was conducted under a constant stirring rate; the results are presented in Table (3).

Table 3: Impact of various factors on the reaction time.

Sulfuric acid concentration(%)	Methanol to oil ratio	Reaction time (min) at 50°C	Reaction time (min) at 55°C	Reaction time (min) at 60°C
5%	1:3	50 min	50 min	40 min
5%	1:4	60 min	70 min	40 min
5%	1:5	60 min	70 min	50 min
5%	1:6	60 min	80 min	50 min
10%	1:3	40 min	40 min	30 min
10%	1:4	60 min	50 min	30 min
10%	1:5	60 min	50 min	50 min
10%	1:6	60 min	50 min	50 min

Numerous studies have emphasized the proficient role of concentrated sulfuric acid as a catalyst in converting free fatty acids within oils.^[24] With the oil mixture revealing a free fatty acid content surpassing 0.5%, as outlined in Table (1), sulfuric acid stands out as an appropriate catalyst for the direct esterification reaction.

The previous findings indicate a consistent reduction in reaction time with increased sulfuric acid concentration from 5% to 10%. This can be attributed to the improved catalyst efficiency, as higher concentration enhances the interactive forces between the reactants. Furthermore, an escalation in catalyst concentration may influence the chemical reaction equilibrium, thereby improving the reaction's selectivity and increasing the production of desired products. Additionally, the results highlight a connection between the change in reaction time and variations in the methanol-to-oil ratio. As the methanol-to-oil ratio rises, the reaction time extends at all examined temperatures, signifying a slowdown in the esterification reaction. Consequently, the reaction reaches equilibrium more quickly when the methanol-to-oil ratio is low. This phenomenon is attributed to the dual role of alcohol, functioning not only as a reactant but also as a solvent. When an excess of alcohol is employed, its impact as a solvent becomes more pronounced, resulting in a reduced reaction rate and a slower overall reaction.^[7]

Regarding the temperature impact, esterification reactions are identified as endothermic, consistent with various referenced studies.^[25] Notably, the reaction time decreases with increasing temperature, as heightened temperatures augment the kinetic energy of reacting molecules, increasing the likelihood of effective collisions. Additionally, the temperature rise enhances the probability of surpassing the activation energy barrier required for the reaction, thus accelerating the reaction rate.^[26] The optimal conditions for achieving a rapid reaction in the shortest time were observed at a temperature of 60°C, a methanol-to-oil ratio of 1:3, and a catalyst concentration of 10%. Further kinetics studies will be undertaken under these specific conditions.

3.3. Applying the Kinetic Model

3.3.1. Reaction Rate Constants

Utilizing the initial rates method allows for direct comparisons with the laws of differential rates. This approach offers two advantages: Firstly, there is no need to integrate the rate law, and secondly, it remains unaffected by reverse or side reactions. The reaction is monitored in small time intervals at the onset of the reaction.^[27]

The direct rate constant, k_1 can be calculated using initial rates at $t=0$; here the initial concentrations of the

resulting substances are zero, represented as C_E, C_W . Equation (5) becomes

$$v_0 = -\frac{dC_F}{dt}_{t=0} = k_1 C_{F_0} C_{m_0} = k_1 C_{F_0} M C_{F_0} = k_1 C_{F_0}^2 M$$

From Equation (8), we deduce:

$$v_0 = C_{F_0} \frac{dX_F}{dt} = k_1 C_{F_0}^2 M \tag{10}$$

$$v_0 = k_1 C_{F_0} M \tag{11}$$

The initial rates are determined using Equation (11).

A graph was created to show the relationship between $\frac{dX_F}{dt}$ and MC_{F_0} . This graph demonstrates how the initial ratio of methanol to free fatty acids influences the process. The graph was created using a 10% catalyst concentration at a temperature of 60°C. The value of k_1 was derived from the slope of the plot as shown in Fig.1, and the k_1 value was 2.3×10^{-2} lit/(mol.min).

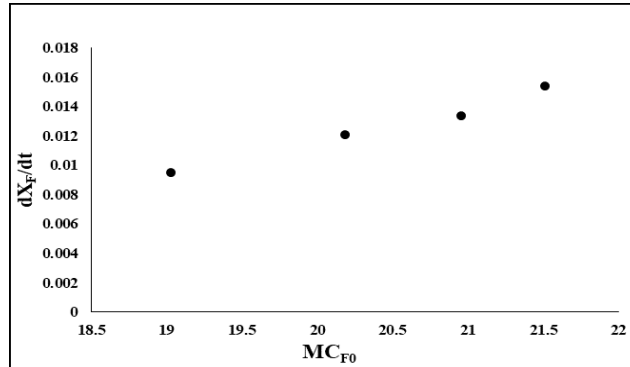


Fig. 1: Initial rate vs Methanol-FFA molar ratio.

3.3.2. Reaction Rate Constants

To investigate the effect of temperature on the reaction rate constant, we utilized the Arrhenius equation

$$k = Ae^{-E_a/RT} \tag{12}$$

Or

$$\ln k = \frac{-E_a}{RT} + \ln A \tag{13}$$

Using a similar approach employed for determining the rate constant at 60°C, we calculated the rate constants at temperatures of 50°C and 55°C, recording the results in Table (4).

Table 4: The reaction rate constants for the esterification of free fatty acids in the oil blend at controlled temperatures.

T (°C)	T(K)	1/T (K ⁻¹)	k ₁ (lit/mol.min)	-lnk ₁
50	323	0.00309	8×10^{-4}	7.1309
55	328	0.00304	1.1×10^{-3}	6.8124
60	333	0.00300	2.3×10^{-3}	6.0748

In Fig.2, the graph illustrates the calculation of activation energy (Ea) by plotting the changes in -lnk1 against the reciprocal of temperature. The resulting straight line,

with a slope (m) equal to (Ea/R) as per the Arrhenius equation, allows us to deduce that Ea is 95.73571 kJ/mol.

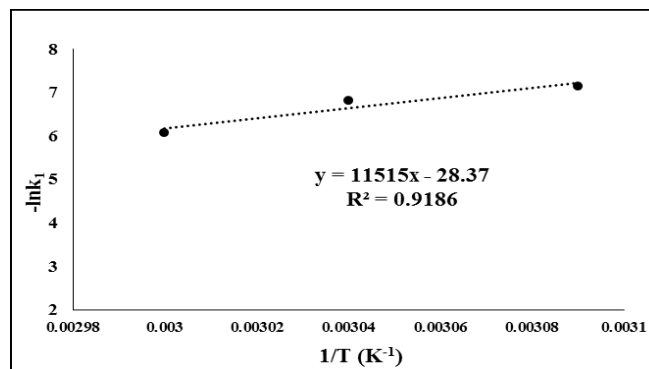


Fig. 2: Effect of temperature on the reaction rate constants.

4. CONCLUSION

Our study on esterification kinetics in a (palm/cotton) oil blend for biodiesel production yielded vital findings. Optimized condition 3:1 methanol-to-oil ratio, 10% catalyst, 60°C significantly enhanced reaction rate. Advancing our understanding of biodiesel synthesis, the research underscores vegetable oils as a sustainable diesel alternative. Rigorous analysis, revealing a rate constant (2.3×10^{-3} Lit/mol.min) and an activation energy (E_a) of 95.7357 kJ/mol, guides future refinements. These insights pave the way to a cleaner, and more efficient energy landscape. The research not only contributes to biodiesel kinetics but aligns with global sustainability objectives.

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