

# Equilibrium Study on Reactive Extraction of Lactic Acid with Tri-*n*-Butyl Phosphate in *n*-Hexane

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Extraction of carboxylic acids from dilute aqueous solution using traditional solvents such as ketones, alcohols, ethers, and ester is inefficient because the distribution ratio is rather low. Reactive extraction which exploits reversible chemical complexation is an effective separation process for extraction of carboxylic acids from aqueous streams such as fermentation broths and wastewaters. In the extraction process, selection of the solvent is an important aspect to be considered. Considering its solubility in water, cost and availability, tri-*n*-butyl phosphate (TBP) seems to be an attractive solvent for the extraction of lactic acid from aqueous solution.

The purpose of this experiment is to study the equilibrium of the reactive extraction of lactic acid in aqueous solution with TBP in *n*-hexane. The parameters studied in this experiment were initial concentration of lactic acid in the aqueous phase, TBP concentration in *n*-hexane phase, and the extraction temperature. The experiments at ambient temperature were carried out using a separatory funnel, while the experiments at other than ambient temperature were carried out using erlenmeyer flask and water bath shaker to adjust the temperature. In this experiment, the initial concentration of lactic acid was varied from 0.1 to 0.5 gmol/dm<sup>3</sup>. The range of initial TBP concentrations in *n*-hexane was 0.1 to 1.0 gmol/dm<sup>3</sup> and the extraction temperature range was 283 to 313 K.

The experimental results showed that the higher the initial concentration of lactic acid in aqueous solution, the higher the distribution ratio for a fixed TBP concentration and extraction temperature. For a fixed initial concentration of lactic acid in aqueous solution and extraction temperature, the distribution ratio of lactic acid is increased by increasing TBP concentration. The overall equilibrium constants ( $K_{pq}$ ) for the experiments using TBP concentration ranging from 0.1 to 1.0 gmol/dm<sup>3</sup> at the extraction temperature of 293 K are calculated to be 0.0668 to 0.5144.  $K_{pq}$  for the experiments at the temperature ranging from 283 to 313 K at the initial concentration of lactic acid of 0.2 gmol/L are found to be 0.0122 to 0.8856. The  $K_{pq}$  as a function of temperature (T) in K can be expressed as  $\ln K_{pq} = 10,596/T - 38.08$  with sum of square of error of 0.14.

**Keywords:** Reactive Extraction, Equilibrium, Lactic Acid, TBP, *n*-Hexane

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## INTRODUCTION

Production of carboxylic acids by fermentation usually produces low concentrations of carboxylic acids in water which is sometimes less than 10% (w/w) (Tamada and King, 1990). Separation, purification and even to concentrate the fermentation products are very difficult because affinities between acids and water is very strong. Separation of acids and water by distillation is energy consumable to evaporate water (Tamada and King, 1990). The classical process to recover a carboxylic acid from a wide variety of dilute aqueous solutions is based on precipitation of the calcium salt upon addition of calcium hydroxide to the acid aqueous solution (Malmay *et al.*, 1997). This method of recovery results low yield of acid crystallization, costly, and unfriendly to the environment (Juang and Huang, 1994; Poposka *et al.*, 1997).

To recover carboxylic acids from dilute acid solutions can be conducted by extraction method. Traditional solvents which are usually used are ketone, alcohol, ether and ester. Using traditional solvent is not very effective because its distribution ratio is very low. To increase the distribution ratio, reactive extraction is one of the method used to recover carboxylic acids from dilute solutions (Juang and Huang, 1997). Many researchers have investigated reactive extraction of carboxylic acids using some extractants. Juang and Huang (1997) has studied the equilibrium of reactive extraction of lactic acid with an amine extractant. Synergistic extraction of lactic acid with tri-*n*-octylamine and TBP has been studied by Matsumoto *et al.* (1998).

This paper presents the equilibrium of reactive extraction of lactic acid with TBP in *n*-hexane. TBP reacts with lactic acid to form complexes, while *n*-hexane functions to dilute TBP. TBP has an advantage as solvent for use in aqueous extraction due to its negligible solubility in water (Shah and Tiwari, 1981).

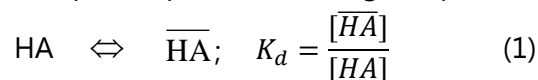
## MATHEMATICAL MODELING

### Extraction of Lactic Acid with Pure *n*-Hexane

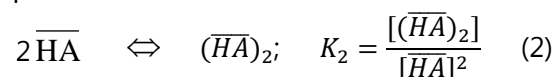
Most carboxylic acids such as lactic acid mainly exist as dimers in organic phase due to the strong intermolecular hydrogen-bonding, especially in the non-polar or lowly polar solvents (Juang and Huang, 1997). On the contrary, in the aqueous phase, they exist mainly as monomers because the intermolecular hydrogen bonding between the acids is destroyed owing to their preferential hydrogen bonding with water molecules (Juang and Huang, 1994; Juang and Huang, 1997).

The reactions in this extraction as the aqueous pH is far less than the dissociation constant of lactic acid ( $pK_a = 3.86$ ) are as follows (Juang and Huang, 1997):

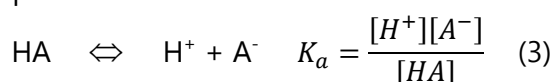
Distribution of lactic acid monomer from the aqueous phase to the organic phase.



Dimerization of lactic acid in the organic phase.



Dissociation of lactic acid in aqueous phase.



The overbar refers to the organic phase. The concentrations of lactic acid in organic phase and aqueous phase at equilibrium are calculated from the following equations:

$$\overline{[HA]}_t = \overline{[HA]} + 2[(\overline{HA})_2] \quad (4)$$

$$[HA]_t = [HA] + [A^-] \quad (5)$$

The distribution ratio ( $D_0$ ) of lactic acid in the absence of TBP is defined by Eq. (6).

$$D_0 = \frac{\overline{[HA]}_t}{[HA]_t} \quad (6)$$

where  $\overline{[HA]}_t$  is the total concentration of lactic acid in the organic phase and  $[HA]_t$  is the total concentration of lactic acid in the aqueous phase at equilibrium.

The concentration of lactic acid in aqueous phase at equilibrium is determined by titration using NaOH solution with phenolphthalein as indicator, while its concentration in the organic phase is calculated by mass balance as follows:

$$[HA]_t V_0 = \overline{[HA]}_t V_{org} + [HA]_t V_a \quad (7)$$

Substitution of  $\overline{[HA]}_t$  from Eq. (4) and  $[HA]_t$  from Eq. (5) to Eq. (6) we have the following equation :

$$D_0 = \frac{\overline{[HA]} + 2[(\overline{HA})_2]}{[HA] + [A^-]} = \frac{\frac{\overline{[HA]} + 2[(\overline{HA})_2]}{[HA]} + \frac{2[(\overline{HA})_2]}{[HA]} \frac{[\overline{HA}]^2 [HA]}{[\overline{HA}]^2 [HA]}}{\frac{[HA]}{[HA]} + \frac{[A^-] [H^+]}{[HA] [H^+]}} \quad (8)$$

or

$$D_0 = \frac{K_d + 2K_d^2 K_2 [HA]}{1 + \frac{K_a}{[H^+]}} \quad (9)$$

The value of  $[HA]$  at Eq. (9) can be obtained from Eq. (5) and at the measured equilibrium pH, that is  $[A^-] = [H^+]$ , so that:

$$[HA] = [HA]_t - [H^+] \quad (10)$$

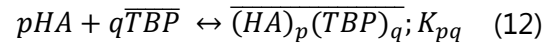
Eq. (9) can be arranged as:

$$D_0 \left(1 + \frac{K_a}{[H^+]}\right) = K_d + 2K_d^2 K_2 [HA] \quad (11)$$

By plotting  $D_0 \left(1 + \frac{K_a}{[H^+]}\right)$  versus  $[HA]$ , we get the value of  $K_d$  as the intercept of the graph and  $2K_d^2 K_2$  as its slope. The values of  $K_d$  and  $K_2$  can also be obtained by linear regression method.

### Extraction of Lactic Acid with TBP

The extraction of lactic acid with TBP in *n*-hexane at pH far less than  $pK_a$  can be expressed by the following stoichiometry (Juang and Huang, 1997):



where  $K_{pq}$  is the extraction equilibrium constant, which is given by Eq. (13).

$$K_{pq} = \frac{[(HA)_p(TBP)_q]}{[HA]^p [TBP]^q} \quad (13)$$

The total concentration of lactic acid in the organic phase can be expressed by Eq. (14) or Eq. (15) as follows:

$$\overline{[HA]}_t = \overline{[HA]} + 2[(\overline{HA})_2] + \sum_p \sum_q p [(HA)_p(TBP)_q] \quad (14)$$

$$\overline{[HA]}_t = K_d [HA] + 2K_2 K_d^2 [HA]^2 + \sum_p \sum_q p [(HA)_p(TBP)_q] \quad (15)$$

Eq. (15) shows that the amount of lactic acid complexed with TBP is corrected by subtracting that physically extracted by pure *n*-hexane from the measured  $[\overline{HA}]_t$ . The values of  $K_d$  and  $K_2$  in Eq. (15) are calculated from the experimental data of the extraction of lactic acid using pure *n*-hexane. The value of  $[HA]$  is calculated by Eq. (10) and the value of  $[\overline{HA}]_t$  is calculated from the total mass balance. So the value of  $[(HA)_p(TBP)_q]$  can be calculated as follows:

$$\begin{aligned} [(HA)_p(TBP)_q] = \\ \frac{1}{p} [\overline{HA}]_t - K_d [HA] - 2K_2 K_d^2 [HA]^2 \end{aligned} \quad (16)$$

The value of  $[\overline{TBP}]$  in Eq. (13) is obtained from the TBP mass balance as expressed by Eq. (17).

$$\begin{aligned} [\overline{TBP}] = \\ [\overline{TBP}]_0 - \sum_p \sum_q q [(HA)_p(TBP)_q] \end{aligned} \quad (17)$$

Knowing the values of  $[(HA)_p(TBP)_q]$ ,  $[HA]$ , and  $[\overline{TBP}]$ , then the value of  $K_{pq}$  in Eq. (13) can be obtained. In this calculation, both number of lactic acid molecules involved in the complex ( $p$ ) and number of TBP molecules involved in the complex ( $q$ ) are assumed to be 1.

The complexation reaction between lactic acid and TBP in the organic phase is expected to be exothermic. Formation of complex makes the system more ordered and thus decreases the entropy ( $\Delta S$ ). Therefore, as the temperature ( $T$ ) is increased, the amount of lactic acid extracted decreases. If the enthalpy ( $\Delta H$ ) and entropy of reaction are assumed to be constant over the temperature range, the

following expression:

$$\ln K_{pq} = \frac{-\Delta H}{RT} + \frac{\Delta S}{R} \quad (18)$$

indicates that a plot of  $\ln K_{pq}$  vs  $1/T$  gives a straight line (Tamada and King, 1990). The enthalpy of reaction is obtained from the slope, and the intercept is proportional to the entropy.

## EXPERIMENTAL

### Reagent and Solutions

Distilled water with the density of 0.9959 g/cm<sup>3</sup> and viscosity of 0.0089 g/(cm.s) was used in this experiment. Lactic acid, TBP, *n*-hexane, and other chemicals were supplied by Merck Co. as analytical reagent grade, and all were used without further purification.

### Experimental Procedure

The experiments were done for the extraction of lactic acid with pure *n*-hexane and for the extraction of lactic acid with TBP diluted by *n*-hexane. For measuring the distribution ratio of lactic acid in *n*-hexane, the aqueous solution of lactic acid with a fixed concentration and *n*-hexane of equal volume of 50 cm<sup>3</sup> were mixed in a flask and shaken at a fixed temperature for one hour to attain the extraction equilibrium. After two phases were separated, lactic acid concentration in the aqueous phase was determined by titration with known concentration of NaOH solution using phenolphthalein as indicator. The concentration of lactic acid in organic phase was calculated by total mass balance of lactic acid. The experimental parameters

were temperature (283, 293, 303, and 313 K) and lactic acid initial concentration (0.1, 0.2, 0.3, 0.5, and 0.5 mol/dm<sup>3</sup>).

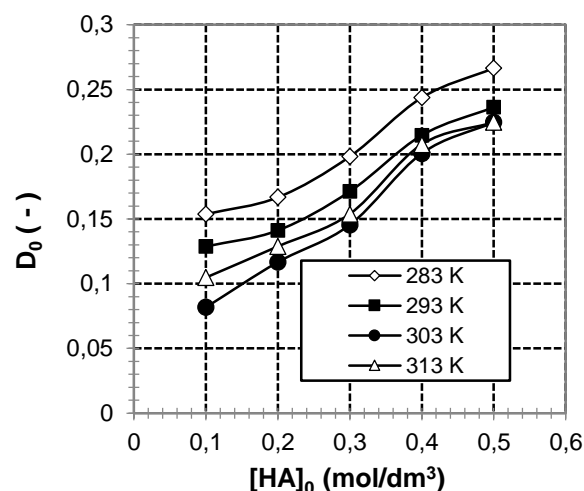
The experiments for the extraction of lactic acid with TBP in *n*-hexane were carried out by shaken of equal volume (50 cm<sup>3</sup>) of aqueous solution of lactic acid with solution of TBP in *n*-hexane. The experimental procedure was similar to that explained above, with the experimental parameters of temperature (283, 303, and 313 K), lactic acid initial concentration (0.1, 0.2, 0.3, 0.5, and 0.5 mol/dm<sup>3</sup>), and TBP concentrations (0.1, 0.2, 0.5, and 1.0 mol/dm<sup>3</sup>).

## RESULTS AND DISCUSSION

Table 1 shows the total lactic acid concentration in aqueous phase ( $[HA]_t$ ) and in organic phase ( $[\overline{HA}]_t$ ) at equilibrium as a function of initial concentrations of lactic acid in aqueous phase ( $[HA]_0$ ) and temperatures for the extraction of lactic acid with pure *n*-hexane. From the experimental data shown in Table 1, then the value of  $K_a$  was calculated by Eq. (3),  $D_o$  was calculated by Eq. (6), and the values of  $K_2$  and  $K_d$  were obtained based on Eq. (11).

Figure 1 shows the distribution ratio of the extraction of lactic acid with pure *n*-hexane as a function of initial lactic acid concentration in the aqueous solution at four different temperatures. It appears in Figure 1 that at a fixed temperature, the distribution ratio increases as a function of the initial concentration of lactic acid in the aqueous solution. It can be seen from Figure 1 that the distribution ratio decreases as the temperature increases for the experiments at the same initial

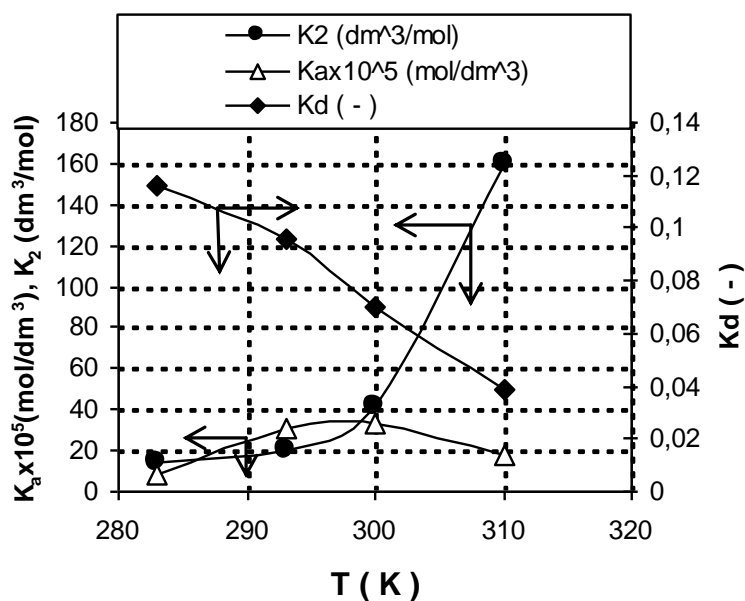
concentration of lactic acid in the aqueous solution.



**Fig. 1:** Effects of initial lactic acid concentration and temperature on the distribution ratio of the extraction of lactic

The values of  $K_a$ ,  $K_2$ , and  $K_d$  as a function of temperature are shown in Figure 2. Figure 2 depicts that  $K_a$  increases as a function of the temperature.  $K_a$  attains the maximum value of  $33.3 \times 10^{-5}$  mol/dm<sup>3</sup> at 300 K, then decreases to the value of  $17.8 \times 10^{-5}$  mol/dm<sup>3</sup> at 310 K. The value of  $K_2$  increases as a function of temperature, while the value of  $K_d$  is decreased by increasing the temperature.

The total lactic acid concentration in aqueous phase ( $[HA]_t$ ) and in organic phase ( $[\overline{HA}]_t$ ) at equilibrium as a function of initial concentrations of lactic acid in aqueous phase ( $[HA]_0$ ) and TBP concentration for the extraction of lactic acid with TBP in *n*-hexane at 293 K is shown in Table 2. From the data shown in Table 2 then the values of distribution ratio and overall equilibrium constant were calculated.



**Fig. 2:** Values of  $K_a$ ,  $K_2$ , and  $K_d$  as a function of temperature

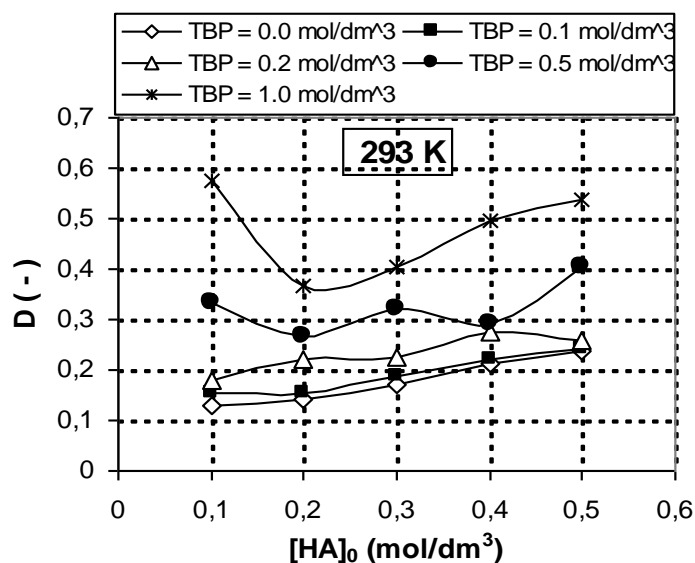
**Table 2.** Total lactic acid concentrations in aqueous phase and in organic phase at equilibrium as a function of initial concentrations of lactic acid in aqueous phase and TBP concentration for the extraction of lactic acid with TBP in *n*-hexane at 293 K

$[HA]_o$ (mol/dm <sup>3</sup> )	0.1 mol/dm <sup>3</sup>		0.2 mol/dm <sup>3</sup>		0.5 mol/dm <sup>3</sup>		1.0 mol/dm <sup>3</sup>	
	$[HA]_t$ (mol/dm <sup>3</sup> )	$[\overline{HA}]_t$ (mol/dm <sup>3</sup> )	$[HA]_t$ (mol/dm <sup>3</sup> )	$[\overline{HA}]_t$ (mol/dm <sup>3</sup> )	$[HA]_t$ (mol/dm <sup>3</sup> )	$[\overline{HA}]_t$ (mol/dm <sup>3</sup> )	$[HA]_t$ (mol/dm <sup>3</sup> )	$[\overline{HA}]_t$ (mol/dm <sup>3</sup> )
0.1	0.0867	0.0133	0.0847	0.0153	0.0751	0.0249	0.0636	0.0364
0.2	0.1733	0.0267	0.1637	0.0363	0.1579	0.0421	0.1464	0.0536
0.3	0.2523	0.0477	0.2446	0.0554	0.2273	0.0727	0.2138	0.0862
0.4	0.3274	0.0726	0.3139	0.0861	0.3101	0.0899	0.2677	0.1323
0.5	0.4025	0.0975	0.3968	0.1032	0.3563	0.1437	0.3255	0.1745

Figure 3 illustrates the distribution ratio of the extraction of lactic acid with TBP in *n*-hexane as a function of initial lactic acid concentration in the aqueous solution at different TBP concentration in the organic phase at 293 K. It is found that at a fixed TBP concentration, the distribution ratio tends to increase with increasing the initial lactic acid concentration in the aqueous solution. It is also shown in Figure 3 that at the same initial acid concentration, the distribution ratio increased with increasing

TBP concentration.

The experimental data of the concentrations of lactic acid in aqueous phase and in organic phase at equilibrium as a function of initial lactic acid concentration in aqueous phase and temperature for the extraction of lactic acid with TBP in *n*-hexane carried out with the concentration of TBP in *n*-hexane of 0.2 mol/dm<sup>3</sup> are depicted in Table 3. From the data depicted in Table 3 then the values of distribution ratio and overall equilibrium



**Fig. 3:** Effects of initial lactic acid and TBP concentrations on the distribution ratio of the extraction of lactic acid with TBP in *n*-hexane at 293 K.

**Table 3.** Total lactic acid concentrations in aqueous phase and in organic phase at equilibrium as a function of initial concentrations of lactic acid in aqueous phase and temperature for the extraction of lactic acid with TBP in *n*-hexane carried out at the TBP concentration of 0.2 mol/dm<sup>3</sup>.

[HA] <sub>o</sub> (mol/dm <sup>3</sup> )	283 K		293 K		303 K		313 K	
	[HA] <sub>t</sub> (mol/dm <sup>3</sup> )	[HĀ] <sub>t</sub> (mol/dm <sup>3</sup> )	[HA] <sub>t</sub> (mol/dm <sup>3</sup> )	[HĀ] <sub>t</sub> (mol/dm <sup>3</sup> )	[HA] <sub>t</sub> (mol/dm <sup>3</sup> )	[HĀ] <sub>t</sub> (mol/dm <sup>3</sup> )	[HA] <sub>t</sub> (mol/dm <sup>3</sup> )	[HĀ] <sub>t</sub> (mol/dm <sup>3</sup> )
0.1	0.0770	0.0230	0.0847	0.0153	0.0905	0.0095	0.0924	0.0076
0.2	0.1560	0.0440	0.1637	0.0363	0.1695	0.0305	0.1733	0.0267
0.3	0.2369	0.0631	0.2446	0.0554	0.2523	0.0477	0.2562	0.0438
0.4	0.3024	0.0976	0.3139	0.0861	0.3178	0.0822	0.3274	0.0726
0.5	0.3736	0.1264	0.3968	0.1032	0.4045	0.0955	0.4083	0.0917

constant were calculated.

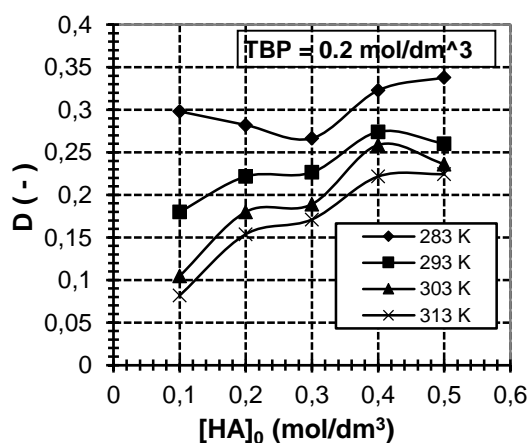
The effect of temperature on the distribution ratio of extraction of lactic acid with TBP in *n*-hexane is shown in Figure 4. The complexation reaction of lactic acid and TBP in the organic phase involve proton transfer or hydrogen bond formation and are therefore expected to be exothermic. Also, formation of a complex makes the system more ordered and

therefore decreases the entropy. Thus, as the temperature increases, the amount of lactic acid extracted decreases (Tamada and King, 1990; Juang and Huang, 1997).

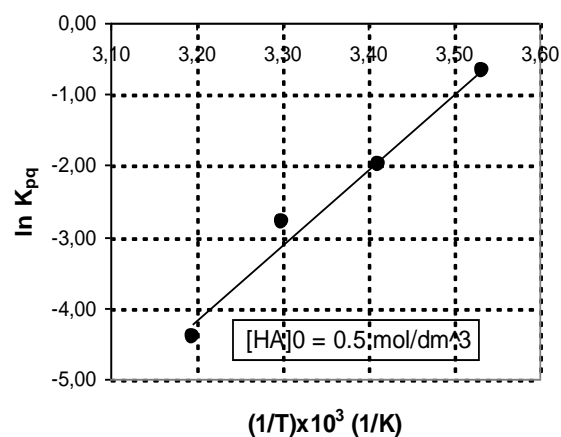
Figure 5 reveals the temperature dependence of the equilibrium constant  $K_{pq}$ . It can be seen at Figure 5 that the extraction of lactic acid with TBP in *n*-hexane is exothermic. The enthalpy of reaction ( $\Delta H$ ) is obtained from the slope of

**Table 4.** Comparison of the values of  $\Delta H$  and  $\Delta S$  obtained in this research with the values reported by previous researchers for the similar systems.

$\Delta H$ (kJ/mol)	$\Delta S$ (J/(mol·K))	Reference
-88.1	-316.6	This research
-69.2	-237.5 $\pm$ 2.3	Extraction of citric acid from aqueous solutions with tri- <i>n</i> -octylamine (Juang and Huang, 1994)
-137.0	-201.2 $\pm$ 8.5	Extraction of lactic acid with an amine extractant (Juang and Huang, 1997)
-69.0	-185.0	Succinic acid extraction with alamine 336/chloroform (Tamada and King, 1990)
-40.3	-113.6	Citric acid extraction with trialkylamine dissolved in 21.7% w/w 1-octanol/ <i>n</i> -heptane (Juang and Huang, 1994)
-41.40	-147.99	Reactive extraction of acetic acid with tri- <i>n</i> -butyl phosphate in <i>n</i> -hexane (Mulyono and Widayanty, 2011).

**Fig. 4:** Effect of temperature on the distribution ratio of extraction of lactic acid with TBP in *n*-hexane at the TBP concentration of 0.2 mol/dm<sup>3</sup>.

the graph and it was found to be -88,1 kJ/mol, and the intercept is proportional to the entropy ( $\Delta S$ ) and it was found to be -316.6 J/(mol.K). The values of  $\Delta H$  and  $\Delta S$  obtained in this research are compared to the values reported by previous researchers for the similar systems as shown in Table 4. The apparent thermodynamic functions strongly depend on the types of diluent,

**Fig. 5:** Temperature dependence of the equilibrium constant  $K_{pq}$ .

solvent, and acid (Juang and Huang, 1994).

## CONCLUSIONS

The reactive extraction of lactic acid from aqueous solution with TBP in *n*-hexane has been studied. Based on the results of this study, the following conclusions are made.



1. The higher the initial concentration of lactic acid in aqueous solution, the higher the distribution ratio for a fixed TBP concentration and extraction temperature.
2. For a fixed initial concentration of lactic acid in aqueous solution and extraction temperature, the distribution ratio of lactic acid is increased by increasing TBP concentration.
3. The overall equilibrium constants ( $K_{pq}$ ) for the experiments using TBP concentration ranging from 0.1 to 1.0 gmol /dm<sup>3</sup> at the extraction temperature of 293 K are found to be 0.0668 to 0.5144.
4.  $K_{pq}$  for the experiments at the temperature ranging from 283 to 313 K at the initial concentration of lactic acid of 0.2 gmol/dm<sup>3</sup> are found to be 0.0122 to 0.8856.
5. The values of  $\Delta H$  and  $\Delta S$  are found to be -88,1 kJ/mol and -316.6 J/(mol·K), respectively. The  $K_{pq}$  as a function of temperature ( $T$ ) in K can be expressed as  $\ln K_{pq} = 10,596/T - 38.08$  with sum of square of error of 0.14.

## NOMENCLATURE

- $D$  : distribution ratio of lactic acid by TBP in *n*-hexane (dimensionless)
- $D_0$  : distribution ratio of lactic acid by pure *n*-hexane (dimensionless)
- $\Delta H$  : apparent enthalpy change (kJ/mol)
- $K_2$  : dimerization constant of lactic acid in the organic phase (dm<sup>3</sup>/mol)

- $K_a$  : dissociation constant of lactic acid in the aqueous phase (mol/dm<sup>3</sup>)
- $K_d$  : distribution constant of the monomeric acid defined in Eq. (3) (dimensionless)
- $K_{pq}$  : overall equilibrium constant defined in Eq. (13) ((mol/dm<sup>3</sup>)<sup>1-p-q</sup>)
- $p$  : number of lactic acid molecules involved in the complex
- $q$  : number of TBP molecules involved in the complex
- $R$  : universal gas constant (8.314 J/(mol·K))
- $\Delta S$  : apparent entropy change (J/(mol·K))
- $T$  : temperature (K)
- [ ] : molar concentration of species in the brackets (mol/dm<sup>3</sup>)

## Subscripts

- $a$  : aqueous phase
- $org$  : organic phase
- $t$  : total value at equilibrium
- $0$  : initial

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