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Hussain, Liu Gui Wei, Zhang Lian Zhen, Yongxue Li 865 165 Heterogeneous Kinetics of Hydration of α - Pinene for α -Terpineol Production: Non-Ideal Approach Herti Utami, Arief Budiman, Sutijan, Roto, Wahyudi Budi Sediawan 872 166 A Software for Calculation of Optimum Conditions for Cotton Bobbin Drying in a Hot-Air Bobbin Dryer Hilmi Kuscu, Ahmet Cihan, Kamil Kahveci, Ugur Akyol 876 167 FEA for Teeth Preparations Marginal Geometry L. Sandu, F. Topal, S.

Porojan 881 168 Computer Vision Applied to Flower, Fruit and Vegetable Processing Luis Gracia, Carlos Perez-Vidal, Carlos Gracia 885 169 Factors of competitiveness in the wine industry: an analysis of innovation strategy M^a del Valle Fernández Moreno; Isidro Peña García-Pardo; Jesús David Sánchez de Pablo González del Campo 894 Proceedings of World Academy of Science, Engineering and Technology file:///D:/JURNAL PROSIDING/Prosiding WASET 2011 AMS/index.html 5 of 9 2/23/2012 3:05 PM Abstract— α -pinene is the main component of most turpentine oils.

In order to obtain more valuable products, α -pinene in the turpentine can be hydrated in dilute mineral acid solutions to produce α -terpineol, which can be used as perfume, repellent of insect, antifungal and disinfectant. This paper presents a study on heterogeneous kinetics model for synthesis of α -terpineol from α -pinene. In this work, the two kinetics models both the ideal and the non ideal solution assumption have been developed to quantitatively describe effects of hydration process of α -pinene in aqueous acid solution.

The results of this study show that the kinetics model of the hydration of α -pinene can

be approached by the heterogeneous model. A good agreement between the experimental data and the model has been observed. Key words— α -pinene, α -terpineol, hydration, kinetics. INTRODUCTION TURPENTINE contains four major classes of components which are hydrocarbon, terpene alcohols, ethers, and sesquiterpenes [5].

α -Pinene ($C_{10}H_{16}$) is the main component of most turpentine oils. α -Pinene and β -pinene are the key compounds for the fine chemical synthesis and the important intermediates in pharmaceutical industry and perfumery [2]. The acid-catalyzed hydration and isomerization of α -pinene yields a complex mixture of monoterpenes, alcohols, and hydrocarbons.

The main products are α -terpineol, limonene, and terpinolene. Minor amounts of camphene, α and β -terpinene, α and β -fenchol, isoborneol, borneol, β -terpineol, and 1,8-terpine are also formed [3]. α -Terpineol ($C_{10}H_{18}O$) is the most important compound of the monocyclic monoterpene alcohols.

Terpineol can be used as perfume, repellent of insect, antifungal and disinfectant [1]. The hydration of terpene via acid catalysis is an important method for alcohol synthesis, and has several applications in the perfume and pharmaceutical industries. Aguirre et al. (2005) used hydrochloric acid, acetic acid, chloroacetic acid and oxalic acid as catalyst for the hydration of α -pinene.

Chloroacetic Herti Utami is with the Lampung University, Bandar Lampung, Indonesia (corresponding author to provide phone : +62-0274-9232121; fax: +62-0274- 902170; e-mail: hertie19@hotmail.com). A. Budiman, is with the Gadjah Mada University, Yogyakarta, 55281, Indonesia. He is now with the Department of Chemical Engineering, Gadjah Mada University, Yogyakarta, Indonesia (e-mail: abudiman@ugm.ac.id) Sutijan is with the Gadjah Mada University, Yogyakarta, 55281, Indonesia He is now with the Department of Chemical Engineering, Gadjah Mada University, Yogyakarta, Indonesia (e-mail: sutijan@chemeng.ugm.ac.id) Roto is with the Gadjah Mada University, Yogyakarta, 55281, Indonesia.

He is now with the Department of Chemistry, Gadjah Mada University, Yogyakarta, Indonesia (e-mail: roto05@ugm.ac.id) W.B. Sediawan is with the Gadjah Mada University, Yogyakarta, 55281, Indonesia. He is now with the Department of Chemical Engineering, Gadjah Mada University, Yogyakarta, Indonesia (e-mail: wbsrsby@indosat.net.id) acid was found to be a good catalyst for the production of terpineol from pinene. After 4 h of reaction at 70 °C, the highest selectivity was 95.5% with the conversion of 10%, whereas the higher conversion was 99% with selectivity of

69% [1]. Pakdel et al.

(2001) used sulphuric acid as catalyst to synthesize terpineol from turpentine, in the presence of excess acetone as solubility promoter. They reported 67% of selectivity to terpineol although the conversion was not reported [4]. II.F FUNDAMENTAL A heterogeneous kinetics model for synthesis of α -terpineol from α -pinene was developed to quantitatively describe effects of hydration process of α -pinene in aqueous acid solution. α -Pinene is assumed to be completely insoluble in water.

The following assumption were applied : the reaction takes place in the oil phase, the liquid film thickness is very small, so the reaction takes place in the bulk of the oil phase and the oil does not diffuse into the water phase. Kinetics Model The hydration reaction of α -pinene using acid catalyst is schematically shown in Figure 1: α -pinene + H₂O $\xrightarrow{H^+}$ α -terpineol Figure 1.

The reaction of α -pinene The reaction in figure 1 can be written as (1) A + B \rightarrow T (1) where A = α -pinene, B = water, T = α -terpineol. The reaction rate can be written as (2) for the ideal solution assumption. _____

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(2) In the reaction modeling for a liquid system where the mixture is non ideal, correction must be made to the concentration.

Usually, a phase model such as the UNIFAC and NRTL phase equilibrium models are used to predict this non ideality factor, the activity coefficient (γ). The rate equation is then written in terms of the activity of each component participating in the mixture (a_i). The reaction rate hence can be written as (3) for the non ideal solution.

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(3) Herti Utami, Arief Budiman, Sutijan, Roto, Wahyudi Budi Sediawan | Heterogeneous Kinetics of Hydration of α -Pinene for α -Terpineol Production: Non-Ideal Approach T k 1 k 2 where _____ and _____ and _____ For heterogeneous model, the mass balance of water (B) in the water phase can be written as (4) _____ ! "
 # ! \$ _
 % _
 & (4) and the mass balance of water (B) in the oil phase can be written as (5) _____
 _ _ ' _ _ (_) \$ _ _ & (5) The mass balance of α -pinene (A) in the oil phase can be written as (6) _____ (6) The mass balance of α -terpineol (T) in the oil phase can be written as (7), with the boundary condition : $t = 0 ; m = m_o ; C_{Bm} = 0 ; C_{Am} = C_{Amo} ; C_{Tm} = 0 .$

The adjustable parameters encountered in the model are then $(k_c A_c)$, k_1 and k_d . _____ \$ _ _ & _____

(7) The kinetics model is then solved using Runge Kutta method. Evaluation of the parameters were conducted by curve fitting method. The chosen values were the ones which give the minimum values of the sum of squares of errors (SSE) for all runs, data. The SSE was defined as equation (8).

++ , _ ?\$ _ !/! __ _ . &

(8) III. EXPERIMENT Experiments were conducted using the batch reactor (three neck flask), condenser, thermometer, heating mantle, vials, pipettes and other glass wares. A 0.25 mol of α -pinene sample was poured into a three neck flask (pyrex 500 ml) equipped with condenser (pyrex), thermometer, stirrer and was warmed up to the desired temperature.

In the other glasses, 0.6 mol of aquadest (the mol ratio aquadest to α -pinene was 2.4/1) and the chloro acetic acid catalyst (the catalyst concentration was 6 mol/L) were warmed up to the desired temperature. Once the desired temperature was reached, the aquadest and the catalyst was loaded into the flask.

The mixture was stirred and the reaction time was started to be calculated. The temperature was kept constant during the process. A sample was taken at every 60 min time interval during the hydration and was kept in a small vial. The experiment was conducted with various temperatures at 60 °C, 70 °C, 80 °C, and 90 °C. The samples were analyzed using a Gas Chromatograph (GC).

The analysis was performed in a Hewlett-Packard model 5890 gas chromatograph. The separation was performed using HP-5 capillary column and Flame Ionization Detector with helium as a carrier gas. The GC oven temperature was set at initial temperature of 80 °C, held for 5 minutes, increased at a rate of 5 °C/min to 115 °C and then increased to 280 °C at a rate of 20 °C/min. The injector and detector temperatures were set at 280 °C respectively. IV.

RESULT AND DISCUSSION The experiment provides data of temperature (T) and conversion of α -pinene (x data) at various time (t) for each variable of temperatures. In the first model (ideal solution assumption), the values of C₁₂ % and K were approximated from the experimental data. The stoichiometric equation implies : __ % __
_ 3 \$1 __ % & (9) _

rather than the interaction between the molecules in the mixture. The UNIFAC model has a combinatorial contribution to the activity coefficients, essentially due to differences in size and shape of the molecules, and a residual contribution, essentially due to energetic interactions.

The values of parameter : $(k_c A_c)$, k_1 and k_d were determined by curve fitting method, where the sum of squares of errors (SSE) was minimized. For the UNIFAC model, the similar trend also occurs on Figure 4 and 5. Figure 4 shows that the conversion increases with the increase of reaction time. And the a-pinene concentration decreases with the increase of reaction time (Figure 5).

The conversion has a tendency to increase by the increase of the reaction temperature. For the UNIFAC model, the constants of degradation rate were found to be $k_d = 0.000108 \text{ ml.mol}^{-1} \cdot \text{min}^{-1}$ (at the temperature of 80 °C) and $k_d = 0.000225 \text{ ml.mol}^{-1} \cdot \text{min}^{-1}$ (at the temperature of 90 °C).

The constants of the reaction rate, k_1 and k_2 could be correlated by the Arrhenius's equation, and the equations can be written as (15) and (16). The relative errors in equation (15) and (16) are 5.547% and 4.734%, respectively. $\Delta H = -5.283 \cdot 10^4 \text{ J/mol}$; $\Delta H_{A,H} = -3.285 \cdot 10^4 \text{ J/mol}$; $\Delta H_{A,H} = -3.285 \cdot 10^4 \text{ J/mol}$ (16) The constants of equilibrium and mass transfer coefficient are listed in table II.

In figure 4 and 5 show the similar results in which the kinetics model with non ideal approach (UNIFAC model) proposed can quantitatively describe the hydration of a-pinene using chloro acetic acid as catalyst. TABLE II CONSTANTS OF EQUILIBRIUM AND MASS TRANSFER COEFFICIENT (NON IDEAL SOLUTION) Temperature (K) $k_c A_c$ (1/minute) 333 0.6730 0.4753 343 1.0464 0.1327 353 1.3691 2.2956 363 1.0428 2.3096 Figure 4.

Conversion profile for the UNIFAC model 0 50 100 150 200 250 300 350 400 450 0 10 20 30 40 50 60 Time, minute Conversion A, % T=60oC, Model T=60oC, Data T=70oC, Model T=70oC, Data T=80oC, Model T=80oC, Data T=90oC, Model T=90oC, Data 0 50 100 150 200 250 300 350 400 450 0.1 0.12 0.14 0.16 0.18 0.2 0.22 0.24 0.26 Time, minute CAm, mol/ml T=60oC, Model T=60oC, Data T=70oC, Model T=70oC, Data T=80oC, Model T=80oC, Data T=90oC, Model T=90oC, Data 0 50 100 150 200 250 300 350 400 450 0 10 20 30 40 50 60 Time, minute Conversion A, % T=60oC, Model T=60oC, Data T=70oC, Model T=70oC, Data T=80oC, Model T=80oC, Data T=90oC, Model T=90oC, Data Figure 5.

Concentration profile for the UNIFAC model. The proposed heterogeneous kinetics model for synthesis of α -terpineol from α -pinene could be approached with the ideal and non ideal solution assumption (UNIFAC model). The ideal solution approach model in Figure 2 and 3 fits the data better than the UNIFAC model in Figure 4 and 5.

In the other words, the deviations found for the UNIFAC model are greater than the ideal equations model. The system consists of α -pinene (2,6,6-trimethylbicyclo (3.1.1) hept-2-ene), water, chloro acetic acid and α -terpineol that they are polar compounds. The slightly deviations of the UNIFAC model influenced by the group interaction parameter values for polar compounds.

The system is thermodynamically deviating from ideal behavior due to several causes as the strong polarity of component, their association behavior, etc. **V.C ONCLUSIONS** The results of this study show that kinetics modeling of the hydration of α -pinene using chloro acetic acid as catalyst could be modeled with a heterogeneous model.

The different kinetics models both the ideal and the non ideal solution assumption (UNIFAC model) have been developed to quantitatively describe effects of hydration process of α -pinene in aqueous acid solution. Evaluation of parameters for the heterogeneous model were conducted. A good agreement between the experimental data and the model has been observed.

NOTIFICATION

A_c = Mass transfer area = total area interface, cm^2
 C_{Am} = α -pinene concentration in oil phase, mol/mL
 C_{Bm} = Water concentration in oil phase, mol/mL
 C_{Amo} = Initial concentration α -pinene in oil phase, mol/mL
 C_{Bmo} = Initial water concentration in oil phase, mol/mL
 C_{Tm} = α -Terpineol concentration in oil phase, mol/mL
 C_{Bm}^* = Water concentration in equilibrium, mol/mL
 C_{Am}^* = α -pinene concentration in equilibrium, mol/mL
 C_{Tm}^* = α -Terpineol concentration in equilibrium, mol/mL
 k_c = Mass transfer coefficient, 1/minute
 k_1 = Constant of kinetic reaction, $ml.mol^{-1}.min^{-1}$
 k_2 = Constant of kinetic reaction, $ml.mol^{-1}.min^{-1}$
 K = Constant of equilibrium reaction
 m = Water mass in water phase, g/mL
 m_0 = Initial water mass, g/mL
 M_B = Molecular weight of water, g/gmol
 t = Reaction time, minute
 V_m = Total volume of oil, mL
 x_A = Conversion of α -pinene
 x_A^* = Conversion of α -pinene at equilibrium
 x_i = Conversion of i-component
 γ_i = Activity coefficient of α -pinene in oil phase
 γ_w = Activity coefficient of water in oil phase

= Activity coefficient of α -terpineol in oil phase γ = Activity coefficient of α -component A
ACKNOWLEDGMENT The authors acknowledge Directorate General of Higher Education of Indonesia (DIKTI) through Doctoral Grant no.LPPM-UGM/1166/2009 for funding of this research. REFERENCES [1] Aguirre, M.R, De la Torre-Saénz, L., Flores, W.A., Robau-Sánchez, A., and Elguezabal, A.,

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