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Tan Catalysis and Reacdon Bngineering Code Tideofpaper/Author 3Pro'cRE1 Kinetics Modeling forsynthesis of rerpineol from Turpendne Her'ti Utaml, Arief Budinran, Sotii"q Roo, Wahyudt Budi Scdiawan 3Pro'CRE2 The Effect ofUnburned Carbon on Coal FlyAsh Toward CrIVI] Adsorption Cnpacfty Widt Astuti I Made Bendiyasq Endang Trt Wahyuni and Agus prasetyo 3Pro'CRE3 Kinetic Sbdy on the Hydrrolysis of Waer Hyacinth to levulinic Acid Buana Girisuta, L P. B. M.lansseU H, J, Heeres 3Pro'CRES Aerobic Oxtdadonof Benryl Alcohol Ustng Molecutar Ox1ryen OverSurfrce-Modified Tud-l Supported palladium Caalysts Yuanting Che4 yanhui yang 3Pro{RE12 Synthesk of Nanosized Platinum Catelysts for Cinnamaldehyde Hydrogenation Reacdon Qwlka Krupro*rt, Choowo4g Chahuk Akorn Meka*lwandurarong 3Pro'CREl3 Immobilization of Phosphoramidite tigands-Rh Complexes on SBA-1S and its 241 Page 243 246 250 254 257 266 275 279 282 26L 271 Page 286 290 294 297 302 306 f.g,_ ''f ASEAN REG/,o/.LsvaPosluM oN c,IEMIuL ENGINEER,NG Df'rcmber 7-2,2qP, Monlta Hof.et, Phlllpfines tPro-CREt Kinetics Modeling for synthesis of Terpineol from Turpentine Herd Utamlrl, Arlef Budlmanl, Sudfanr, Roto2, Wahyudt Budl Sediawanl tChemlcal Engineering Departnent, Gadjah Moda llnlvercily, jl.Grafika No.2

Yogyakarta, lndonesia zChemlstry Departmen| Gadjah Mada llniver,ier, Yogyakarto, Indonesia Email address: hertiel 9@hohna ll.co m ABSTRACT:Turpc[t|ne|soncoftheessendaloi|sobtalnedfromp|netreeandt"""".y pharmaceutical industrlcr and In processlng of ollq resins and varnl:;hes. Substantlal part oi pine tree of the forest is-regularly tapped an4 processed to produce gum, rosln" and turpendne.

Most of plnes ln tndonesla are plne mercusii specleg whlch typlcally produce turfenttni thai contalns of about 82% alpha plneng 12% delta carenc and balanced wlth othe: nurnerous components such as canrpheng beta-pinene ani limonun". tn order to obtaln more valuable productt, th€ dpha plnene In the turpcndne can be hydrated ln dllute mlneral acld iolutions to iroduce terplneol, which can be used us perfume, repellent of InsecL andfungal and dlsinfecrant In thls work a kinetics model for synthesis of terpineol from turpentine was developed to quandtatively descrlbe effects of hydratlon process rtf

alplra ptnene ln aqueous acid solutlon, Thc results of this study show that kinetics mo4eilng of the hydratton of alpha plnene uslng both chloro acetlc acid and oxatic acid as catalyst could be approached with the heterogeneous model.

11re constants of drc reacdon rate for the flrst method rvere kr = 13,2476 and ki = 8,6836x10o3 rnl.mol-r.min-t for chloro acetlc acld, a-nd k, = 17,0005 and ft1' = 7,3042x10'@ mlmol't.mln{ fur oxatlc ac'id, The sum of sqoares of ermr of the first and the last catalysts were 0, 3223 96 and O1627%, respcctively.

T[re constants of the rcaclion rat€ for the second method were kr = O,03130 and ki = 0,01239 ml.mo]r.min.r for chloro acetic acid and kr =O0267andki=0,04198ml.motr.mln'rforoxallcacid. Thesumofsquaresoferrorofthefirstandthelastcatatystswere0,35g6%and0,02653%, respectively. Ke5morde urrpendnc, terplneol, hydrado[ ldnedcs TNTRODUCTION urpentine is distilled from oleo resin obtained from various I species of pinus.

lt is colorless and transparent oily liquid with a strong specific odor and a pungent bitter taste, It is insoluble in water but soluble ln alcohol, etheo chloroform and carbon disulphite ( Pandhejt K,et al, 1994,). The composition of different hrrpentines depends on the species of pine from which they are extracted. Most of pines in Indonesia are pine mercusii species, which typically produce turpentine that ccnains of about 82016 alpha pinene, 12% delta carene and balanced with' other numerous Gomponents such ag camphene, beta-pinene and limonene.

In order to obtain more valuable products, the alpha pinene in the turpentine can be hydrated in dilute mineral acid soludons b produce terpineol, which can be used as perfume, repellent-ofinsecgentifo ngal and disinfefi nt Alpha pinene is tlte main consdhrent of turpentine oil. The acid-catalyzed hydradon and isomerization of alpha-pinene yields a complex mixture of monoterpenes, alcohols, and hydrocarbons The rnain products are alpha- terpineol, limonene, and terpinolene.

Minor amounB of camphene, alpha and gamma- terplne4g alpha arrd beta-fenchol, isoborneol, borneol, gamma- terpineol, and l,8-terpine are also formed (Mqnteiro, l.LF., 2004) Alpha-terpineol is one of the most important monocyclic monoterpcnic alcohols and one of the top 30 commonly used flavor compounds. It is produced on an industrial scale by hydration of alpha-pinene or turpentine oil to the cis-terpin hydrate with aqueous mineral acids, followed by its partial dehydratlon to alpha-terpineol (Bauer, K, 1985). This process presents some difficulties such as the production of complex mlxtures and the disposal of mineral agids.

Hydration and isomerizadon of alpha-pinene producing alcohols and terpenic hydrocarbon had been studied since 1947. ln 1947, Mosher shrdied the product distribution of acid- cahlyzed alpha-pinene hydration products. Tlre author brought to the fore the Featment of alpha-pinene with 1-cNoro-4- naphthalene sulfonlc acid which led to the formation of beta- pipene, limonene, terpinoleng and alpha-terpinene. The hydration/isomerization of alpha-pinene at 329 K catalyzed by zeolite H-beta (Van der Waal, f.C.,

1996) is fasr and leads mainly to monocyclic terpenes and alcohols with alpha-terpineol as the main product (up to 48%J. The selectivity toward the commercially interesting bicyclic products (such as borneol and camphene) is about 26Yo, which is significantly bettel than that observed when HzSOr is used as catalysL The reaction rate increases with increasing Si/Al ratio, which is possibly due to the increase of hydrophobicity of the zeolite; the selectivities are, however, not significantly affected (Van der Waal, I.C.,

1996) Pakdel et al (2001) used sulphate acid as catalyst to synthesize terpineol from turpentine, in the presence of excess acetone as solubility promoter. They reported 67V6 of selectivill to terpineol although the conversion was not reported. Aguirre eL al (2005) used hydrochloride acid, asetate acid, chloro acetic acid and oxalic acid as catalyst for the hydration of alpha-pinene.

Choro acetic acid was fotrnd as good catalyst for the production of terpineol from pinene.The highest selectivity was 95,5% with the conversion of 107o, whereas the higher conversion was 99% with selectivity of 69Va after 4 h of reaction at 70oC. The reaction is schemadcally shown in figure 1: Hzo + -t..- o" Flgure l.The reaclon of alpha-plnene terplneol MATERIALSAND METHODS A simulation taking into account a heterogeneous model kinetics is performed utilizing MATLAB 7.

Experirnental data were obtained from the paper of Aguirre, et al.(2005) in which hydration witl several kinds of catalyst (hydrochloride aci4 acetate acid, chloro acetic acid and oxalic acid) had been I O+ lpha pinene H' g..--l||G*a Synthesis of Te rpineol.... SProCRET done. The tempcrature of hydration was Z0oC and 6,4 mol/L catalyst concentration was applied.

The reaction time str-rdied was in the rangc of 25 to 240 minutes, Thc conversion was in tie nnge 0,27 w 4,62 for chloro acetic acid, and in the range 0,19 to 0,27 for oxalic acid. This paper reports the kinetics modeling of hydration using chloro acetic acid and oxalic acid as catalysl . RESULTSANDDISCUSSION A heterogeneous kinetics model for synthesis ofterpineol from turpentine was developed to quantitatively describe effects of hydration process of alpha pinene in aqueous acid solution. Turpentine is a.ssumed to

be insoluble in water. The bllowing assumption were applied : 1.

The reaction take place in the oil phase. ?. 'l'he liquid fllm thickness is very small. 3. The rea*ion in liqutd film is negligible and the oil does not diffuse into the water phase. I(nedcs Model Mass balance water (B) in the water phase dm dt = krMsA.(Cin, - Ca^) Mass balance water [B] in the oil phase : o'!" =g- 1: + kr cAmcrm - krCr^ dc dt ,ilRvm Mass balance alpha-pin€ne (A) in the oil phase: ry: krcAmcat - krcr^ dt dt uavm ' d,t Boundary condition : t= 0 ; m = filo i CBm = 0 ; Ca- = Cemo Adjuiable parameter : [kAc), C* sm , kr and kr' The kinetics model is then solved using Runge Kutta method @/ITLAB 7). b'valuation of the parameters were conducted in the two methods.

The Hrst method was determining the values of (kA"), C* am , kr and krty curve fitting methotl, where the srm of squares of errors (SSE) was minimized. The generated profiles are depicted in fig. 2 to 5. Fig. 2 and,4 show that the alpha pinene concentration decreases with the increase ofreaction time. The conversion increascs i,fith the inLTeasc of reaction time [Fig.3 and 5). The constants ofthe reaction rate were found to be kr = t3,2476 and kr' = 6,8836x10.03 ml.mol-r.min-1 for chloro acetic acid, and kr = 17,0005 and ki = 7,3042xL0-o2 ml.mol-r.min-1 for oxalic acid.

The sum of squares of error of the first and the last catalysts were 0, 3273 Vo and 0,L627o/o. Fig. 2 to 5 show that the kinetics model proposed can quantitatively describe tlte hydration ofalpha-pinene using chloro acetic acid and oxalic acid as catalyst 50 tm r50 200 Time, minute Figure 2. Concentration Profitc for Chloro Acetic Acid Catalvst JU --L_... -_..-__!* lm 150 Time, mirute o.roj, a.rA t\ 0.22f \ o "ri ".,

E o.1sr ^ g o.'o|. ,l 0.r{[ o.12l I o'l o.o8 r i 0.6l- I o.r f I s o"l al xl O.3f / l, "/' orl i ,,/ o.ti / t/ li r8-----__ i ;.*ii , 1\ Da6 .] l I I i I I I I I I l------- Model l-:,es- "-l'\ o,zr | \ t\ ''i \ o.zz l- \ i\ 0.21 [ \ l l a. o.2 | l ''*i o.r"L-- - - Mo(bl O Data (1) t2) t3) (4) Figure 3. Conversion prollle for Chloro Acetic Acid Catalyst \--- --- ' - --ir- 50 100 1S0 mo -fime, mlrutr E E E U 287 Figure 4, Concentration Profile for Oxrtic Acid Catalyst F,*-, Sf!. Sy nthesls of Terp i neol.... ithoCtlEl E 5 0.26;--- t ;:.- rr - 'r I o'2rl\ L' *" ll o..l\ t\ 1 0.21- \ | t\ l o.rsl- a I l\. I 0.16l- \ | l\.tol o.r.i

\ .l | \--\- | o.r2l .-=a__ I l-..---."----.-.-.-.-....-...-_l o.lf I I al o'*o'_----6-- - - - fi- ---fi 19) ux) Ftgure 5. Gonrcrdon *ot" *ffidd cetehEr ln the second metho4 the values of Cr an and K were approxlmated frrom the available daa (Aguirre, et a1.,2005) at equilibrium conditioq appro:dmately 240 minutes ( ri = OS+) for chloro acedc acid and 6O minutes ( rj = eZ5) foi oi<alic acia, respectively.

The stolchlometrlc equadonlmples' : Cim= Ct^o(l - ri) Ciz.= Ct*ri Ci^= Ce^o - Cn-oxi

where, 9= fi - .qi" 11 Ci-ci- ftnq r{]r b Flgure 6, Conccntradon proflIc for Chloro AccdcAcId C.talyst Flgure 7. Converslon kof,le for Ghloro Accdc AtId Cat lrEt ts) t6) (7) t8) Base! on, lhe approxlmated values of xj from the approdmaed data dre vdues of Csrt'and K can becalculated by equadons [5), (6r, (n and (8).

tt turned out that for chloro acetii aGid, C3''r'= 0,465 mol/ml and K = Zj2S, while for oxalic acId, Cs6' = 05364 moyml and K = e636. Stnce C6-. and K have been catcutate4 the adfustable parameter yariables are then (kcA:) and la. The naluesof parameter (kcA.), and kr were determined by the same medlo4 wherc theiumcf squares of enon (sSE) was mtnimized. Fig.

6 shows that the alpha pinene concentradon decreases wlth the lncrease of reacdon time for chloro accdc acId catal'6L The slmilar trend also occrrns on the lig. 8 for oxalic ackl catalyst The constants of the reactlon rate were kI = 0,03130 and kr'= 0,01239 ml.mol.r.min.r for chloro acedc acId, and lq = 0,0267 and kr'= 0,04198 ml.molt.mln-r for oxalic acid, wherras, the sum of squales of ernor of the first and $e last catal!,stc were O3596 96 and 0,02653%, respecdvely.

Flgures 6 to 9 show the similar results in which the HneUcs model prcposed can.quantltadvety describe the hydration of alpha-plnene tsing chloro acetic acid and oxalic acid as catal'rst I ne parameters evaluation In the second method was simpler than in'the first method.

It is suggested tlre values obtained by the second method weFe mone accurate since less adjustable palameters applied and the values of Cnt'and K were directly evaluated from the experimental data. E $ Tln€, m|nrE Flgure & Cono:nb-adon proflle forOxallcAdd 200 Catalyst l:,'r'lil :l: ::; ,.:':, f.il}ggt ffi 288 Synthesisc!ferPing- I I l SPttCgEt TkrE, mrr.(c Figure 9.

Convcrslon Prof,le for Oxalic Acld Catatyst CONCLUSIONS The results of this shrdy show that Hnedcs modeling of the hydration of alpha pinene usingboth chJoro-acetic acid and oxalic acia as catalyst could be approached wlth the heterogeneous modei, Evaluatiou of parameters for the heterogeneous model were conducted in the two methods, The constants of the reaction rate for the first method were kr = 13,2476 and kr' = 8,8836x10or mlmokr.min't for chloro acetic add, and kr = 17,0005 and kI = 7,3}42x1$oz pl.6eF.min'1 for bxalic acid' On the other hand, the sum of squares of error of the first and the last catalysts were 0, 3273 Vo and O,L627VI.

The constants of the r€acdon iate for the second method were kr = 0,03130 and kr' = 001239 ml.mol'1'min-1 for chloro acetic acid, and kr = 0,0257 and le'= Q04198

ml,rnolr.min-r for'oxalic acid. While the sum of squares of error of the first and the last catalysts were O,3596 Vo anA O,OZO5g%, respecdvely. The parameter evaluation in the second.

method was simpler than in the _ first method' lt is suggesied the values obtained by the second method were more aciirate slnce less adiustable Parameters applied and the values of Cs6' and K were directly evaluated fronr the experimental data. NOTATION Ac = Mass transfer area = total area lnterface, cmz Ce- = Concentration alpha-pinene in oil phase, mol/ml Cs.

= Water concentration In oil phase, mol/ml CAmo = Inltlal of concentration alpha-pinene in oil phase, mol/mL Csmo = Inidal of water concentration in oil phase, mol/ml Cr' = Terplneol concentration in oil phase, mol/ml Cum'= Water concentration in equilibrium, mol/mL Ca'' = Alpha-pinene concenFation in equilibrium, molfml Cr.'= Terpineol concentration in eqrrilibrium, mol/ml k" = Mass transfer coeflicient kr = Constant kinetic reaction, ml'mol'1.min-r kr' = Constant kinetic reaction, ml.mol-l.min-l K = Constant equilibrium reaction m = Water mass in water phase,gr/mL mo = lnidal of water mass,gr/mL Ms = Molecular weight water,gr/gmol t = Time reaction, minute Vm = Total volume of oil, mL xr = Conversion :<e' = Conversion atequilibrium REFERENCES Aguirre, M.&

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