Articles

Equilibrium Studies of the Extraction and Re-extraction of Lactic Acid

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Equilibrium studies on the extraction of lactic acid in various organic phases and its re-extraction into aqueous solutions have been carried out. This includes a variety of experimental situations: using only solvents, single carriers with solvent, mixed carriers with solvent and with mixed solvents. The organic phase consists of a carrier (or a mixture of carrier) dissolved in a solvent (or a mixture of solvents). The extraction and re-extraction processes have been characterized by using distribution coefficient for the respective processes. The values of the distribution coefficient have been obtained by varying the operating conditions (i) for extraction: feed solution pH, types of carrier and its concentrations, solvent and its concentration in the organic phase; and (ii) for re-extraction: the type, pH, and concentration of recovery solution. It was found that 20 wt % of trioctylamine (an ionic carrier) dissolved in tributyl phosphate (an active solvent) was found to be the best extracting phase. For re-extraction, an aqueous solution of sodium carbonate gave the best recovery from the organic phase.

Introduction

Lactic acid has many applications in the food, chemical, pharmaceutical, and cosmetic industries.[1] It can be converted to ethanol, propylene glycol, acrylic polymers, and polyesters. Another application of lactic acid in the chemical industry is in the synthesis of biodegradable copolymers used for packaging.[2] Due to strict environmental laws being legislated, there is a growing demand for biodegradable polymers as a substitute for conventional plastic materials. Biodegradable copolymers are also used for the production of new materials in biomedical applications such as drug delivery systems.[3]

An important source of lactic acid is from the fermentation of biomass.[4]One of the problems associated with the production of lactic acid is that, as the fermentation process continues, the lactic acid bacteria become inhibited, preventing the production of lactic acid. A solution to this problem was adding calcium hydroxide and precipitation of calcium lactate.[1] However, this method is rather costly and environmental unfriendly as it consumes lime and sulfuric acid and also produces a large quantity of calcium sulfate as waste.[4]

Liquid−liquid extraction is an alternative method for the extraction of lactic acid. The advantage of using this method is that lactic acid can easily be removed (thus increasing the productivity) from the fermentation broth. The remainder solution can be recycled to the fermentation process. Table 1 shows a few examples on the extent of research on the technology based on liquid−reactive liquid extraction.[5−14] A recent review[15] provides useful information on the requirements of the fermentative production of glucose to lactic acid coupled with reactive extraction. All of these above-mentioned literature covered extensively the effects of various solvents and the effects of carrier/carrier mixtures and determined the thermodynamic equilibrium of lactic acid between organic and aqueous phases. Only a few of them include the re-extraction (recovery) of lactic acid from the organic phase and the effects of various components of a real fermentation broth. This part of the process is important because the overall efficiency of the extractive process depends on, and in many cases is determined by, the re-extraction ability from the organic phase. Therefore, there is still the need for more experimental data for practically useful systems covering both extraction and re-extraction processes, especially in presence of other components of the reaction mixture. The mechanisms of these processes and the elucidation of transport rates in the organic and aqueous phases is also required to understand and enhance the performance. This paper intends to present (i) the data on reactive extraction of lactic acid into organic phases using various solvents and carriers and in the presence of various components of the fermentation media and (ii) the data on re-extraction from these organic phases using various recovery solutions. The effect of the concentration of the strippant will also be covered.

Therefore, the aim of this work was set to investigate (i) the extraction of lactic acid into various organic solvents (tributyl phosphate, oleyl alcohol, decanol, hexane, dodecane, polypropylene glycol, and Shellsol TK) with single or mixed carrier (trioctylamine, trihexylamine, and tridodecylamine) added to it; (ii) its recovery into an aqueous solution (NaOH, Na_2CO_3, NaCl, and distilled water); and (iii) the effects of lactose and salts on theses processes.

Materials and Methods

Materials. Lactic acid (85 % pure) was obtained from Sigma-Aldrich (St. Louis, MO). The feed solutions were prepared by dissolving lactic acid in distilled water. In practical situations of lactic acid recovery from fermentation broths, the acid

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Table 1. Examples of Reactive Extraction of Lactic Acid from Aqueous Solution at Temperature of 25 °C Using Various Carrier−Solvent Combinations

solvent or a mixture of solvents	carrier or a mixture of carriers	*D*	ref
chloroform	diethylbutylamine	1.80	6
chloroform	tributylamine	1.40	6
chloroform	triamylamine	2.70	6
chloroform	trioctylamine	4.50	6
tributyl phosphate	tributylamine	0.19	7
tributyl phosphate	tripentylamine	0.62	7
tributyl phosphate	trioctylamine	7.42	7
kerosene	triamylamine	0.14	7
kerosene	tributylamine	0.24	7
kerosene	tripentylamine	0.16	7
xylene	trioctylamine	3.1	8
kerosene	trialkylphosphine oxide	2.4	9
hexane	triocylamine and tributyl phosphate	2.4	10
butyl acetate		2.8	
toluene		2.8	
chlorobenzene		3.0	
1-decanol		2.9	
methyl isobutyl ketone (MIBK)	alamine 336	4.24	11
1-octanol and kerosene (Parasol, Paz)	primene, JMT, and tris(2-ethylhexyl)amine	2.8	12
dodecane and decanol	quaternary ammonium chloride (aliquat 336)	0.65	13
dodecane and hexanol	tri-*n*-octylamine and triisooctylamine	16.5	14
dodecane and octanol		7.5	
dodecane and decanol		4.5	

concentrations are not expected to be high. Hence, a low concentration range of lactic acid was used. The organic solvents used were dodecane (99 %, ACROS Organics, USA), hexane (99 %, ACROS Organics, USA), oleyl alcohol (OA, 85 % pure, Lancaster Synthesis, England), decanol (Lancaster Synthesis, England), Shellsol TK (STK, Pacific Speciality Chemicals, New Zealand), polypropylene glycol (ACROS Organics, USA), and tributyl phosphate (TBP, 97 % pure, Sigma-Aldrich, USA). The carriers or extractants used were trioctyl amine (TOA, 98 % pure, ACROS Organics, USA), tridodecylamine (TDA), and trihexylamine (THA). The other chemicals used were lactose (Sigma-Aldrich, USA), sodium chloride (NaCl, Scharlau, Spain), sodium hydroxide (NaOH, Scharlau, Spain), and sodium carbonate (Na_2CO_3, BDH, England).

Apparatus and Procedure. The method of liquid−liquid extraction in the removal of lactic acid from fermentation broths is by using a carrier in an organic solvent. In this work, 5 mL of lactic acid (the aqueous phase) was mixed with 5 mL of the organic phase, which contained the solvent as well as the extractant in a 15 mL centrifuge tube. A magnetic stirrer was added, and the mixture was stirred for 2 h at 1000 rpm. After that, the mixture was placed in the centrifuge for 15 min at 4000 rpm and 25 °C. This was considered sufficient to separate the two phases after extraction.[7]

The effect of different pH values from 2 to 5 was investigated. This is within the range of natural pH of lactic acid. Its extraction from the fermentation broth (possibly without much pH adjustment) will reduce the inhibitory effect and enhance the process performance. Aqueous solution of 0.1 M HCl/0.1 M NaOH was used for adjusting the solution pH when required.

After the two phases were separated, the lactic acid concentration was determined by titration with NaOH (0.1 M) and by using phenolphthalein indicator.[15] The amount of lactic acid in the organic phase was determined by a mass balance taking in to account the volumes of the organic and aqueous phases, respectively. This is an approximate method and has been used in the literature.[11,12] For more accurate determination the HPLC method or enzymatic method should be used.[13,14]

Liquid−liquid extraction of lactic acid was first performed with solvent only (physical extraction). This was done to find out which solvent has the highest degree of extraction. Then extraction of lactic acid was performed with different carriers mixed with the solvent. This was done in order to find out which carrier performed the best. After that, extraction of lactic acid was carried out with mixed carriers combined with the solvent. In addition to this, extractions were carried out with mixed carriers and mixed solvents, and extraction of lactic acid in the presence of salts and lactose were performed.

In back-extraction, the same procedure was repeated with recovery solvents such as distilled water (DW), sodium chloride (NaCl), sodium hydroxide (NaOH), and sodium carbonate (Na_2CO_3). The lactic acid that was back-extracted from the organic phase consisted of 10 wt % TOA in TBP and was used for extraction from an aqueous solution of 0.2 M lactic acid.

Equilibrium Modeling. The extraction of lactic acid (HLA) with an amine carrier dissolved in a solvent (B) gives a reaction complex (BHLA), which remains largely in the organic phase and may be represented by

$$(\mathrm{HLA})_{\mathrm{aq}} + (\mathrm{B})_{\mathrm{org}} \leftrightarrow (\mathrm{BHLA})_{\mathrm{org}} \tag{1}$$

The distribution coefficient, *D*, and the degree of extraction, *E*, are defined by the following:

$$D = \frac{C_{\mathrm{LA}}^{\mathrm{org}}}{C_{\mathrm{LA_f}}^{\mathrm{aq}}} \tag{2}$$

and

$$\mathrm{E}/\% = \frac{C_{\mathrm{LA_i}}^{\mathrm{aq}} - C_{\mathrm{LA_f}}^{\mathrm{aq}}}{C_{\mathrm{LA_i}}^{\mathrm{aq}}} \times 100 \tag{3}$$

Back-Extraction Modeling. The organic reaction complex $(\mathrm{BHLA})_{\mathrm{org}}$ when put in contact with a salt, with positive and negative ions, results in the following reaction:

$$(\mathrm{BHLA})_{\mathrm{org}} + \mathrm{X^+Y^-} \leftrightarrow \mathrm{B} + \mathrm{X^+LA^-} + \mathrm{H^+Y^-} \tag{4}$$

$$\mathrm{DS} = \frac{C_{\mathrm{LA_f}}^{\mathrm{aq}}}{C_{\mathrm{LA_f}}^{\mathrm{org}}} \tag{5}$$

$$\mathrm{BE}/\% = \frac{C_{\mathrm{LA_f}}{}^{\mathrm{aq}}}{C_{\mathrm{LA_i}}{}^{\mathrm{org}}} \times 100 \quad (6)$$

where DS and BE/% are the distribution coefficient and percentage recovery for the back-extraction process, respectively.

Results and Discussion

Extraction. The results of the extraction process are presented as the distribution coefficient and the percentage of extraction efficiency.

The solvents can be classified into active and inactive diluents. In this study, tributyl phosphate, oleyl alcohol, decanol, and polypropylene glycol were used as active diluents and as the aqueous phase with an initial pH of 2.4 and concentration of 0.2 M for lactic acid solution. As shown in Table 2, the extraction efficiency of active solvents is higher than the inactive solvents such as hexane, dodecane, and Shellsol TK. Active diluents have functional groups that enable greater solvation of the acid−amine complex. The active solvent polypropylene glycol was an exception to this trend due to cloudiness in the extraction process, which led to difficulty in analyzing results. The best solvent for the extraction of lactic acid was found to be tributyl phosphate. Inactive diluents gave a low distribution of the lactic acid into the solvent phase.

The carrier THA was added in TBP to find out the effect of single carrier and single solvent on extraction of lactic acid. From the results in Table 3, it can be concluded that using a carrier dissolved in a solvent increases the extraction efficiency as compared to using just a single solvent as shown in Table 2. The 20 wt % THA in TBP gave a distribution coefficient of 2.57, which is higher than 80 wt % trihexylamine that gave a distribution coefficient of 0.41. This can be explained by the fact that, since TBA is an active solvent, its concentration affects the uptake of lactic acid in the organic phase (i.e. that is reducing its concentration is expected to decrease the value of the distribution coefficient). It is noted that maximum extraction with lower wt % of carrier is preferable because the re-extraction from the organic phase will be easier than those extracted with higher wt % of carrier.

Comparing the results in Table 3, TDA dissolved in TBP is a better extractant than THA. The 20 wt % TDA in TBP gave a distribution coefficient of 4.13 (the value for THA is 2.57), which is higher than 80 wt % tridodecylamine that gave distribution coefficient of 1.05. The effect follows a similar trend (i.e., the reduction in concentration of TBA decreases the value of *D*, but to a lesser extent as compared to the organic phase with THA). The higher extraction with lower wt % of carrier is considered to be a better system than that of higher wt % of carrier as the carrier is more expensive than diluent.

From the results of Table 4, it is observed that 20 wt % of TOA in TBP appears to be the best organic phase for extraction of lactic acid. Compared to the phases mentioned above, 5 % TOA can achieve almost the same percentage extraction as 20 % THA/TDA. As the length of the tertiary amine increases, the value of the distribution coefficient decreases.

As the wt % of TOA increases from 5 to 20, the distribution coefficient increases and reaches a maximum value of about 8.1. As the wt % of TOA in TBP increases to more than 20, the distribution coefficient decreases (Figure 1). A possible reason for this could be the effect of smaller concentration of the active diluent (as the TOA concentration increases TBP concentration decreases) having lesser degree of solvation of the complex. At higher concentration of TOA the solvent−carrier system could have different solubility characteristics and also could initiate other interactions. However, for the ease in re-extraction from the organic phase, it is better to perform extractions with smaller concentrations of carrier (i.e., TOA).

The mixture of oleyl alcohol and Shellsol TK gives smaller values of the distribution coefficient. Not much increase in the values were obtained by mixing the two solvents tributyl phosphate and Shellsol TK (Table 5). However, the values of the distribution coefficient increased as the % of TBP increased in the organic phase.

The mixture of TBP and STK with carrier TOA was difficult to analyze due to cloudiness. As a consequence of this, 0.5 mL of octanol was added to the mixture. This made it easier to analyze the experimental data. From the results shown in Table 6, the equal mixture of TBP and STK with 20 wt % TOA appears to be a good combination for the extraction of lactic acid. The distribution coefficient is still lower than that of 20 wt % of TOA in TBP, but STK is much cheaper than TBP. Hence, using a mixture of TBP and STK would save some money rather than using just TBP, with slight difference in extraction efficiency (about 6 %).

Oleyl alocohol and alkanes are stated to be nontoxic to anaerobic, acid-producing bacteria. Solvation extractants such as alkylamines are partially toxic.[5] However, the organic phase comprised of TOA dissolved in a mixture of OA and STK gave an extraction efficiency of about 66 % (Table 7). This result is about 17 % lower than the mixture of TBP and STK (Table 6).

Table 2. Effect of Various Solvents on Physical Extraction of Lactic Acid[a]

solvent	concn of HLA final value/M	*D*	*E*/%
tributyl phosphate (TBP)	0.112	0.78	43.90
oleyl alcohol (OA)	0.156	0.28	21.95
decanol (D)	0.144	0.39	28.05
hexane (H)	0.159	0.26	20.73
dodecane (DD)	0.159	0.26	20.73
polypropylene glycol (PPG)	0.163	0.22	18.29
Shellsol TK (STK)	0.183	0.09	8.54

[a] Initial concentration in aqueous solution is 0.2 M.

Table 3. Effect of Single Carrier, Trihexylamine (THA)/ Tridodecylamine (TDA) Mixed with Tributyl Phosphate (TBP) on Extraction of Lactic Acid[a]

wt % of carrier	equilibrium HLA concn in aqueous phase/M	*D*	*E*/%
	Trihexylamine		
20	0.056	2.57	72
80	0.141	0.41	29
	Tridodecylamine		
20	0.039	4.13	80
80	0.141	1.05	51

[a] Initial concentration in aqueous solution is 0.2 M.

Table 4. Effect of Single Carrier, Trioctylamine (TOA) Mixed with the Solvent Tributyl Phosphate (TBP) on Extraction of Lactic Acid[a]

wt % of carrier	equilibrium HLA concn in aqueous phase/M	*D*	*E*/%
5	0.049	3.10	76
10	0.024	7.20	88
20	0.022	8.11	89
40	0.027	6.45	87
60	0.049	3.10	76
80	0.068	1.95	66

[a] Initial concentration in aqueous solution is 0.2 M.

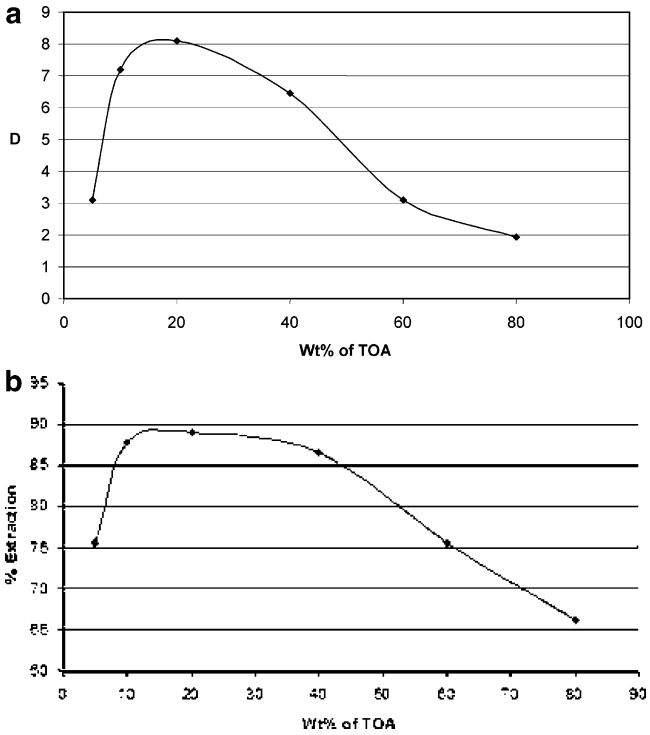

Figure 1. Effect of concentration of TOA on (a) distribution coefficient, *D*, and (b) on % extraction of lactic acid.

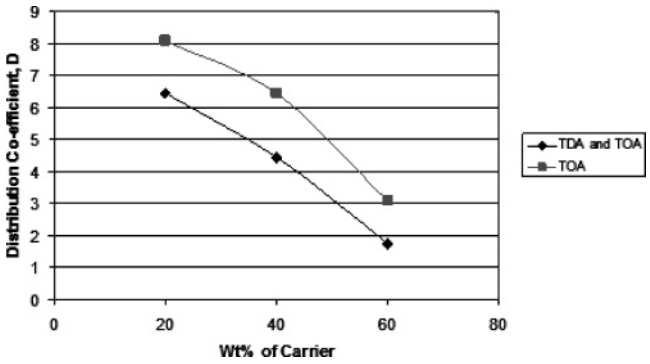

Figure 2. Plot of distribution coefficient vs wt % of TOA only and a mixture of TDA and TOA.

Table 5. Physical Extraction of Lactic Acid[a] with Mixed Solvents

solvents	equilibrium HLA concn in aqueous phase/M	*D*	*E*/%
OA (50 %) in STK (50 %)	0.1780	0.12	10.98
TBP (25 %) in STK (75 %)	0.1463	0.21	17.07
TBP (50 %) in STK (50 %)	0.1659	0.37	26.83
TBP (75 %) in STK (25 %)	0.1244	0.61	37.80

[a] Initial concentration in aqueous solution is 0.2 M.

Table 6. Extraction of Lactic Acid[a] with Single Carrier (TOA) and Equally Mixed Solvents, TBP and STK, with 0.5 mL of Octanol

wt % of carrier	equilibrium HLA concn in aqueous phase/M	*D*	*E*/%
20	0.166	4.86	82.93
40	0.149	2.90	74.39
60	0.127	1.73	63.41
80	0.151	3.10	75.61

[a] Initial concentration in aqueous solution is 0.2 M.

Table 7. Extraction of Lactic Acid[a] with Single Carrier (TOA) and Equally Mixed Solvents, OA and STK

wt % of carrier	equilibrium HLA concn in aqueous phase/M	*D*	*E*/%
20	0.124	1.65	62.20
40	0.132	1.93	65.85
60	0.102	1.05	51.22
80	0.078	0.64	39.02

[a] Initial concentration in aqueous solution is 0.2 M.

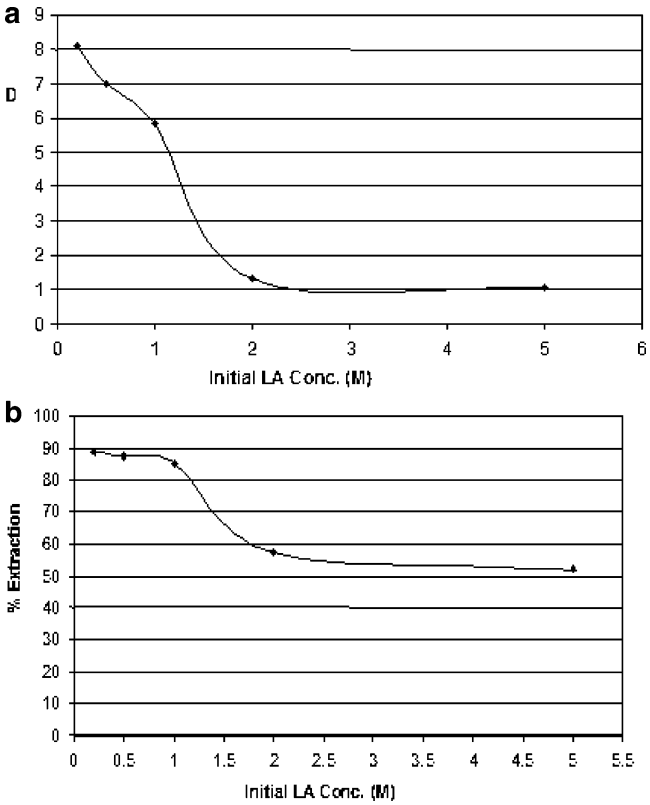

Figure 3. (a) Plot of distribution coefficient as a function of initial lactic acid concentration. (b) Plot of extraction and initial concentration of lactic acid.

The extraction results of single carrier TOA and mixed carriers TOA and TDA are shown in Figure 2. It shows that single TOA carrier is better than mixed carriers and that beyond 20 wt % of the carriers, the distribution coefficient decreased for both the carrier systems.

Figure 3a shows that as the concentration of lactic acid increases, the distribution coefficient decreases. One of the reasons for this could be that, because the carrier amount remaining fixed in the organic phase, there was not enough TOA to react with the increasing acid ions. The value of distribution coefficient was highest, about 8 for 0.2 M lactic acid, and the values decreased to 1 for higher lactic acid concentrations (2 to 5 M). A similar trend was observed in the extraction performance (i.e., the % extraction decreased from 90 % for low concentration of lactic acid (at 0.2 M) to about 56 % at higher concentrations (2 M and greater), as shown in Figure 3.

The effect of feed solution pH on distribution coefficient is presented in Figure 4. The values obtained in the pH range of 2 to 3 was high (ca. 8). Beyond this pH, the value of distribution coefficients decreased sharply to very low to 0.4 at pH 5. This could be due to the effect of available concentration of undissociated lactic acid, which decreases considerably with the increase in pH. At pH 2.3, this concentration is approximately 97 % and decreases to 27 % at pH 4.2.[17] The carrier TOA can only solvate the free acid. As its concentration decreases, a lower amount is transferred to the organic phase. Also at the stated

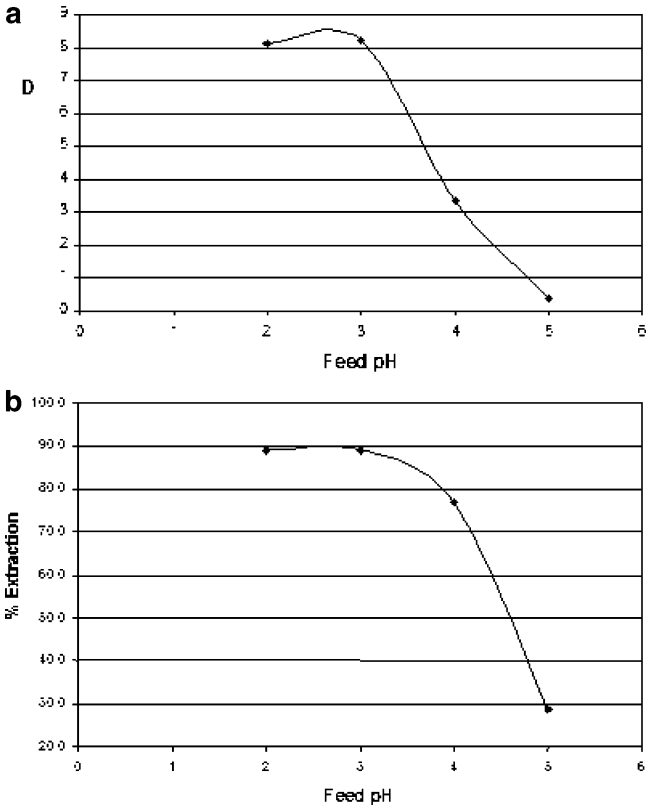

Figure 4. (a) Distribution coefficient vs feed pH of lactic acid. (b) Extraction (%) vs feed pH.

Table 8. Effect of Lactose on Extraction of Lactic Acid[a]

	equilibrium HLA concn in aqueous phase/M	*D*	*E*/%
lactose	0.0559	0.79	44.14
lactose and LA	0.0559	0.79	44.14

[a] Initial concentration in aqueous solution is 0.1 M.

Table 9. Effect of Salt on Extraction of Lactic Acid[a] with 20 wt % TOA in TBP

concn of NaCl in HLA solution/g·L^{-1}	equilibrium HLA concn in aqueous final phase/M	D	*E*/%
1	0.028	6.1	85.9
3	0.03	5.64	84.9
5	0.039	4.13	80.5

[a] Initial concentration in aqueous solution is 0.2 M.

Table 10. Re-extraction of Lactic Acid[a] from an Organic Phase of 10 wt % TOA in TBP with Various Recovery Solutions

solvent	pH	equilibrium BHLA concn/M	equilibrium HLA concn in recovery phase/M	DS	BE/%
DW	6.5	0.0176	0.0019	0.11	10
NaCl	10	0.0216	0.003	0.14	12
NaOH	11	0.0218	0.0033	0.15	13
Na_2CO_3	11	0.0836	0.0628	0.73	43

[a] Initial concentration in aqueous solution is 0.2 M.

conditions of carrier and lactic acid concentrations, the organic system can be assumed to attain saturation (i.e., the extent of complex formation is complete); so the decrease in initial concentration would decrease the value of the distribution coefficient. The percentage extraction also decreased from around 90 % (at pH 2 to 3) to around 30 % at pH 5. It would have been good to determine a system that will provide a good

Table 11. Effect of Concentration of Sodium Carbonate (Recovery Solvent) on the Back-Extraction of Lactic Acid[a] from an Organic Solution of 10 wt % TOA in TBP

concn of Na_2CO_3/M	equilibrium BHLA concn/M	equilibrium HLA concn in recovery phase/M	DS	BE/%
0.1	0.086	0.0628	0.73	43
0.5	0.108	0.127	1.17	54
1	0.120	0.18	1.50	60
2	0.182	1.884	10.35	91

[a] Initial concentration in aqueous solution is 0.2 M.

distribution coefficient and extraction at higher pH with more environmentally friendly organic system. Further investigation is being carried out along this direction.

The addition of lactose to the feed solution did not affect the extraction efficiency of the organic phase, so the *E*/% of lactic acid remained the same (Table 8) in presence of these components (as being the components of a fermentation system). The concentration of salt in initial lactic acid solution was chosen to closely represent the actual concentration found in the fermentation broths.[2] The effect of sodium chloride salt is shown in the Table 9. Comparing the results between Tables 5 and 9, it is observed that the extraction efficiency of 20 wt % TOA in TBP is 89 % (no salt in feed) and that in presence of the salt the effect is minimal, about 86 % of lactic acid can be extracted. It is also found that as the amount of salt in the feed solution increased, the extraction efficiency decreased further. A possible explanation for this could be that the H^+ from the lactic acid combines with the Cl^- and decreases the amount of lactic acid effectively available to react with the carrier in the organic phase.

Re-extraction of Lactic Acid. The re-extraction results are presented in Tables 10 and 11. It is shown that the best solution for recovery of lactic acid is Na_2CO_3, followed by NaOH, then NaCl, and last, distilled water (Table 10). Na_2CO_3 is the best solvent possibly because it has more cations (two Na^+ ions) to take part in the decomplexation reaction (represented in eq 4) responsible for the re-extraction from the organic phase.

As the concentration of Na_2CO_3 is increased, the amount of lactic acid recovered increases as well. At 2 M concentration of Na_2CO_3, 91 % of the lactic acid was recovered as shown in Table 11. These values are higher or at least comparable to those found in the literature (only a few have covered these part).

Conclusions

It is possible to extract lactic acid into an organic phase and recover it into an aqueous phase. The best extraction of lactic acid was achieved with 20 wt % of trioctylamine (TOA) in tributyl phosphate (TBP). It was found that TOA was the best carrier and that TBP was the best solvent for extraction of lactic acid at its natural pH. The value of the distribution coefficient and % extraction decreased as the pH was increased and as the initial concentration of lactic acid was increased. The presence of lactose did not affect the percentage extraction of lactic acid, but the amount of salt such as sodium chloride does affect the extraction. Extraction of lactic acid decreases as the amount of salt increases. An aqueous solution of sodium carbonate was found to give very good recovery, and this increased with the concentration of sodium carbonate in the recovery solution.

Glossary

D	distribution coefficient for extraction
DS	distribution coefficient for back-extraction
E	degree of extraction

BE	degree of back-extraction
HLA	lactic acid
B	organic solvent
C	concentration, mol·L^{-1}
BHLA	complex of lactic acid in organic phase
X^+Y^-	ionic salt with positive and negative ions

Superscripts/Subscripts

LA	indication of lactic acid
aq	indication of aqueous phase
org	indication of organic phase
LA_i	indication of lactic acid initial
LA_f	indication of lactic acid final

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