Ketalization of Glycerol and Acetone to Solketal: Effect of Temperature, Concentration & Mathematical Model

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Abstract
Solketal is a viable method for using glycerol, a by-product of biodiesel production. This study aims to identify the optimal operating parameters for solketal compounds generated from the glycerol ketalization reaction with acetone by using mathematical models that effectively forecast an appropriate framework for this process. This research consists of three critical phases: the ketalization reaction of glycerol with acetone, the characterization of the result solketal products, and the ketalization reaction utilizing the Amberlite IR 120 Na catalyst. The process begins by introducing glycerol and acetone in a mole ratio of 1:3, followed by mechanical Stirring at 500 rpm. The temperature is regulated using a water bath to maintain a constant reaction temperature under specified conditions of 20 °C, 120 °C, 150 °C, and 180 °C, with catalyst masses of 1%, 3%, 5%, and 7%. The mathematical model used is of exponential and polynomial order 2. The findings indicated that the optimal glycerol conversion of 46.01% was attained at 50 °C, using a 5% catalyst concentration throughout a reaction duration of 120 minutes. Second-order polynomial regression is the most appropriate mathematical model to represent this process.

Keywords: Glycerol ketalization; Mathematical model; Solketal

1. Introduction

Thanks to its significant natural resources, Indonesia is one of the largest palm oil producers globally . Biodiesel derived from palm oil is experiencing considerable growth, coinciding with Indonesia's initiative to implement B30 as a transportation fuel to substitute fossil fuels [1][2]. Biodiesel is an ester derived from triglycerides and alkyl, produced via a transesterification reaction, commonly called FAME (Fatty Acid Methyl Ester). This process involves transesterifying oils or fats with alcohol compounds in an alkaline medium [3][4]. This fuel is considered an environmentally friendly alternative and can be derived from biomass [2][5][6]. The increasing demand for renewable fuels, coupled with the exhaustion of fossil fuels and environmental concerns associated with greenhouse gas emissions [7][8][9], has led to the growing utilization of Biodiesel as an alternative fuel for diesel engines, which generates reduced CO2 emissions [10]. Researchers have concentrated on investigating renewable energy sources for future energy requirements. Biodiesel derived from biomass has been recognized as a viable renewable resource [11]; biomass can generate bio-additives and various chemicals [12].

Meanwhile, the rising global demand for biodiesel production is leading to an increased supply of glycerol as a by-product of the biodiesel process [13-16]. Consequently, it is essential to implement measures to utilize glycerol as a by-product of Biodiesel. One is solketal, which can operate as a bio-additive in diesel engines. Solketal is created from the ketalization of glycerol and acetone using particular catalysts [5][14]. Solketal formed from the ketalization of the two components is thermodynamically considerably more stable [5] and has vast benefits as a fuel additive, ecological solvent and plasticizer [17]. In addition, the price of solketal is relatively high on the market.

Solketal improves cold flow characteristics, boosts octane number, improves combustion, reduces viscosity and regulates pollutants. It is more environmentally friendly and sustainable for the ecosystem than other gasoline additives,

notably fish toxicity testing [18]. Mahreni et al. (2022) conducted previous research on solketal produced from glycerol, acetone and ethanol using Woifram Acid Phosphotungistic catalyst; based on the research obtained, the optimal solketal time is 4 hours with the highest solketal conversion, namely at a reaction temperature of 60 °C, catalyst percentage 2%, reactant mole ratio 1: 3: 1 which is 66.7% [19]. In contrast to earlier studies, this study focuses on the use of catalysts that are more sustainable, widely accessible and the maintenance procedure is much simpler. Where, Amberlite IR 120 Na catalyst has parameters as a solid catalyst that has a particle size of 300-1200 μm, density ± 1.28 g/mL, stable over a broad pH range and resistant to high temperatures. According to its specs, this catalyst is deemed extremely suited in the ketalisation process. Additionally, this work seeks to mimic this process using the suggested mathematical model so that it may describe the dynamics of the ketalisation system to make solketal. This study aims to determine the ideal operating conditions for solketal products created from the reaction between glycerol ketalization and acetone.

2. Methods

This study comprises three crucial stages, namely the reaction of glycerol ketalization with acetone, the characterization of the resulting solketal product, and the development of a mathematical equation model that is highly effective in forecasting an appropriate model for this process. Prior research has examined the characterization of solketal compounds in this process [20]. The reaction of glycerol and acetone ketalization was carried out using a variety of instruments consisting of a glass reactor of three necks of the round-bottom flask, thermometer, condenser, erlenmeyer, sampler, and water bath as shown in Figure 1. At the same time, the primary materials used are Glycerol (Brataco, Indonesia), Acetone (Merck, Germany), Amberlite IR 120 Na Catalyst (Rohm & Haas, USA), Chloroform (Merck, Germany), Sodium Thiosulfate (Merck, Germany), 0.1 N Periodic Acid Solution, 10% Potassium Iodide Solution, 1% Starch Indicator, Potassium Dichromate (Merck, Germany), 37% Hydrochloric Acid (Merck, Germany), and Aquades.

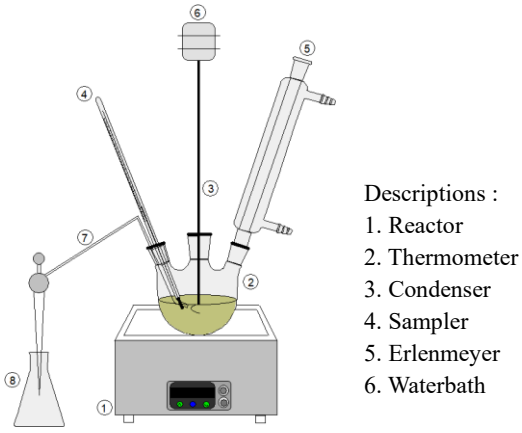

Figure 1. Glycerol ketalization process apparatus [6], [20].

2.1. Glycerol Ketalization Reaction with Acetone to Solketal

The ketalization process of glycerol with acetone using an Amberlite IR 120 Na catalyst begins by introducing glycerol and acetone with a mole ratio of 1:3. Stirring is done using a mechanical stirrer at a speed of 500 rpm, and the water bath temperature regulator is controlled so that the reaction temperature is obtained and steady. The catalyst is added according to the amount of particular needs once both reactants have reached the necessary homogeneity and reaction temperature. After that, samples were taken for the initial reaction time (t = 0), and further samples were taken every 30 minutes for 3 hours. Furthermore, the free glycerol content per time (Gbn) and the initial free glycerol level (Gb0) were examined. The operating conditions employed were temperatures of 20°C, 120°C, 150°C, and 180°C and catalyst masses of 1%, 3%, 5%, and 7%. Past operation conditions have also been investigated earlier with varied temperature variations at 40 °C, 45 °C, 50 °C, 55 °C and 60 °C [6]. However, the expected results in previous studies using these operating conditions have not been achieved.

2.2. *Analysis Glycerol Levels*

Quantitative analysis was conducted to determine the glycerol content released before (Gb0) and after reaction (Gbn). The determination of glycerol content was analysed by iodometric method. This stage begins with dissolving the sample in the ketalisation process as a total of 1 gram in 100 mL. Then, 50 mL of chloroform was added to 1 gram of the previously diluted sample, diluted again to 500 mL and left for 24 hours. Before analysis, the test solution in the previous dilution process was taken as much as 250 mL and added 2 mL of potassium iodide solution and titrated using 0.01 N sodium thiosulfate solution and starch solution as an indicator [6]. Before determining the glycerol conversion, first calculate the glycerol content released as in the following equation:

$$\boldsymbol{Gb}\ (\%) = \frac{\mathbf{2{,}302}\ (\boldsymbol{V_{tb}} - \boldsymbol{V_{ts}}) \times \boldsymbol{N} \times \mathbf{450} \times \mathbf{100}}{\boldsymbol{W_2} \times \mathbf{50} \times \left(\frac{\boldsymbol{W_1}}{\boldsymbol{\rho_g}}\right)} \tag{1}$$

To calculate the glycerol conversion rate, use the following equation:

$$\boldsymbol{X_G} = \frac{\boldsymbol{Gb_0} - \boldsymbol{Gb_s}}{\boldsymbol{Gb_0}} \times \mathbf{100}\ \% \tag{2}$$

Where, V_{tb} refers to the titration volume for the sample (mL), V_{ts} represents the titration volume for the blank sample (mL), N indicates the normality of the sodium thiosulphate solution (N), W_2 refers to the weight of the analyzed sample (grams), W_1 equates to the initial weight of the reconstituted sample (grams), and ρ_g is the density of glycerol (g/mL).

2.3. Mathematical Models

This work used a statistical analytic approach, such as exponential regression and 2nd order polynomials, which may forecast the proper values and variables under specified operating conditions [21]. In addition, the objective of this approach is to be the basis for doing simulations, optimizations, or more extensive sensitivity studies, typically used to describe the dynamics of a system. The mathematical equation model proposed is,

a. Exponential regression.

$$y = y_0 + A.exp^{(R_0.x)} \tag{3}$$

Where y is the glycerol conversion, y_0 is the glycerol conversion at a time approaching zero, A is the amplitude, R_0 is the exponential rate of change, and x is the time (minutes).

b. 2nd Order Polynomial Regression

$$y = ax^2 + bx + c \tag{4}$$

Where y is the glycerol conversion, x is the time (minutes), and a, b, and c are the coefficients of the regression polynomial. Furthermore, The OriginLab fits the equation and determines the R^2 value.

3. Results and Discussion

This study evaluated parameters to identify the most appropriate operating conditions to produce high glycerol conversion values. The parameters observed include reaction temperature and catalyst concentration.

3.1. Effect of Temperature

One characteristic that can affect a reaction's pace is the reaction temperature [6]. If there is an increase in the reaction temperature in a system, the reaction will occur faster due to increased collisions between molecules. The Arrhenius hypothesis posits that the velocity of a chemical reaction escalates exponentially with temperature, as a greater number of reactant molecules acquire enough energy to surmount the activation energy barrier. As temperature

raises, the kinetic energy of molecules escalates, leading to a bigger proportion of collisions possessing energy equal to or above the activation energy, thereby greatly enhancing the reaction rate.

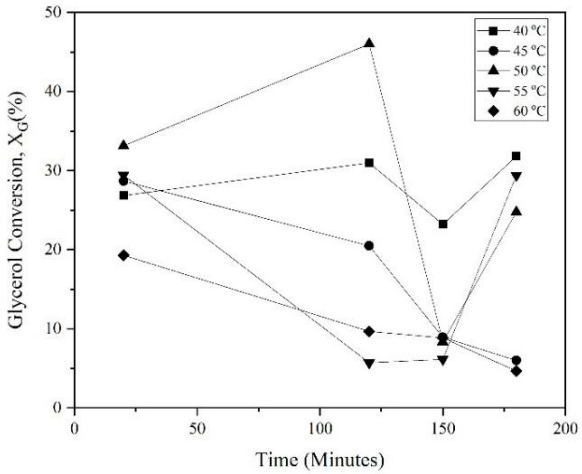

Figure 1. Effect of Reaction Temperature on Glycerol Conversion

The reaction proceeds faster and the conversion of glycerol produced is greater than the reactant obtained [6], [16] The reaction temperature investigated was 40°C - 60°C with a reaction time of 3 hours, a catalyst concentration of 5%, and sampling time at intervals of 20 minutes, 120 minutes, 150 minutes, and 180 minutes with a ratio of glycerol and acetone of 1:3.

As seen in Figure 2, there was an increase in glycerol conversion at several temperature conditions such as at temperatures of 40 oC and 50 ºC with a reaction time of 120 minutes, while at temperatures of 45 ºC, 55 ºC, 60 ºC it tended to decrease. Each temperature showed a varying pattern along with increasing reaction time. The fluctuating pattern between glycerol conversion and time shows that glycerol conversion is not always linear with its reaction time. Conversion tends to be stable or decreases only at lower temperatures around 40-45 °C. Meanwhile, at high temperatures, glycerol conversion increased at the beginning of the reaction but then reduced quite drastically. There is a large temperature drop caused by the thermal degradation of glycerol after reaching a certain temperature. This fluctuating data trend may be due to the decomposition of products or by-products formed when the reaction temperature has been reached. The highest glycerol conversion was obtained at a temperature of 50 ºC with a reaction time of 120 minutes, which was 46.01%, while the lowest yield was at a temperature of 60 ºC with a reaction time of 180 minutes, which was 4.67%.

3.2. Effect of Catalyst Concentration

The selection of the type and amount of catalyst utilized can also alter the conditions of the chemical reaction. Catalysts work to accelerate the reaction rate by reducing the activation energy without changing the equilibrium of a reaction [6]. The selection of catalyst type is highly related to temperature and other operating variables.

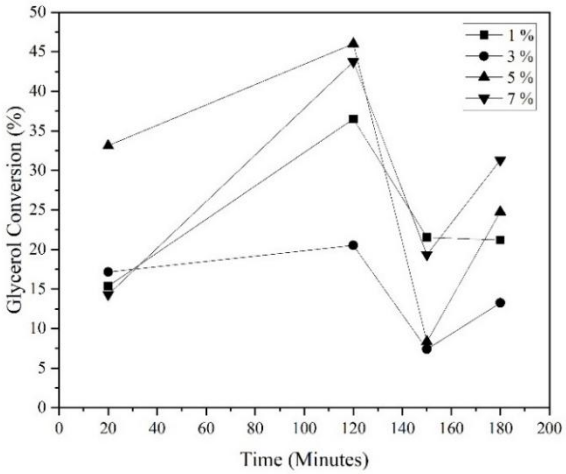

Figure 2. Effect of Catalyst Concentration on Glycerol Conversion

The catalyst mass concentrations investigated were 1%, 3%, 5%, and 7% of the glycerol mass used, with a stirring speed of 500 rpm and a reaction temperature of 50°C. The goal of employing various catalyst concentration changes is to discover the optimum amount in the glycerol ketalization reaction with acetone to become solketal and determine the maximum reaction conversion to establish equilibrium.

Overall, glycerol conversion increased at 120 minutes for all variations of mass concentrations, as shown in Figure 3. However, there was a reasonably drastic decrease in conversion after 120 minutes which can be seen at a catalyst concentration of 1%, 3%, 5%, and 7%. Even though there was a decline in conversion at 150 minutes, at 180 minutes, there was an increase in concentration again. Under certain circumstances, variations in the glycerol conversion value at a catalyst mass of 7% may signal the possibility of saturation or adverse reactions. The highest glycerol conversion was obtained at 46.01%, with a reaction time of 120 minutes and a catalyst mass of 5%. The lowest conversion was obtained at 3.49%, with a reaction time of 150 minutes and a catalyst mass of 3%. The average conversion value declined at 150 minutes, potentially due to side reactions produced, catalyst deactivation, or substrate limits, causing the conversion value at each concentration to decrease. Therefore, it can be seen that the catalyst mass and reaction time have a significant effect on the glycerol conversion obtained. Increasing the amount of catalyst will raise the reaction rate, because when increasing the mass of catalyst there will be more active sites for transesterification reaction in the system [21].

3.3. The Exponential Regression Equation

In this study, a mathematical prediction was made of the glycerol conversion produced per unit of time. The present research is to see the relationship between factors such as temperature, reaction time and catalyst concentration on the glycerol conversion value. The exponential function is a function whose independent variable is the power of a constant [22]. The function usually describes growth or decay that occurs continuously with a steady percentage change [23][24]. The results of the experimental data obtained were then modelled using equation (1) to get the results as in Table 1.

From the data generated on the temperature variable, it can be observed that practically all y_0 values created from the calculation are relatively large, even at a temperature of 60°C, reaching 643.8. The resulting data indicates that at the beginning of the process (t = 0), most of the glycerol has been converted before the measurement begins, in addition to the possibility that the reaction system does not start from time zero. Meanwhile, the total rate constant value (R_o) generated is very small, even harmful; this can be interpreted as the slow response rate at each temperature. The R^2 value on the temperature variable is below 0.5. Only at 45°C and 60°C do R^2 values approach 1. The results obtained imply that the proposed exponential equation has a match on specific data patterns alone but does not match as a whole.

Meanwhile, from the data generated on the catalyst concentration variable, it can be seen that the y_0 value is quite significant, and the reaction rate constant generated using this equation is very low, indicating that the reaction rate is slow. The R^2 score demonstrates no fit between the data trend and the proposed mathematical model. This can be observed from the R^2 value ranging from 0.2 to 0.3.

3.4 The Polynomial Regression Equation of 2nd Order

Regression is a quantitative method based on the relationship between independent variables X and variable Y [25]. In this investigation, mathematical predictions were made using equation (2). Polynomial functions are more suitable in the regression method since they are more versatile and not tied to linear data.

Table 1. Exponential Regression of Glycerol Conversion versus Reaction Temperature & Catalyst Concentration

Variable	Equation	Parameter			R^2
		y_0	A	R_0	
Temperature					
40 °C	$y = y_0 + A.exp^{(R_0.x)}$	27.02	1.18 Exp -14	0.1801	0.359
45 °C		37.21	- 6.857	0.0086	0.943
50 °C		38.13	- 1.367	0.0145	0,200
55 °C		17.67	3.352 Exp 20	-6.84 Exp 28	2.22 Exp -16
60 °C		634.8	-613.85	1.41 Exp -4	0.982
Concentration of Catalyst					
1 %	$y = y_0 + A.exp^{(R_0.x)}$	26.42	-348.31	-0.1725	0.374
3 %		19.30	-0.979	0.0116	0.256
5 %		38.13	-1.367	0.0145	0.200
7 %		-36345	36362	2.472 Exp -6	0.225

Tabel 2. 2nd Order Polynomial Regression of Glycerol Conversion against Reaction Temperature & Catalyst Concentration

Variable	Equation	Parameter			R^2
		a	b	c	
Temperature					
40 °C	$y = ax^2 + bx + c$	27.55	-0.023	1.94 Exp -4	0.078
45 °C		29.12	0.002	-7.70 Exp -4	0.948
50 °C		30.12	0.023	-0.0018	0.929
55 °C		45.47	-0.873	0.0012	0.255
60 °C		21.01	-0.088	2.77 Exp -6	0.982
Concentration of Catalyst					
1 %	$y = ax^2 + bx + c$	6.431	0.514	-0.0024	0.745
3 %		16.53	0.059	-5.14 Exp -4	0.299
5 %		30.12	0.237	-0.0017	0.255
7 %		5.690	0.512	-0.0022	0.466

This model is developed by adding the influence of each prediction variable (X) increased to the power [26]. Based on the data acquired, it is evident that the temperature variable has an R^2 value > 0.5 in some data. At low temperatures <45 °C, the coefficient values of variables a and b are generally small, and c is positive, while this is inversely proportional at high temperatures > 55 °C displaying a substantial value, but c tends to be tiny. The R^2 value at a temperature of 40 °C suggests the model is inadequate for this data. While at a temperature of 60 °C, the R^2 value is very high, which indicates that this model can explain practically all of the data. Meanwhile, for the catalyst concentration variable, the average R^2 value is <0.5. At a concentration of 1%, it is 0.74. However, when the catalyst concentration grows, the R^2 value declines substantially. Likewise, for the parameter values a, b, and c, at low concentrations (1% & 3%), the resulting a value tends to be modest, while for b and c, it varies. At high concentrations (5% & 7%), the lesser a and c values indicate a less substantial contribution between the quadratic and constant components. The polynomial model tends to be more suited at temperatures over 40 °C, while the catalyst concentration variable, the model, is less suitable in representing the trend of the resulting data.

4. Conclusions

The highest glycerol conversion at temperature variation & catalyst concentration is at temperature 50 °C & catalyst concentration 5% with a reaction time of 120 minutes, which is 46.01%. The mathematical equation model proposed at temperature variation tends to match the 2nd-order polynomial regression model with a high R^2 value. Meanwhile, there is no match between the data trend and the proposed model for catalyst concentration variation.

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