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21 DOI: 10.7763/IJCEA.2013.V4.253 ? Abstract —Tmajcooent tuentiis -pinene and the main hyratipdt a -pinen a -terpineol. To obtain the optimal process design of a -terpineol production, some initial information regarding the principle of the thermodynamics and phase equilibrium are required. The aim of this paper is to present liquid-liquid equilibrium (LLE) of sym ntnga -pinene, a -terpineol and water.

Ternary LLE a -pinene, a -terpineol and water system was determined by experiment at the temperatures of 301, 327 and 353 K and atmospheric pressure. The three component mixture was stirred for about 30 min, then the mixture was left for about 2 h for complete phase separation. The composition of both phases was analyzed by using a Gas Chromatograph. The thermodynamic equilibrium models were proposed. LLE model in this study includes activity coefficient model based on NRTL and UNIQUAC models. The NRTL model (a = 0.2) correlates the LLE data for the system of a -pine+a -terpineol + water at the temperatures 301, 327 and 353 K with RMSD of 0.5464%, 0.000169% and 0.0054%, respectively. The UNIQUAC model correlates the LLE data for the system of a -pine + -terpineol + water at the temperatures 301, 327 and 353 K with RMSD of 0.756%, 1.314% and 1.6615%, respectively.

The LLE data for the steof -pine -terpineol + water were successfully correlated using the NRTL and UNIQUAC models at that temperatures. Index Terms —a -Pinene, a -terpineol, liquid-liquid equilibrium. I. INTRODUCTION a -Pinene, one of the most widespread bicyclic monoterpenes, is a chiral compound obtained from turpentine. This compound which is used in the synthesis of a variety of chemicals, such as a -terpineol which is widely used as flavor in the cosmetic industry, as mineral flotation in the mineral benefit industry, and as antifungal and disinfectant in the pharmaceutical industry [1]. Therefore, the direct hydration of turpentine to synthesize a -terpineol has

an important value in industrial application.

During the process o f -terpineol production, some initial information regarding the principle of the thermodynamics and phase equilibrium are required. The design of separation processes requires information on phase equilibrium and related thermodynamic properties. However, information related to LLE involving terpenes and Herti Utami is with the Lampung University, Bandar Lampung, Indonesia. She is now Ph.D student at the Chemical Engineering Department, Gadjah Mada University, Indonesia (corresponding author to provide phone : +62-0274-9232121; e-mail: hertie19@ hotmail.com and hertie19@yahoo.com).

Sutijan, Roto, and Wahyudi Budi Sediawan are 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, roto05@ugm.ac.id, wbsrsby@indosat.net.id)). water is relatively scarce in the literature. LLE information fo -pine ne ?3caene plarcoo (acetonitrile, nitromethane, and dimethylformamide) systems has been studied [2]. Hengde Li studied the ternary LLE for water + ethano - pne + ß -pinene, or + limonene and quaternary L rwa ethal a -pinene + limonene systems at the temperature of 298.15K [3].

The authors also report data for ternary LLE for water + terpene + 1-propanol or 1-butanol systems at the same temperature [4]. Xiaoli Li studied the ternary liquid-liquid equilibrium for water + acne a - pnene,+ -pinene, or + limonene mixtures [5] and Ghizellaoui studied the LLE of water + 1-propanol + 1-pentanol system at 298.15 and 323.15 K [6]. Gramajo studied the LLE of water + linalool + limonene ternary system [7] and Bilgin studied the LLE of water + propionic acid + oleyl alcohol ternary system at several temperatures [8].

This paper reports on the experimental data on liquid-liquid equilibrium of ter narmieoa -pinene + a -terpineol + water at the temperatures of 301, 327 and 353 K. It is generally believed that LLE are influenced by several factors including the nature of the components, concentrations in both phases, and temperature of the system [9]. To predict LLE in multi component system, it needs adequate equilibrium model. This paper also reports the theoretical studies on this system. NRTL [10] and UNIQUAC [11] models are used to correlate the phase equilibrium in the system using the interaction parameters determined from the experimental data. II.

EXPERIMENTAL a -Pinene was purchased from Alfa Kimia, Yogyakarta, Indonesia. The content of a -pinene is 99% w/w (Aldrich) and a -Terpineol is 90% w/w, technical grade (Aldrich), distilled water was purchased from General Lab, Yogyakarta, Indonesia.

Liquid-liquid equilibrium was determined at 301 and 353 K temperatures. The three component mixture was stirred for about 30 min, then the mixture was left for about 2 h for complete phase separation. The composition of both phases was analyzed. Concentration of each component was determined by using a Gas Chromatograph (GC). The analysis was performed with 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 80oC, held for 5 min, increased at a rate of 5oC/min to 115oC and then increased to 280oC at a rate of Liquid-Liquid Equilibrium for System Composed of a -Pinene, a -Terpineol and Water Herti Utami, Sutijan, Roto, and Wahyudi Budi Sediawan M anuscript received October 2, 2012; revised January 14, 2013. 22 20oC/min. The injector and detector temperatures were set at 280oC respectively.

TABLE I: EXPERIMENTAL AND CALCULATED LLE DATA FOR THE TERNARY OF ? -PINENE (1) + ? -TERPINEOL (2) + WATER (3) SYSTEM AT 301 K Aqueous phase (x1) exp (x1)calc NRTL (ALPHA=0.68) (x1) calc UNIQUAC 0.000002225 0.000192424 0.001011309 0.000004267 0.000157599 0.000802614 0.000019032 0.000158519 0.000767702 0.000033192 0.000144116 0.000655445 0.000036191 0.000134829 0.000601017 0.000104414 0.000157205 0.000572298 TABLE II: EXPERIMENTAL AND CALCULATED LLE DATA FOR THE TERNARY OF ?-P INENE (1) + ?-T ERPINEOL (2) + WATER (3) SYSTEM AT 301 K Aqueous phase (x2) exp (x2)calc NRTL (?LPH =0.68) (x2) calc UNIQUAC 0.000010082 0.000044693 0.000283883 0.000024375 0.000054624 0.000301635 0.000021976 0.000055809 0.000312639 0.000040927 0.000071048 0.000342576 0.000052685 0.000077049 0.000337998 0.000126311 0.000116041 0.000329832 TABLE III: EXPERIMENTAL AND CALCULATED LLE DATA FOR THE TERNARY OF ? -PINENE (1) + ? -TERPINEOL (2) + WATER (3) SYSTEM AT 301 K Aqueous phase (x3) exp (x3)calc NRTL (?LPH =0.68) (x3) calc UNIQUAC 0.999987693 0.999762883 0.998704808 0.999971359 0.999787777 0.998895750 0.999958992 0.999785672 0.998919659 0.999925881 0.999784836 0.999001979 0.999911124 0.999788122 0.999060985 0.999769274 0.999726754 0.999097870 TABLE IV: EXPERIMENTAL AND CALCULATED LLE DATA FOR THE TERNARY OF ?-P INENE (1) + ?-T ERPINEOL (2) + WATER (3) SYSTEM AT 301 K Organic phase (x1) exp (x1)calc NRTL (?LPH =0.68) (x1) calc UNIQUAC 0.564726685 0.564700622 0.557989595 0.451498317 0.451399494 0.453578837 0.432219468 0.432120367 0.433870732 0.370363017 0.370340900 0.371675496 0.341529596 0.341561288 0.345625693 0.328045769 0.328101275 0.334104587 TABLE V: EXPERIMENTAL AND CALCULATED LLE DATA FOR THE TERNARY OF ?- PINENE (1) + ? -TERPINEOL (2) + WATER (3) SYSTEM AT 301 K Organic phase (x2) exp (x2)calc NRTL (ALPHA=0.68) (x2) calc UNIQUAC 0.318301276 0.318456456 0.311479336 0.345289912 0.345294882 0.343838283 0.358439524 0.358416912 0.356361307 0.395538159

0.395476427 0.392366941 0.394719656 0.394664687 0.395456963 0.390350616 0.390300587 0.394139561 TABLE VI: EXPERIMENTAL AND CALCULATED LLE DATA FOR THE TERNARY OF ? -PINENE (1) + ? -TERPINEOL (2) + WATER (3) SYSTEM AT 301 K Organic phase (x3) exp (x3)calc NRTL (?LPH =0.68) (x3) calc UNIQUAC 0.116972039 0.116842922 0.130531069 0.203211772 0.203305624 0.202582879 0.209341009 0.209462721 0.209767961 0.234098824 0.234182673 0.235957563 0.263750748 0.263774025 0.258917343 0.281603615 0.281598138 0.271755852 TABLE VII: EXPERIMENTAL AND CALCULATED LLE DATA FOR THE TERNARY OF ? -PINENE (1) + ? -TERPINEOL (2) + WATER (3) SYSTEM AT 327 K Aqueous phase (x1) exp (x1)calc NRTL (ALPHA=0.65) (x1) calc UNIQUAC 0.000008650 0.000035108 0.000023123 0.000009954 0.000031517 0.000020449 0.000038964 0.000046471 0.000019896 0.000058617 0.000054768 0.000018412 0.000064382 0.000051434 0.000016498 0.000072607 0.000046076 0.000015096 TABLE VIII: EXPERIMENTAL AND CALCULATED LLE DATA FOR THE TERNARY OF ? -PINENE (1) + ? -TERPINEOL (2) + WATER (3) SYSTEM AT 327 K Aqueous phase (x2) exp (x2)calc NRTL (?LPH =0.65) (x2) calc UNIQUAC 0.000000000 0.000025821 0.000641272 0.000000000 0.000025915 0.000642158 0.000001112 0.000026189 0.000635522 0.000010255 0.000031225 0.000662790 0.000025561 0.000039070 0.000660995 0.000105242 0.000108801 0.000666428 TABLE IX: EXPERIMENTAL AND CALCULATED LLE DATA FOR THE TERNARY OF ? -PINENE (1) + ? -TERPINEOL (2) + WATER (3) SYSTEM AT 327 K Aqueous phase (x3) exp (x3)calc NRTL (?LPH =0.65) (x3) calc UNIQUAC 0.999991135 0.999939071 0.999335605 0.999990046 0.999942569 0.999337393 0.999959924 0.999927340 0.999344583 0.999931128 0.999914007 0.999318798 0.999910057 0.999909496 0.999322507 0.999822151 0.999845123 0.999318476 III.

RESULTS AND DISCUSSION Liquid- liq uilrm rtery ysteo -pinene, a -terpineol and water was measured at 301, 327 and 353 K and atmospheric pressure. The experimental and calculated results were shown on Tables I to XVIII. Six effective binary interaction parameters are required for ternary system. The optimum NRTL and UNIQUAC binary interaction parameters were determined by minimizing the differences between the experimental and calculated mole fractions for each of the components over all the tie lines.

The Root Mean Square Deviations (RMSD) has been defined as follows: R M SD = 100 ?? ?????? -?? ???? 2 ?? =1 2 ?? =1 ?? ?? = 1 6 ?? 2 1 2 (1) whe re M is the number of tie lines, xexp indicates the expe rimental mol fraction, xcalc th e calculated mole fraction, and subscripts i, j and k denote, respectively, component, phase and tie line. It is expected that the RMSD can represent the goodness of fit. TABL E X: EXPERI MENTAL AND CAL CULATED LL E DA TA FOR THE TERNA RY OF ? -PI NENE (1) + ? -TE RPINEOL (2) + WAT ER (3) SYST EM AT 32 7 K Org anic phase (x1) exp (x1)calc NR TL (?LPH A=0.

65) (x1) calc UNIQUAC 0. 501655204 0.501662594 0.512785063 0.

445442353 0.445431259 0.446315119 0.434455541 0.434443370 0.433110660 0.402987883 0.402978593 0.399372670 0.363334566 0.363337914 0.355119378 0.334778001 0.334798850 0.324655959 TABL E XI: EXP ERIMENTAL AND CA LCULATED LL E DAT A FOR THE TERNA RY OF ? -PI NENE (1) + ? -TER PINEOL (2) + WATE R (3) SYSTE M AT 327 K Org anic phase (x2) exp (x2)calc NR TL (ALPH A=0. 65) (x2) calc UNIQUAC 0. 360829708 0.360847055 0.372298428 0. 365779133 0.365773277 0.369989707 0.363790691 0.363783360 0.366097753 0.381083792 0.381068561 0.381720314 0.384943674 0.384936712 0.380641529 0.392538688 0.392548579 0.385590983 TABL E XI I: EXPE RIMENTAL AND CA LCULATED LL E DATA FOR THE TERNA RY OF ? -PI NENE (1) + ? -TER PINEOL (2) + WATE R (3) SYSTE M AT 327 K TABL E XI II: EXP ERIMENTAL AND CALCULAT ED LL E DATA FO R THE TERNA RY OF ? -PI NENE (1) + ? -TER PINEOL (2) + WATE R (3) SYSTE M AT 353 K TABL E XI V:EX PERIMENTAL AND CA LCULATED LL E DATA FOR THE TERNA RY OF ? -PI NENE (1) + ? -TERP INEOL (2) + WATE R (3) SYS TEM AT 353K Aqueous phase (x2) exp (x2)calc NR TL (?LPH A=0. 64) (x2) calc UNIQUAC 0.

000000000 0.000000470 0.007237430 0. 000000000 0.000000496 0.007621110 0.000000000 0.000000508 0.007826990 0.000000000 0.000000533 0.008123779 0.000013844 0.000009768 0.008634235 0.000020252 0.000027933 0.009387390 TABL E XV: EXPE RIMENTAL AND CA LCULATED LL E DATA FOR THE TERNA RY OF ? -PI NENE (1) + ? -TER PINEOL (2) + WATE R (3) SYSTE M AT 353 K Aqueous phase (x3) exp (x3)calc NR TL (ALPH A=0. 64) (x3) calc UNIQUAC 0. 999997699 0.999989528 0.991438473 0. 999980818 0.999981738 0.991121494 0.999972789 0.999976316 0.990923330 0.999931208 0.999930826 0.990773679 0.999903678 0.999897281 0.990294727 0.999901560 0.999909967 0.989826272 TABL E XV I: EX PERIMENTAL AND CALCULAT ED LL E DATA FOR THE TERNARY OF ? -PIN ENE (1) + ? -TERPI NEOL (2) + W ATER (3) SYSTEM A T 353 K Org anic phase (x1) exp (x1)calc NR TL (?LPH A=0. 64) (x1) calc UNIQUAC 0. 451655204 0.451656189 0.455993227 0.

429172353 0.429172818 0.430126648 0.426216241 0.426216366 0.421906815 0.377987883 0.377987829 0.382681678 0.366677111 0.366676270 0.361239026 0.271290444 0.271289167 0.276134517 TABL E XV II: EXP ERIMENTAL AND CALCULAT ED LL E DATA FOR THE TERNA RY OF ? -PI NENE (1) + ? -TER PINEOL (2) + WATE R (3) SYSTE M AT 353 K Org anic phase (x2) exp (x2)calc NR TL (?LPH A=0. 64) (x2) calc UNIQUAC 0. 338416677 0.338417471 0.340612301 0. 356664246 0.356664699 0.355651610 0.365543443 0.365543787 0.359279289 0.383345147 0.383344968 0.386051510 0.405789459 0.405788978 0.395786865 0.448373084 0.448371495 0.452642847 TABL E XV III: EX PERIMENTAL AND CALCULAT ED LL E DATA F OR THE

TERNA RY OF ? -PI NENE (1) + ? -TER PINEOL (2) + WATE R (3) SYSTE M AT 353 K Org anic phase (x3) exp (x3)calc NR TL (ALPH A=0. 64) (x3) calc UNIQUAC 0. 209928119 0.209926340 0.203394472 0. 214163401 0.214162483 0.214221742 0.208240315 0.208239847 0.218813896 0.238666970 0.238667203 0.231266812 0.227533430 0.227534753 0.242974109 0.280336472 0.280339338 0.271222636 TABL E XI X: THE VOLUME AND SUR FACE AREA PA RAMETERS FOR THE UNIQ UAC MODE L a -Pinene a - Terpineol Water r 6.056 7.0389 0.920 q 4. 760 5.8800 1.400 In the UNIQUAC model, the values of volume and surface area parameter of pure component ri and qi, for water and a -pinene have been taken from literature [3]. The volume and surface area of the a -terpineol was estimated from the Bondi's method [12].

The pure-component molecular parameters, ri and qi were listed in Table XIX. In the NRTL model, the values of the non randomness parameter (a) was set as 0.2 and obtained from fitting. The values of interaction parameters for the NRTL and 23 UNIQUAC models at different temperatures are shown in Tables XX to XXIII. The deviation between experimental and calculated values, expressed in terms of the root mean square deviations (RMSD) defined by using (1).

TABL E XX: INTE RACTION PARA METERS OF THE NRTL MODEL FOR THE TERNA RY OF ? LPH A-PINE NE (1) + ? LPHA-TERPI NEOL (2) + WA TER (3) SYSTEM AT 301 K Parameters of NRTL a (alpha) pairs bij (Jmol-1) bji (J mol-1) RMSD (%) 0. 2 1-2 4363.2449 -323.2558 0.5464 1- 3 7964.4715 7994.0665 2-3 -1952.7141 8490.8635 0.68 1-2 -213.6191 662.6515 0.0233 1-3 4777.8387 5141.9908 2-3 5306.6406 5878.5794 TABL E XX I: INT ERACTION PARA METERS OF THE NRTL MODEL FOR THE TERNA RY OF ? LP HA -PIN ENE (1) + ? LPHA -TER PINEOL (2) + WATE R (3) SYSTEM AT 327 K Parameters of NRTL a (alpha) pairs bij (Jmol-1) bji (J mol-1) RMSD (%) 0. 2 1-2 -45041.503 27272.681 0.000169 1- 3 48895.3203 31335.093 2-3 0.5576 -7563.4117 0.65 1-2 488.5559 -309.3192 0.000049 1-3 5652.4886 7305.7548 2-3 8197.413 7314.2554 TABL E XX II: INT ERACTION PAR AMETERS OF THE NRT L MODE L FOR THE TERNA RY OF ? LP HA -PIN ENE (1) + ? LPHA -TER PINEOL (2) + WATE R (3) SYSTEM AT 353 K Parameters of NRTL a (alpha) pairs bij (Jmol-1) bji (J mol-1) RMSD (%) 0. 2 1-2 -67.306 90877.4904 0.0054 1- 3 60890.8031 34522.7342 2-3 25926.1675 38171.0575 0.64 1-2 6876.9054 4744.9432 0.0008 1-3 16042.7182 8985.6788 2-3 43413.1749 10791.1033 TABL E XX III: IN TERACTION PA RAMETERS OF THE UN IQUAC MO DEL FOR THE TE RNARY OF ? LPHA-PINEN E (1) + ? LPH A-TERPIN EOL (2) + WA TER (3) SYSTEM Parameters of UNIQUAC Te mperature, K pairs uij (Jmol-1) uji (J mol-1) RMSD (%) 301 1-2 -885.2238 878.8773 0.7560 1- 3 4359.9185 498.7212 2-3 -1207.1664 6368.9558 327 1-2 -318.9388 -528.671 1.3140 1-3 6464.8237 90.6234 2-3 675.4101 -164.7618 353 1-2 -1079.2635 267.9923 1.6615 1-3 19890.7311 -469.4888 2-3 -223.578 90.5193 The e xperimental tie lines data and the correlated results of the ternary LLE of the a -pinene, a -terpineol and

water system at 301, 327 and 353 K in terms of NRTL and UNIQUAC models are shown in Fig. 1, 2 and 3. Fig . 1.

Experimental and calculated LLE data of the a -pinene + a -terpineol + water system at T= 301 K. (O experiment; ? NRTL model; x UNIQUAC model) Fig. 2. Experimental and calculated LLE data of the a -pinene + a -terpineol + water system at T= 327 K. (O experiment; ? NRTL model; x UNIQUAC model) Fig . 3. Experimental and calculated LLE data of the a -pinene + a -terpineol + water system at T= 353 K. (O experiment; ? NRTL model; x UNIQUAC model) Fig . 4. Experimental and calculated LLE data of the a -pinene + a -terpineol + water system at T= 301, 327, and 353 K.

(O experiment; ? NRTL model ; x UNIQUAC model ; 301 K; 327 K; 353 K) 24 In fitting the models to the ternary LLE data, the two models well represented the ternary system. The NRTL model (a = 0.2) correlates the LLE data for the system of a -pinene + a -terpineol + water at the temperatures 301, 327 and 353 K with RMSD of 0.5464%, 0.000169% and 0.0054%, respectively. And the NRTL model (a = 0.68) at the temperature 301 K with RSMD of 0.0233%, the NRTL model (a =0.65) at the temperatur e 327 K with RSMD of 0.000049% and the NRTL model (a =0.64) at the temperature 353 K with RSMD of 0.0008%, between the experimental and calculated mole fractions.

The UNIQUAC model correlates the LLE data for the system of a -pinene + a -terpineol + water at the temperatures 301, 327 and 353 K with RMSD of 0.756%, 1,314% and 1.6615%, respectively. The resu lts show that the RMSD for the ternary systems are less than 2%. In Fig. 4 the slope of tie lines changes slightly with the different temperatures. The calculated points are relatively very close to the experimental ones, so it proves that the models proposed can well approximate the equilibrium data. IV.

CONCLUSI ON Th e experimental data on liquid-liquid equilibrium of t ernary mixtures of a -pinene + a -terpineol + water were obtained at the temperatures of 301, 327 and 353 K. The NRTL and UNIQUAC models were used to correlate the experimental LLE data. The optimum NRTL and UNIQUAC parameters were determined using the experimental liquid-liquid data. It was found that both the NRTL and UNIQUAC methods fitted satisfactorily to the experimental data. The results show that the RMSD for the ternary systems are less than 2%.

NOTIFICA TION bij interaction parameter in the NRTL equation uij in teraction parameter in the UNIQUAC equation LLE liquid-liquid equilibrium NRTL non random two liquid T temperature (K) UNIQUAC universal quasi chemical equation x composition in mole fraction a (alpha) non randomness parameter in the NRTL equation ACKNOWL EDGMENT Fin ancial support from the Directorate General of Higher Education of

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Prausnitz, “Statistical thermodynamics of multi component liquid mixtures: A new expr ession for the excess Gibbs energy of partl y or completely miscible systems,” AIChE J., vol. 21, pp.116, 1975. [12] A. Fredenslund, J. Gmehling, and P. Rasmussen, Vapor-Liquid Equilibria Using UNIFAC, Elsevier, Amsterdam, 1977. Herti Utam i is Ph.D student at the Chemical Engineering Department, Gadjah Mada University, Indonesia. She also serves as a faculty member at the Chemical Engineering Department, Lampung University, Indonesia. In r ecent years, she focuses on the kinetic and thermodynamic study on the reactive distillation for a -terpineol production from turpentine.

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