

Integrated Sci-Tech :
The Interdisciplinary Research Approach
Volume 1

Editorial board:

Ardian Ulvan

Irza Sukmana

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Preface

This book is all about the interdisciplinary research that integrates engineering, life and applied sciences, medical and biomedical engineering, agriculture engineering and food sciences. The aim was to provide the initial roadmap at a cross section basic research, technological and social developments, processes development, applications integrity, and real-world usage. The genuine motivation for the book was to provide a suitable reference text for those who interested in the multi and interdisciplinary studies which might be beneficial for basic and advance researches, enhancing the curriculum and enriching teaching and learning materials, mostly in the level of postgraduate studies.

In addition, the book was also planned to provide advanced orientation and understanding for related industries and governments to looking across industrial partnerships, business strategic, and policy and regulations. In general, the book is expected to be beneficial for a wide range of readers.

This book consists of twenty five chapters divided into four sections i.e., engineering, life and applied sciences, medical and biomedical engineering, agriculture and food science. Each chapter is a completely self-directed contribution in chained discussion which aims to bring academia, researcher, practitioners and students rise to speed with the novel developments within the particular area.

In order to enhance the reader experience, each book chapter contains its own abstract, instruction, main body, as well as conclusion sections. Moreover, bibliography resources are available at the end of each chapter.

To achieve all these aims and goals, the book should deliver a breadth of information. We are pleased and thankful for all distinguish authors and reviewers for their contribution that have made this book possible. We do hope that you will enjoy this book and find it as a useful guide and reference.

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Reaction Kinetics of Acetic Acid and Ethanol Esterification Catalyzed by ZSM-5 Catalyst

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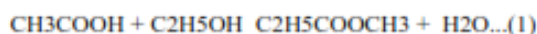
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Abstract. Kinetics of esterification of acetic acid and ethanol use solid catalyst ZSM-5 has been studied in this research. Experiments were carried out in a batch reactor, reactant ratio of sulfuric acid and ethanol 1:2, stirring at 1000 rpm, reaction time in 150 minutes and catalyst concentration 6 g/L. Variable varied is reaction temperature of 308°K to 348°K. Reaction kinetics data obtained use Matlab 7.8 software. From the experimental results, the greatest conversion is obtained at temperatures of 348°K is 70.16%. Activation energy obtained from the calculation was 28,566.8 J/mol. The experimental data were compared with data from the calculation of Matlab is used to see the approach of the model used. It can be concluded that the modeling approach used in accordance with the experimental data.

Keywords: acetic acid, ethanol, esterification, reaction kinetics, ZSM-5 catalyst

I. Introduction

On the industrial scale, ethyl acetate produced from the esterification reaction between acetic acid (CH₃COOH) and ethanol (C₂H₅OH) using sulfuric acid catalyst (H₂SO₄). Ethyl acetate is a compound resulting from the exchange of the hydroxyl group in acetic acid with a hydrocarbon group in ethanol. Ethyl acetate on an industrial scale is widely used as a solvent in industrial paints, thinner, material for plastics, adhesives, cosmetics, pharmaceutical and organic chemical industry. The overall reaction between acetic acid and ethyl alcohol over a catalytically ZSM-5 catalyst is as follows:



One example of an esterification reaction using sulfuric acid catalyst is esterification of free fatty acids and alcohol [1]. The highest conversion was obtained by 96% at a temperature of 328°K with a ratio alcohol / free fatty acids was 6.13 and the concentration of catalyst 2.2% by mass. But the use of sulfuric acid as a catalyst has several weaknesses such the difficulty of separation of the product with the catalyst so that the waste treatment plant has a greater load in the presence of sulfuric acid inseparable in refining and high levels of corrosion in equipment.

Because there is a weakness in the sulfuric acid catalyst, then the next researcher using other alternative replacement for sulfuric acid catalyst. Esterification reaction of ethanol and acetic acid is done by using a catalyst Lewatit monoplus-100 [2]. The highest conversion of 87.3% was obtained at a temperature of 358°K

and a catalyst concentration of 0.8 mass resin / mass ethanol. Esterification reaction of acetic acid and ethanol also been carried out using acid catalyst Amberlyst-15 resin from Rohm and Haas [3]. The highest conversion was obtained by 75% at a temperature of 353^oK with a mole ratio of acetic acid: ethanol is 1: 2 and a catalyst concentration of 5.4 gram per 100 grams of acetic acid. The kinetics of the reaction rate obtained from the study are:

$$k = 2.6 \times 10^{14} e^{\left(\frac{-104,129}{RT}\right)}$$

Kinetics of esterification reaction between acetic acid and methanol was studied using a catalyst and not using the catalyst [4]. The results obtained showed that the esterification reaction by using a catalyst is faster and give conversion 54% compared to the reaction that does not use a catalyst with a conversion of 14% within 180 minutes. The highest conversion using a catalyst obtained by 54% at a temperature of 328^oK with a mole ratio of acetic acid and methanol is 1: 3 as well as the weight of the catalyst at 10% by weight of acetic acid. In this research, used a stirrer speed of 1,000 rpm. In previous research has been learned about the resistance of the external mass that is by varying the stirring speed of 800 rpm to 1,200 rpm [4], because the stirring speed in excess of 800 rpm can eliminate external diffusion, internal diffusion ignored during the esterification reaction conditions. The result, for the reaction with stirring speeds above 1,000 rpm did not experience significant changes in the rate of reaction. So the esterification reaction was carried out with stirring speed of 1,000 rpm. The activation energy reactions using the catalyst obtained is 37,626 kJ / mol.

II. Materials and Methods

A. Chemical

Acetic acid and ethyl alcohol were supplied by Merck AG. Acetic acid of 99.5% (w/w) purity (Merck) and ethanol aqueous solution with a content of 96% w/w ethanol (Merck) were used as supplied.

B. Catalyst

The catalyst used is a catalyst ZSM-5, which has been synthesized from lignite bottom ash and rice husk. The synthesis of ZSM-5 performed in an autoclave at temperature of 180^oC with an autogenous pressure, the mole ratio parameter Na⁺ / SiO₂ 0.2 mol / mol, mole ratio H₂O / SiO₂ 30 mol / mol, mole ratio TPABr / SiO₂ 0.05 mol / mol, the mole ratio of SiO₂ / Al₂O₃ 50 mol / mol and crystallization time of 48 hours using a template TPABr. Percent crystallinity of ZSM-5 product obtained by 86.95%. ZSM-5 catalyst concentration used adalam 6 g / L.

C. Reaktor Batch

The equipment used in this research is shown in Fig. 1. The reactor used is a three-neck flask with a volume of 1 L, equipped with a condenser, thermometer, hot plate and magnetic stirrer.



Fig. 1. Research Equipment

D. Procedure

Acetic acid and ethanol was added into the reactor with a mix volume of 0.5 L, then the stirrer is moved so that the system is mixed. Samples were taken for analysis of the initial concentration of acetic acid, then the catalyst ZSM-5 was added and the reactor was shut down (reactor equipped with a thermometer). Hot plate turned on and the stirrer set at 1,000 rpm. The heater is adjusted to the desired temperature is reached which is 35°C then 10 mL samples are taken every 15 minutes during 150 minutes. The experiment was repeated by varying the reaction temperature 45°C, 55°C, 65°C and 75°C. Each sample taken is analyzed to determine the conversion.

E. Result Analysis

The analysis of concentration acetic acid is done by using the titration method. Samples were taken and put in erlenmeyer, then add 3 drops of indicator PP. Furthermore, the sample is titrated with 0.1 N NaOH solution until the color changes from clear to pink. Be noted the volume of titrant, repeat the experiment two times and calculated initial concentration acetic acid using the formula:

$M_1 \times V_1 = M_2 \times V_2$ with :

V1 - volume of titrant solution (NaOH)

V2 - the volume of titrated solution (CH₃COOH)

M1 - concentration the titrant solution (NaOH)

M2 - concentration of the solution being titrated (CH₃COOH)

Conversion calculation based on the results of the reduction reaction between the initial acetic acid concentration of acetic acid reduced residual divided by the initial acetic acid.

$$X = \frac{A_0 - A_t}{A_0}$$

with :

A₀ - initial concentration acetic acid

A_t - concentration residual acetic acid every 15 minutes

F. Derivation of A Catalytic Rate Equation

In the esterification reaction reversible and irreversible mechanism used in this study. The reaction reversible mechanism can be written as follows [3] :

$$C_{AS} = \frac{k_A am C_A}{(k_A am + k_1 m C_{ES})} \dots\dots\dots(14)$$

Where:

$$k_o = \frac{k_1 m C_{ES} k_A am}{k_A am + k_1 m C_{ES}} \dots\dots\dots(15)$$

$$\frac{1}{k_o} = \frac{1}{k_A am + k_1 m C_{ES}} \dots\dots\dots(16)$$

$$\frac{1}{k_o} = \frac{1}{k_1 m C_{ES}} + \frac{1}{k_A am} \dots\dots\dots(16)$$

Because ethanol is given excess then $K_r = k_1 m C_{ES}$, assuming :

$$k_c = k_A am$$

$$\frac{1}{k_o} = \frac{1}{k_r} + \frac{1}{k_c}$$

$$k_o = \frac{1}{\frac{1}{k_r} + \frac{1}{k_c}} \dots\dots\dots(17)$$

Because the speed of stirring is done at 1000 rpm, then $K_c = 0$, so that $K_o = K_r$

$$\frac{C_{A0} dX_A}{dt} = k_r (C_{A0} - C_{A0} X_A) \dots\dots\dots(18)$$

$$\frac{dX_A}{dt} = \frac{k_r}{C_{A0}} (C_{A0} - C_{A0} X_A) \dots\dots\dots(19)$$

The equation to find the reaction kinetics of reversible and irreversible mechanism which is obtained of a decrease in the formula is the same, that is equation (6) and (19). K_r value can be searched by means of minimizing the sum of square of errors (SSE) using Matlab program of conversion of acetic acid acetic acid calculated and conversion data.

$$SSE = \sum (X_{strong} - X_{data})^2 \dots\dots\dots(20)$$

III. Results and Discussions

From the experiments obtained acetic acid conversion data as in Fig. 2.

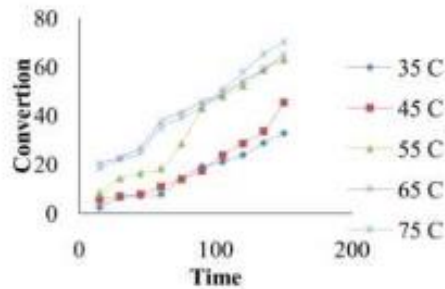


Fig. 2. Conversion Data Acetic Acid vs. Reaction Time Temperature Variation In each reaction

From Fig. 2 shows that the highest conversion at a temperature of 75°C is 70.16% in a reaction time of 150 minutes. This happens because by increasing the reaction temperature, the energy possessed by the molecules of reactant increased so as to overcome the activation energy. This leads to collisions between molecules increased, resulting in increased reaction rates. This result is not much different from the experiments using a catalyst Amberlyst 15 [3]. The highest conversion obtained at a temperature of 80°C is 75% in a reaction time

of 350 minutes. This means that the catalyst ZSM-5 is good enough to be used as a catalyst at esterification between acetic acid and ethanol.

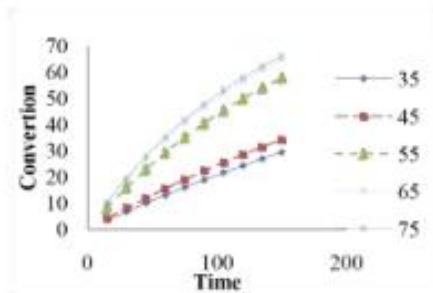


Fig. 3. Conversion Acetic Acid Reaction Time Calculate vs. Temperature Variation In each reaction

As seen in Fig. 3, the model used is good enough to describe the process that occurs during the reaction process. It can be seen from the results that calculate conversion value has a similarity with data conversion. So that the k_r value obtained from the calculation at each temperature variation can be used to calculate the activation energy of the reaction.

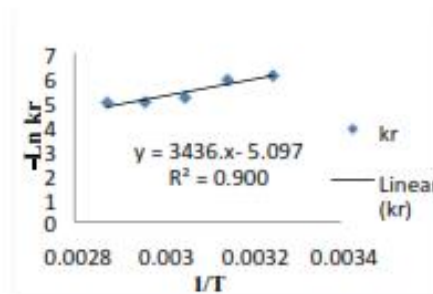


Fig. 4. Curve Relationship Between -Ln K and 1 / T

From the results linearized k_r value at each temperature in Fig. 4, the reaction kinetics equation:

$$k_r = 163.5306 \exp\left(\frac{-28.566,8}{RT}\right) \dots\dots\dots(21)$$

Activation energy obtained in this study is 28,566.8 J / mol. Activation energy obtained in this study is smaller when compared to the activation energy is obtained by using a catalyst Amberlyst 15 [3] is 104, 129 J / mol.

IV. Conclusions

From the research that has been done can be concluded that:

1. The catalyst ZSM-5 can be used as a catalyst at esterification of acetic acid and ethanol with the highest conversion is obtained at a temperature of 75°C is 70.16%.
2. The activation energy obtained of the esterification reaction mechanism is 28,566.8 J / mol, the reaction kinetics:

$$k_r = 163.5306 \exp\left(\frac{-28.566,8}{RT}\right)$$

SYMBOL

A	- the surface area of the catalyst
$r_A, r_E, r_{EAS}, r_{WS}$	- Mass transfer speed
m	- catalyst mass per volume of solution
C_A, C_E	- concentration acetic acid and ethanol
$C_{AS}, C_{ES}, C_{EAS}, C_{WS}$	- concentration a the surface of the catalyst for the acetic acid, ethanol, ethyl acetate and water
r_{AX}	- acetic acid reaction speed
r_{EX}	- ethano reaction speed
k_A, k_E, k_{EA}, K_W	- mass transfer coefficient acetic acid, ethanol, ethyl acetate and water
k_c	- equilibrium constants
C_{A0}	- initial acetic acid concentration
X_A	- conversion reaction
K_u	- constant overall
K_f, k	- the reaction rate constants
K_c	- mass transfer coefficient
t	- time
C_E	- concentration ethyl acetate
C_W	- the concentration of water
C_C	- catalyst concentration
CAH, CEH	- concentration acetic acid and ethanol on the surface of the catalyst
k1, k2, k3	- forward reaction rate constants
k-1, k-2, k-3	- backward reaction rate constants
K	- equilibrium constants
kw	- constant water absorption

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