

The Kinetics of Ethanol Volume Change With Variation of Input Volume and Heating Temperature in The Re-Distillation Process of Glucomannan Extraction Residue Using Batch Distillator

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Article History :

Received : 25 February 2022

Received in revised form : 10 March 2022

Accepted : 18 March 2022

Keywords :

Arrhenius model

Distillation

Ethanol

Glucomannan

Re-distillation

ABSTRACT

Ethanol is commonly used as a solvent in extracting glucomannan from porang. However, the extraction process often leaves ethanol. The remaining ethanol can be re-distilled to save the use of it. The remaining ethanol is used in the re-distillation process with input volumes of 50L and 100L with variations in heating temperatures of 80°C, 85°C, and 90°C. This study aimed to analyze the effect of the ethanol input volume and temperature on the output volume of re-distilled ethanol and determine the constant change in volume of re-distilled ethanol using kinetics and Arrhenius equations. The results showed that the input volume and heating temperature variation differed significantly from the ethanol output volume. The k value changes in the ethanol output volume from re-distillation with an input volume of 50L and a temperature variation of 80°C, 85°C, and 90°C respectively were 0.0016, 0.0023, and 0.0027 L/min, while the input volume of 100L was 0.0009, 0.001, and 0.0014 L/min. The results of the k value as a function of temperature using the Arrhenius equation showed that the re-distillation process with an input volume of 50L and 100L produces activation energy (Ea) of 55.83 kJ/mol and 46.94 kJ/mol, while the collision frequency value (A) of 3.03×10^5 /min and 7.7×10^3 /min.

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1. INTRODUCTION

Ethanol, commonly known as ethyl alcohol, pure alcohol, or absolute alcohol with the molecular formula C_2H_5OH (Erawati, 2008), is a solvent widely used in the pharmaceutical, food, and chemical industries because it is easily soluble in water, ether, acetone, benzene, and all other substances organic solvent. The use of ethanol varies depending on the content, such as levels of 20–40% for cigarette sauce ingredients, mixtures of

beverages and perfumes, levels of 60–70% for kerosene substitution, and levels of 80% for sterilization materials in hospitals, medical centers, and drug raw materials. Ethanol with more than 90% levels is used for the chemical industry and large pharmaceutical factories (Usman & Dwinanda, 2013), which is used as a solvent in the extraction process (Verdiana *et al.*, 2018).

Extraction is the process of separating a material based on differences in its solubility, and the extraction method is an effective method to increase the value of material by separating other substances to obtain a purer material (Sönmez *et al.*, 2018). The solvent in the widely used extraction is ethanol, such as in extracting glucomannan from porang (*Amorphophallus oncophyllus*) (Suryani *et al.*, 2016). In the process of glucomannan extraction, ethanol functions to bind water which dissolves glucomannan.

Glucomannan is a water-soluble dietary fiber with strong hydrocolloid properties and low in calories, so in the food industry, glucomannan is used as a food and non-food additives such as in cosmetics and health product industries. In the food sector, glucomannan can be used to manufacture syrups, edible films, gelatin, noodles, and binders in making sauces (Akesowan, 2002).

Extraction of glucomannan from porang flour using ethanol as a solvent can be done with various kinds of mixing. In a study conducted by (Anindita *et al.*, 2016), extracting glucomannan from salak seed flour used ethanol as a solvent with a ratio of 1:9, 1:11, 1:13, 1:15, and 1:17 (gram/ml), with an ethanol concentration of 95%. The use of ethanol in the extraction depends on the required glucomannan requirement. The higher the ethanol concentration in the extraction process, the higher the percentage of glucomannan produced (Desi & Widjanarko, 2010).

However, besides producing glucomannan, the extraction process also has processing residues, namely ethanol and other processing residues. Ethanol used in each extraction process is not in small amount. Ngatirah & Syaflan (2016) reported on the extraction of glucomannan from porang flour, ethanol used in a ratio of 1:2, which means that in every 1000 ml of supernatant, 2000 ml of 96% ethanol is needed, so the remaining ethanol from water binding is very large. The ethanol savings can be done by re-distillation so that higher purity ethanol is obtained and used again for the extraction process.

In this study, the material used was ethanol remaining from the glucomannan extraction process with a concentration of 48-52%, where the concentration of residual ethanol was still very high. The re-distillation process is carried out to increase the ethanol concentration in the extraction or other procedures. Distillation separates liquid mixtures according to their respective components based on differences in boiling points and solution concentrations (Komariah *et al.*, 2009). When a liquid is heated, the component with the lower boiling point will evaporate first than the component with the higher boiling point.

The distillation process selection depends on the characteristics of the components of a liquid and the desired result. Several methods have been carried out, such as simple distillation, fractional (stratified) distillation, vacuum distillation, steam distillation, and azeotrope distillation (Setiawan, 2018). Simple distillation separates a mixture of water and alcohol because water and alcohol have very different boiling points, namely 78.4°C for alcohol and 100°C for water boiling points (Yanti *et al.*, 2019). The aims of this study were (1) to analyze the effect of the ethanol input volume, and re-distillation temperature on the volume and concentration of re-distilled ethanol

output, (2) determine the constant (k) changes in the volume of re-distilled ethanol using kinetic equations and (3) determine the value of activation energy (E_a) and collision frequency (A) using the Arrhenius equation so that the value of k is obtained as a function of temperature. The re-distillation process in this study was carried out using variations in the input ethanol volume and heating temperature. During the process, changes in volume and concentration of re-distilled ethanol were analyzed using kinetic equations, and changes in temperature during the process were used to analyze the constant k as a function of temperature using the Arrhenius equation.

2. MATERIALS AND METHODS

The research was conducted at the Food and Postharvest Engineering Laboratory, 2nd floor, Department of Agricultural and Biosystem Engineering, Faculty of Agricultural Technology, Gadjah Mada University. The material used in this research is ethanol leftover from the glucomannan extraction process from porang with a 48-52% concentration. The tools used are distillator, alcohol meter, stove, regulator, gas, water pump, reservoir, jerry can (output container), measuring cup, and hand pump.

Preparation begins by entering ethanol whose initial alcohol concentration has been measured into the distillation chamber, with variations in the input volume of 50L and 100L. After the material is in the distillation chamber, it is heated with variations in the heating temperature of 80°C, 85°C, and 90°C. The first drop of distilled ethanol was used as a reference to calculate the time of data collection. The amount of ethanol produced was measured every 10-minute interval using a measuring cup along with data collection on the percentage of ethanol concentration.

2.1. The Distillation Rate

The distillation rate changes the output volume every time during the distillation process. The distillation rate indicates whether there is a difference in the given variation to time.

2.2. Statistical Analysis

Statistical analysis was carried out to see whether there was a significant difference between the volume of ethanol input and heating temperature to the volume of ethanol output. Statistical testing was carried out using SPSS. The statistical analysis results will determine whether the initial hypothesis (H_0), namely that there is a significant effect of variations in the input volume and heating temperature on the total output volume produced, is acceptable or not. Statistical testing was carried out using two-way ANOVA analysis.

2.3. Determination of Reaction Order and First-Order Analysis

First-order states that the reaction rate is affected by the concentration of the reactants raised to the power of one.

$$\frac{dV}{dt} = -k [V]^1 \quad (1)$$

Data included in the first-order is determined by examining the formed graph. The following are the characteristics of graphs that use first-order shown in Figure 1 and Figure 2.

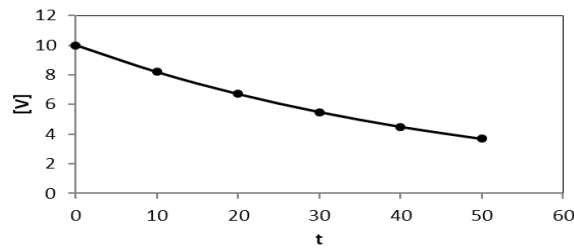


Figure 1. The relationship of the concentration of V during the re-distillation process which shows the first-order reaction ($n = 1$)

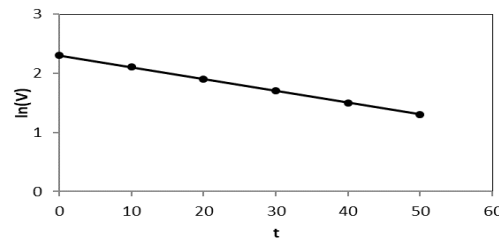


Figure 2. The relationship of $\ln(V)$ during the re-distillation process which shows the first-order reaction ($n = 1$)

The n value on the first-order is equal to 1, which means that the reaction rate is affected by the concentration of the reactants raised to the power of one (Panggabean, 2011).

$$\frac{dV}{dt} = -kV$$

$$\int_{V_0}^{V_t} \frac{dV}{V} = - \int_0^t k dt$$

$$\ln \left(\frac{V_t}{V_0} \right) = -k(t_t - t_0) \tag{2}$$

$$\ln V_t - \ln V_0 = -k(t_t - t_0) \tag{3}$$

The value of equation 3 is expressed in a linear graph of $y = ax$, where $\ln(V_t/V_0)$ is the value of y , $t_t - t_0$ is the value of x , and k is the value of a , which is obtained from the graph.

$$\ln V_t = \ln V_0 - k(t_t - t_0) \tag{4}$$

$$V_t = V_0 e^{-kt} \tag{5}$$

The k value, the slope value obtained from the graph, is used to calculate the predicted value of the parameter each time on the first-order by entering the k value in the formula for equation 4.

2.4. Arrhenius Equation

The Arrhenius equation is an equation that is used to generate an equation to predict the distillation process based on the results of the kinetic model for each variation. The Arrhenius equation determines the effect of temperature changes (T) on the distillation rate and the reaction constant. The Arrhenius equation is generally written as follows:

$$k = A \exp \frac{E_a}{RT} \tag{6}$$

$$\ln k = \ln A - \left(\frac{E_a}{R}\right) \left(\frac{1}{T}\right) \quad (7)$$

Where $\ln k$ is y , the a value is the value of $-(E_a/R)$, R is the universal gas constant (8.314 J/mol), the b value is $\ln A$, and the x value is $(1/T)$ (Gita *et al.*, 2018). After obtaining the equation from the graph of $\ln k$ compared to $\ln T$, the activation energy value (E_a) and frequency factor (A) can be obtained by:

$$a = -\left(\frac{E_a}{R}\right)$$

$$E_a = -a(R) \quad (8)$$

$$b = \ln A$$

$$A = e^b \quad (9)$$

The reaction rate constant (k) depends on the heating temperature (T) and the required activation energy (E_a). The higher the heating temperature, the lower the activation energy, which causes the value of the reaction rate to be greater. Using the Arrhenius equation, we can find the value of E_a , which is the minimum energy required to start a chemical reaction.

3. RESULTS AND DISCUSSION

3.1. The Observation Result of Decreasing Output Volume

The main determining factor in the distillation process is the heating temperature. The higher the heating temperature, the greater evaporation will occur. The temperatures required to evaporate ethanol and water are 78.4 °C and 100 °C. Figure 3 shows a decrease in the output volume (V_t) during the distillation process, where the output volume of ethanol during the heating process decreases due to the reduction of ethanol in the solution, at temperature variations of 80 °C, 85 °C, and 90 °C along with input volumes variation of 50 L and 100 L. Ethanol which has a lower boiling point than water, will evaporate first. The volume of re-distilled ethanol every 10 minutes of data collection showed different concentration values. The distribution of the ethanol concentration in the output volume is shown in Figure 4.

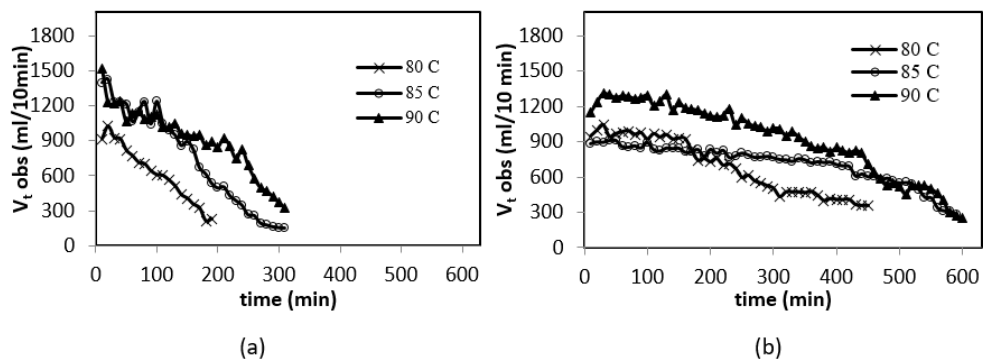


Figure 3. The ethanol output volume during the distillation process every 10 minutes with variations in heating temperature; (a) input volume 50L and (b) input volume 100L

Figure 4 shows the ethanol concentration produced at variations in heating temperature of 80 °C and 90 °C at the highest concentration of 85-89%, both for variations in the input volume of 50 L and 100 L. However, the ethanol concentration with a heating temperature variation of 90 °C continued to decrease to a concentration of 60-64%. In contrast, the 80 °C heating temperature variation produces ethanol with a concentration of 85-89% until the end of the re-distillation process. The largest volume of ethanol output with an 85-89% concentration is also shown at the 80 °C heating temperature variation. This means that the higher the heating temperature, the lower the concentration.

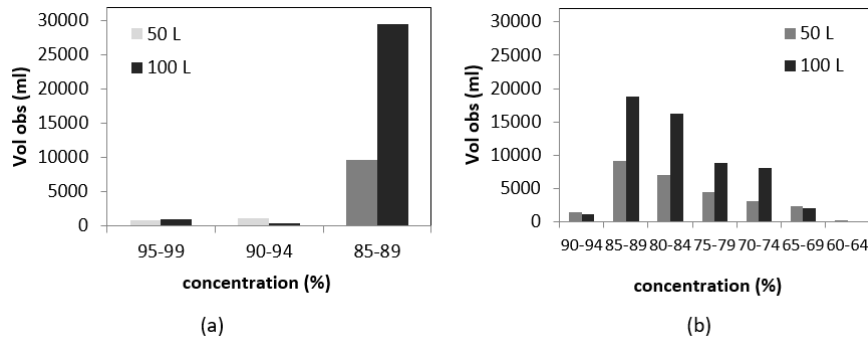


Figure 4. Distribution of ethanol re-distillation concentration based on the output volume with variations in heating temperature (a) 80 °C and (b) 90 °C

3.2. The Observation Result of Cumulative Output Volume

Cumulative processing of distillation data is data processing by adding up the distillation output at each data collection time. With the cumulative method, the distillation results are added every 10 minutes so that the final distillation result is the total result of one distillation process. Cumulative distillation results are shown in Figure 5.

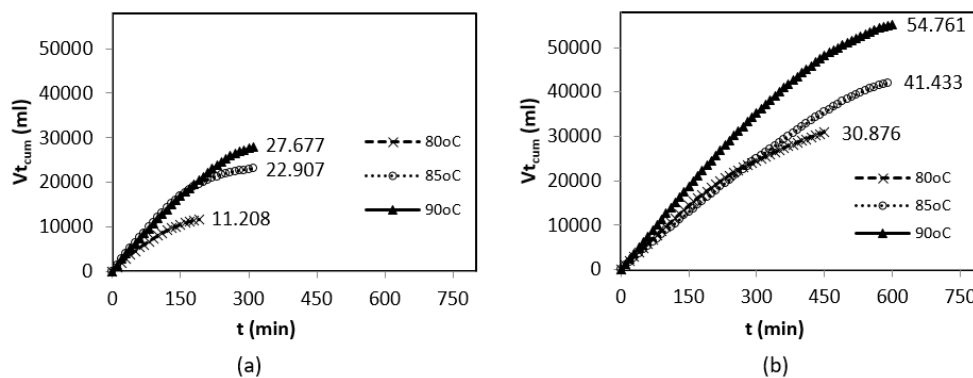


Figure 5. The cumulative volume of ethanol output during the distillation process with variations in heating temperature; (a) 50L volume; (b) 100L volume

Figure 5 shows that the distillation output increases with the length of the distillation process. The greater the input volume of the ethanol solution, the greater the ethanol output produced. Likewise, with the heating temperature, the higher the heating temperature, the greater the output volume produced. The results of statistical analysis using two-way ANOVA in Table 1 show that variations in the input volume and

heating temperature have a significant effect on the total output volume, with significant values of variation in input volume and heating temperature being 0.00 and 0.00, respectively, where the value is smaller than the limit of significance value of 0.05. The interaction of the variation of the input volume and heating temperature to the total output volume also shows a significant difference to the output volume, with a significance value of 0.046.

Table 1. The effect of input volume and heating temperature variations on the total output volume

Source	Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	3.346E9 ^a	5	6.693E8	203.908	.000
Intercept	1.866E10	1	1.866E10	5.687E3	.000
VOL	2.093E9	1	2.093E9	637.769	.000
suhu	1.221E9	2	6.105E8	186.003	.000
VOL * suhu	3.204E7	2	1.602E7	4.882	.028
Error	3.939E7	12	3282161.833		
Total	2.205E10	18			
Corrected Total	3.386E9	17			

a. $R^2 = 0,988$ (Adjusted $R^2 = 0,984$)

The initial hypothesis (H_0) is accepted, and it is concluded that there is a significant difference or effect of variations in the input volume and heating temperature on the total output volume produced. There is a significant difference between the treatment of variations in the input volume and heating temperature to the total output volume, then Duncan's test is carried out to see the best result.

The results of Duncan's test on the effect of variations in heating temperature on the total output volume in Table 2 show a significant difference between the treatment with 80 °C, 85 °C, and 90 °C heating temperature variations. The heating temperature variation of 90 °C has the highest distillation output volume value, which is 41,856.16 ml, so it can be concluded that the higher the heating temperature used in the distillation process, the greater the volume value of ethanol produced.

Table 2. Duncan's test the effect of heating temperature variations on the total output volume

Temp_variations	N	Subset
		1
Duncan ^{a,b,c}	80 °C	21,727.50 ^a
	85 °C	33,024.33 ^b
	90 °C	41,856.16 ^c
	Sig.	1.00

3. 3. The Distillation Rate

The distillation rate indicates whether there is a difference in the given variation to time. The distillation rate changes the output volume every time during the distillation

process. The distillation rate of the research results is shown in Table 3. The distillation rate calculation data shown in Table 3 are presented in Figure 6, showing that there is an increase in the value of the distillation rate at a temperature variation of 80°C, 85°C, and 90°C, both at the input volume variation of 50L or 100L. It can be seen from the graph that the higher the heating temperature, the higher the distillation rate.

Table 3. The distillation rate of ethanol output volume

Volume (liter)	Temperature (°C)	Distillation time (minutes)	Average output volume (ml)	Distillation rate (ml/minutes)
50	80	190	11650	61.32
	85	310	23059	74.38
	90	310	28000	90.32
100	80	450	30876	68.61
	85	590	42025	71.23
	90	600	55285	92.14

At 80 °C, 85 °C, and 90 °C, the distillation rates with the 50 L input solution variation were 61.32 ml/minute, 74.38 ml/minute, and 90.32 ml/minute, respectively. A similar increase also applies to the 100L input solution variation. This means that the higher the heating temperature given, the higher the distillation volume obtained every minute. The increase in distillation results was since at a temperature of 90 °C, the water in the ethanol solution remaining from the glucomannan extraction evaporated so that it affects the concentration of the distillate, where the higher the heating temperature, the lower the average concentration of ethanol produced.

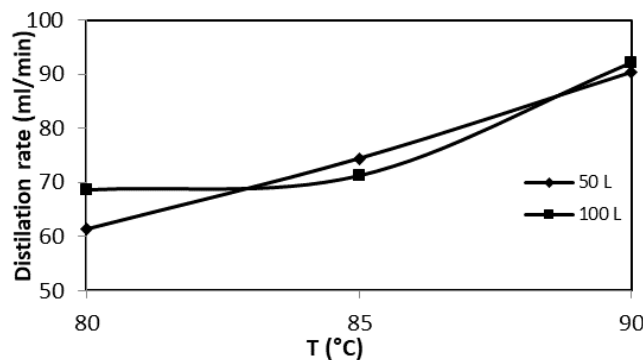


Figure 6. Distillation rate of total ethanol output volume to the heating temperature with input volume variation

3.4. Reaction Order and First-Order Kinetics Analysis of the Output Volume

In this study, the solution used for distillation is the remaining solution from the extraction process with an ethanol content of 48-52%, which means that the ethanol available in the solution is half of the total solution. The initial amount of ethanol in the total solution is the equilibrium limit to be achieved in the distillation process. The equilibrium value indicates the maximum amount of ethanol that can be distilled from the solution. The equilibrium limit is applied in calculating the predicted distillation results.

Equation 3 still uses $\ln (V_t - V_o)$, while in its application, the equilibrium limit is used in the equation. The equilibrium distillation limit is shown in $(V_t - V_e)/(V_o - V_e)$, where V_t is the amount of output at time t , V_o is the amount of initial output, and V_e is the amount of ethanol at its equilibrium condition. The analysis with first-order kinetics is shown in Figure 7.

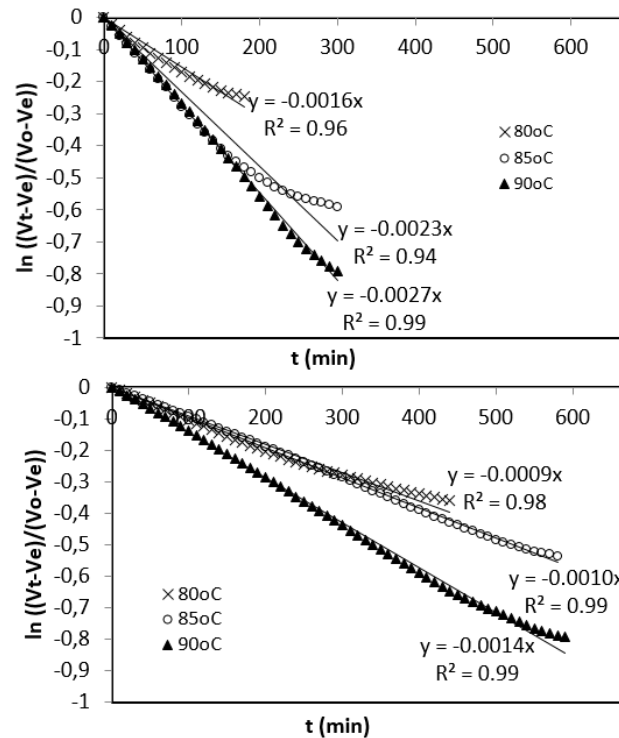


Figure 7. Comparison of \ln cumulative output during the distillation process with variations in heating temperature; (a) 50 L input volume; (b) 100 L input volume

Figure 7 shows the reaction rate of the distillation process by applying equation 3. The result of the graph is a linear equation $y = ax$, where y is $(V_t - V_e)/(V_o - V_e)$, x is the time t , and a is the value of k that will be used to predict the distillation process results. The k value resulting from the first-order kinetics equation shown in Figure 7 with a volume variation of 50 and 100 liters at a heating temperature variation of 80 °C, 85 °C, and 90 °C, respectively, is -0.0016, -0.0023, -0.0027, -0.0009, -0.001 and -0.0014. With the k value from the first-order kinetic analysis, the k value is entered back into equation 5 to predict the results of the next distillation.

The coefficient of determination (R^2) test is carried out to determine the suitability of the results between observations and predictions. The higher the R^2 value, the greater the relationship between the observed values in determining the predictive value. The value of R^2 with an input volume variation of 50 L at a heating temperature variation of 80 °C, 85 °C, and 90 °C is 0.98; 0.97 and 0.99. The R^2 value of the 100 L input volume with variations in the heating temperature of 80 °C, 85 °C, and 90 °C, respectively, is 0.98; 0.99 and 0.99.

The R^2 value of the comparison between observations and predictions obtained using the first-order kinetic analysis equation yielded a high value, almost equal to 1. This indicates that the first-order kinetic analysis predicts the ethanol output very well. So the predicted k value using first-order kinetics analysis can be used in predicting the

output results in other experiments. First-order kinetics analysis is then continued by using the Arrhenius law equation to find out the general equation and obtain the value of k .

3.5. Activation Energy

The Arrhenius law equation states the relationship between the activation energy and the reaction rate. The value of activation energy (Ea) and collision frequency (A) will be obtained using the Arrhenius equation. The values of Ea and A are obtained using the k value obtained from the first-order kinetics analysis, according to equation 7. So that the equation $y = ax + b$, where y is $\ln k$, x is $1/T$, a is $-Ea/R$, and b is $\ln A$. Data on the k value resulting from first-order kinetics analysis and heating temperature are shown in Table 4. The results of the Arrhenius equation using the k value obtained from first-order kinetics analysis compared to one per time are shown in Figure 8.

Table 4. The k value from the first-order kinetics and heating temperature (T)

Input volume	T (K)	1/T (K ⁻¹)	k_{obs}	$\ln k$
50	353	0.00283	-0.0016	-6.4378
	358	0.00279	-0.0023	-6.0748
	363	0.00275	-0.0027	-5.9145
100	353	0.00283	-0.0009	-7.0131
	358	0.00279	-0.0010	-6.9078
	363	0.00275	-0.0014	-6.5713

Figure 8 shows the relationship $\ln k$ versus $1/T$ to determine the value of Ea and A . The graph states the relationship of $\ln k$ versus $1/T$ in the form of $y = ax + b$ from the graph, then look for the values of Ea and A . The Ea value is obtained by multiplying the root of the R^2 with A , which is obtained by taking the anti-log the value of b . The results of Ea and A values are shown in Table 5.

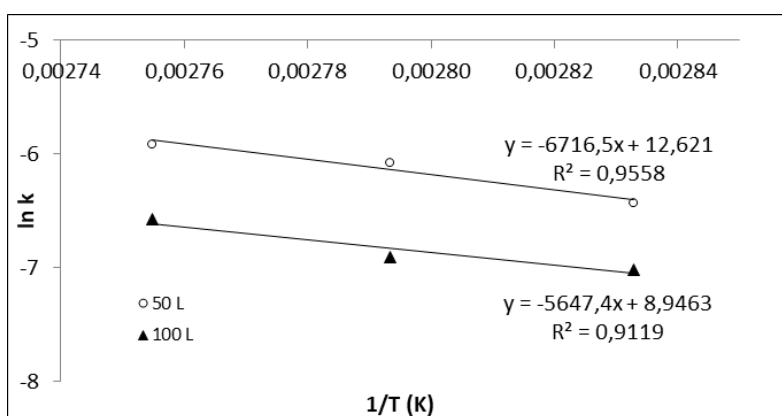


Figure 8. The relationship between $\ln k$ and $1/T$ to determine the Ea and A value

Table 5. The Ea and A value based on the Arrhenius law equation

Input volume (L)	R	a	b	Ea	A
50	8.31	-6716	12.62	-55,836.82	302,549.44
100	8.31	-5647	8.946	-46,949.15	7677.12

The E_a and A values resulting from the Arrhenius law equation shown in Table 5 are then used to find the predicted value of k . The resulting Arrhenius law equation model based on the E_a and A values are shown in Table 6.

Table 6. The Arrhenius equation model

Input volume variations (liter)	Arrhenius equation model
50	$k = 3.03 \times 10^5 \times e^{-\frac{(-55.83 \times 10^3)}{RT}}$
100	$k = 7.7 \times 10^3 \times e^{-\frac{(-46.94 \times 10^3)}{RT}}$

The predicted k value using the Arrhenius law equation model shown in Table 6, then validated with the predicted k value of the first-order kinetics equation expressed in the coefficient of determination (R^2). The validation of the k value is shown in Figure 9. Figure 9 shows the comparison of the predicted k value of the first-order kinetics equation with the predicted k value of the Arrhenius law equation at a heating temperature variation of 80 °C, 85 °C, and 90 °C with an input volume of 50 L and 100 L expressed in the value of the coefficient of determination (R^2). The R^2 value with the input volume variations of 50L and 100L at a heating temperature variation of 80 °C, 85 °C, and 90 °C, respectively, is 0.95 and 0.91. Based on Figure 9, the R^2 value obtained is almost equal to one. This indicates that the predicted k value for the first-order kinetic equation is close to the predicted k value from the Arrhenius law equation.

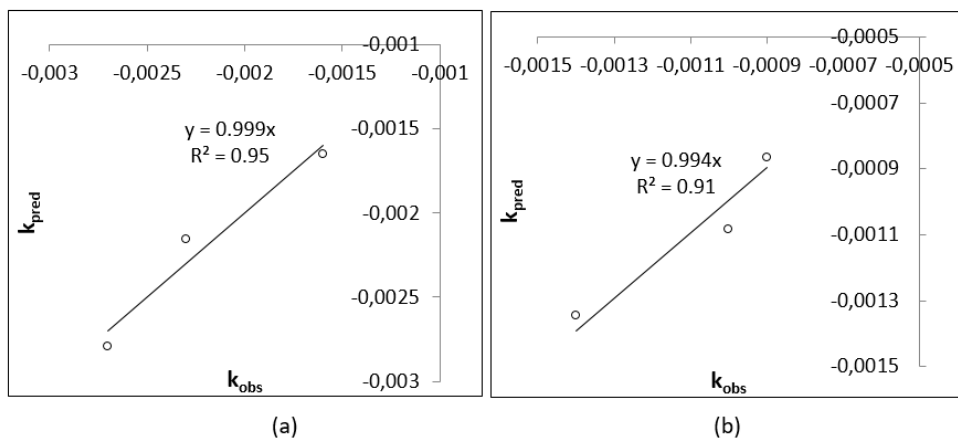


Figure 9. The comparison between the constant value (k) of observation and prediction during distillation; (a) 50 L input volume; (b) 100 L input volume

The value of k from the Arrhenius law equation is reused to predict the volume of ethanol output of the distillation process. The comparison of the output volume of observations, predictions with the first-order kinetics, and predictions with the Arrhenius equation during the distillation process is shown in Figure 10.

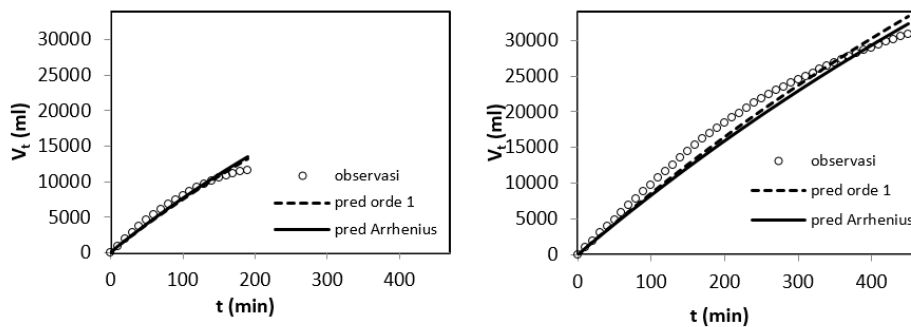


Figure 10. The comparison between the output volume observations, predictions using first-order kinetics, and predictions using the Arrhenius equation during the distillation process; (a) 80 °C temperature, 50 L input volume; (b) 80 °C temperature, 100 L input volume

4. CONCLUSION

Based on the observations, data analysis, and discussion, variations in the input volume of 50 L and 100 L and heating temperatures of 80 °C, 85 °C, and 90 °C showed a significant difference to the distillation output volume. The value of the constant (k) for decreasing the volume of ethanol output with first-order kinetics ($n = 1$) which is calculated based on the cumulative distillation output volume with volume variations of 50 L and 100 L at heating temperature variations of 80 °C, 85 °C, and 90 °C were 0.0016, 0.0023, 0.0027, 0.0009, 0.001 and 0.0014 L/min, respectively. The results of the analysis using the Arrhenius equation for first-order ($n = 1$) at a variation of the input volume of 50 L resulted in an activation energy value (Ea) of 55.83 kJ/mol and a collision frequency (A) of 3.03×10^5 /minute. At the input volume variation of 100 L, the activation energy value (Ea) is 46.94 kJ/mol, and the collision frequency (A) is 7.7×10^3 /minute.

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