

Process Intensification for Separation of Carboxylic Acids from Fermentation Broths using Reactive Extraction

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Abstract

The potential role of a new energy efficient fermentation technology has been receiving growing attention in past 4 decades. Carboxylic acids such as propionic acid, lactic acid, citric acid, tartaric acid, itaconic acid, butyric acid and nicotinic acid etc. have been used as the most efficient fermentation chemicals. This paper presents a state-of-art review on the development of the separation techniques for carboxylic acids from fermentation broths. This paper mainly focuses on reactive extraction that is found to be a promising alternative to the conventional recovery processes in terms of intensifying the process.

Keywords: Carboxylic acids; Fermentation; Separation Processes; Process intensification; Reactive Extraction.

Introduction

Growing demand for biodegradable polymer substitutes for both conventional plastic materials and new materials of specific uses such as controlled drug delivery or artificial prostheses draws attention to the need for improvement of conventional processes for carboxylic acid production. The organic acids are most widely used in the field of food and beverages as an acidulant and also in pharmaceutical and chemical industries. Fermentation technology for the production of organic acids in particular has been known for more than a century and acids have been produced in aqueous solutions. Extractive fermentation has been applied in the production of a variety of carboxylic acids, including propionic acid, as a means of overcoming end-product inhibition [1-4]. The current economic impact of fermentation chemicals, however, is still limited, in large part because of difficulties of product recovery. Thus, for fermentation products to penetrate the organic chemicals industry, substantial improvements in the existing recovery technology are needed.

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Over the last decade several continuous fermentation processes have been described and reviewed. Although there are several advantages claimed for continuous fermentation processes, it is still vitally important for economically attractive recovery technology to be developed if fermentation derived organic acids are to penetrate the organic chemicals industry to a large extent. Reactive liquid-liquid extraction for the recovery of organic acids by a suitable extractant has been found to be a promising alternative to the conventional processes [5].

For Example, the world market for lactic acid is growing every year. The level of production is around 350 millions kg per year and the worldwide growth is believed by some observers to be 12-15% per year. In December 1994, market prices in the U.S. for both fermentation and synthetic food-grade 50 and 88% lactic acid were \$0.71 and \$1.15 lb⁻¹ (\$1.56-2.53 kg⁻¹), respectively. Technical-grade 88% lactic acid was quoted at \$1.12 lb⁻¹ (\$2.47 kg⁻¹) [6]. In April 2003, market prices in the U.S. for 88% foodgrade and technical-grade lactic acid were \$0.77 and \$0.7 lb⁻¹, respectively. These prices were 50% lower than the prices of year 1994, illustrating the economics of scale based on the increasing use of carboxylic acids [7]. Similarly The world-wide production of niacin also called nicotinic acid strongly increased in the last several years to about 22,000 tonnes/year (over 65% is nicotinic acid), the major producers of nicotinic acid being Lonza (Switzerland), Degussa, BASF (Germany), Nepera, Reilly Industries (ex-Vitachem) (US), and Yuki Gosei (Japan) (8, 9).

Carboxylic Acids: Fermentation Products

The carboxyl functional group that characterizes the carboxylic acids is unusual in that it is composed of two functional groups; the carboxyl group is made up of a hydroxyl group bonded to a carbonyl group. It is often written in condensed form as -CO₂H or -COOH.

The carboxylic acids under consideration are obtained by fermentation process. Some of these acids (propionic acid, lactic acid, itaconic acid, tartaric acid, and citric acid) are obtained by the aerobic fermentation of glucose via the glycolytic pathway and glyoxylate bypass. Corn meal hydrolyzed with amylases was used as the carbon source for producing acetic acid, propionic acid, and butyric acid via anaerobic fermentation. The aerobic fermentation process leading to the formation of organic acids is one of the best established microbial conversion pathways [10].

Pyruvic acid is oxidized in a cyclic manner to yield a number of di- and tricarboxylic aliphatic acids of four to six carbon atoms. Some modifications involve the reduction of pyruvic acid to lactic acid, or cleavage to yield formic and acetic acids, or propanoic acid as the major end product. It should be emphasized that this fermentation process will produce several end products in high yields. The most widely used carboxylic acids obtained by fermentation process are shown in Table-1 [11].

Table-1. Various carboxylic acids obtained by fermentation process [11]

S. No	Name of acid	Source	Usage
1.	Lactic acid	whey, potatoes, cornstarch, or molasses	used in biopolymer, as a food additive, sweets, and soft drinks etc.
2.	Citric acid	fruits and vegetables	used in biochemistry, natural preservative, as an antioxidant etc
3.	Tartaric acid	plants, particularly grapes and tamarinds	used as an antioxidant, used in food industries
4.	Propanoic acid	milk(Gk.protus prion)	used as a preservative and to make pesticides, pharmaceuticals etc.
5.	Butanoic acid	butter (L. butyrum)	manufacture of plastics, agricultural chemicals and pharmaceuticals etc.
6.	Itaconic acid	sugar, carbohydrates	used in polymers, paints, coatings, medicines and cosmetics etc.
7.	Gluconic acid	plants, fruits and other foodstuffs, honey	used in detergent, cleaner and industries.
8.	Aspartic acid	plant proteins, sprouting seeds, dairy	used in production of Bio-chemicals, immunoglobulin etc

Conventional Techniques for Carboxylic Acid Separation

Fermentation technology for the production of organic acids in particular has been known for more than a century and acids have been produced in the form of aqueous solutions. They have various severe inhibiting effects on the rate of conversion and recovery methods from fermentation broths. Several separation methods such as liquid extraction [12-14], ultra filtration [15], reverse osmosis [16], electro-dialysis [15, 17-19], direct distillation [20], liquid surfactant membrane extraction [21], anion exchange [22], precipitation and adsorption [23, 24] etc. have been employed to remove carboxylic acids.

For the purification of acid by precipitation, the fermentation liquor is filtered and evaporated. In the calcium precipitation process, the separation and final purification stages account for up to 50% of the production costs and produces a large quantity of solid waste. Electro-dialysis is a recovery process where ion-exchange membranes are used for removing ions from an aqueous solution under the driving force of electrical field. Nomura and Hongo [17] proposed the possibility of electro dialysis for *in situ* recovery of lactic acid to reduce product inhibition in batch fermentation. The amount of lactic acid obtained was 5.5 times greater than that produced in non-pH- controlled fermentation. Carboxylic acids may be recovered by adsorption on solid adsorbent. Kawabata et al. [25] separated carboxylic acid by using a polymer adsorbent of pyridine skeletal structure and a cross-linked structure. The polymer adsorbent showed good selectivity and high adsorption capacity for carboxylic acids even in the presence of inorganic salts. The selected elutants were aliphatic alcohol, aliphatic ketones and carboxylic esters. Chen and Ju [26] studied the coupled fermentation and adsorption to prevent the product concentration from reaching inhibitory levels. Reverse Osmosis has

also been studied for recovery of carboxylic acids from fermentation broths [16]. They concluded that the reverse osmosis could effectively concentrate the acid from 10 to 120 gdm⁻³ at a 6.9 MPa trans-membrane pressure (lesser energy than that used in multiple effect evaporators). Wasewar [27] reviewed the recent developments of recovery of lactic acid from fermentation broth.

Intensification for Carboxylic Acid Separation: Reactive Extraction

Among various available alternate processes for simultaneous removal of the product, extraction is often the most suitable one [28-31]. Reactive extraction with a specified extractant giving a higher distribution coefficient has been proposed as a promising technique for the recovery of carboxylic and hydroxyl-carboxylic acids. Reactive Extraction is developed to intensify separation by solvent extraction and represents a connection between chemical (solute and extractant reaction) and physical phenomena (diffusion and solubilization of the system components). So a reactive extraction method has been proposed to be an effective primary separation step for the recovery of carboxylic acid from a dilute fermentation process [32-34].

This method is advantageous for alcohol and organic fermentations. Some of the advantages are increased reactor productivity, ease in reactor pH control without requiring base addition, and use of a high-concentration substrate as the process feed to reduce process wastes and production costs. This method may also allow the process to produce and recover the fermentation products in one continuous step and reduce the down stream processing load and the recovery costs [35]. Reactive extraction strongly depends on various parameters such as the distribution coefficient, degree of extraction, loading ratio, complexation equilibrium constant, types of complexes (1:1, 2:1, etc.), rate constant of carboxylic acid-extractant reaction, properties of the solvent (extractant and diluent), type of solvent, temperature, pH and acid concentration [36, 37].

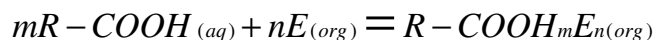
The extraction of carboxylic acids is categorized into three groups: (i) acid extraction by solvation with carbon-bonded oxygen-bearing extractants (also inert aliphatic and aromatic hydrocarbons and some of their substituted homologs); (ii) acid extraction by solvation with phosphorus-bonded oxygen-bearing extractants and (iii) acid extraction by proton transfer or by ion pair formation, the extractant being high-molecular weight aliphatic amines [38]. The distribution coefficients of carboxylic acids between the aqueous phases and organic phases with only first categorized solvents are very low as shown in Table-2 [39].

Table-2. Distribution coefficients (K_D) of Carboxylic acids between Water and Organic solvents at 25 °C

Carboxylic Acid	Solvent	K_D
Propionic acid	n-Hexane	0.005
	Cyclohexane	0.006
	Benzene	0.043
	Toluene	0.034
	Xylene	0.03
	Carbon tetrachloride	0.015
	Chloroform	0.11
	Nitrobenzene	0.16
	Diethyl ether	1.75
	Diisopropyl ether	0.80
	Methylisobutyl ketone	2.15
	Cyclohexanone	3.30
	n-Butanol	3.20
	n-Pentanol	2.95
Lactic acid	Diethyl ether	0.10
	Diisopropyl ether	0.04
	Methylisobutyl ketone	0.14
	n-Octanol	0.32
Succinic acid	Diethyl ether	1.50
	Methylisobutyl ketone	0.19
	n-Butanol	1.20
Fumaric acid	Diethyl ether	1.50
	Methylisobutyl ketone	1.40
Maleic acid	Diethyl ether	0.15
	Methylisobutyl ketone	0.21
Itaconic acid	Diethyl ether	0.35
	Methylisobutyl ketone	0.55
Tartaric acid	Diethyl ether	0.03
	Methylisobutyl ketone	0.02
Citric acid	Diethyl ether	0.01
	Methylisobutyl ketone	0.09
Malic acid	Diethyl ether	0.02
	Methylisobutyl ketone	0.04

Organophosphoric derivatives such as tri-n-octyl phosphine oxide (TOPO), tri-n-butyl phosphate (TBP) and long-chain, aliphatic amines such as lauryl-trialkylmethylamine (Amberlite LA-2), tri-n-octylamine, tri-iso-octylamine, tri-n-(octyl-decyl)-amine (Alamine 336) and quaternary alkylammonium salt (Aliquat 336) are effective extractants for separation of carboxylic acids from dilute aqueous solution [35, 38]. Generally, the amine extractants are dissolved in a diluent, an organic solvent that dilutes the extractant. It controls the viscosity and density of the solvent phase. However, the chemical structure of a diluent may have various effects connected with the formation of acid-amine

complexes in the organic phase. The equilibrium behavior has been studied effectively by postulating the formation of various stoichiometric complexes of acid and amine [40-42]. The reactive extraction mechanism can be expressed by following interface equilibrium:



Hong and Hong [43] carried out the Reactive extraction of lactic acid with mixed tertiary amine extractants. The mixture of tripropylamine (TPA) and trioctylamine (TOA) dissolved in 1-octanol/n-heptane was used in the reactive extraction of (L+) lactic acid in aqueous solution. Equilibria and Kinetics for lactic acid extraction by alamine 336 in methyl – iso – butyl ketone (MIBK) as a diluent have been studied by Wasewar *et al.* [44]. Malmay *et al.* [45] proposed experimental and mathematical studies for recovery of carboxylic acids from aqueous solutions by liquid-liquid Extraction with a triisooctylamine diluent system. They studied the liquid-liquid equilibria between the triisooctylamine + 1-octanol + n-heptane system as solvent and an aqueous solution of an individual carboxylic acid such as citric acid, lactic acid and malic acid. Tamada *et al.* [40, 41] studied the extraction of acetic acid, lactic acid, succinic acid, malonic acid, fumaric acid, and maleic acid by Alamine 336, an aliphatic, tertiary amine extractant, dissolved in various diluents. The effect of the modifier, (1-decanol) on the extraction of tartaric acid using the reactive extractants has been carried out by Mariya *et al.* [46]. The effect of the modifier was examined as a function of the initial pH value of the aqueous solution for the various extractants. Equilibrium of tartaric acid extraction from aqueous solution with HOSTAREX A 324 (commercial tri-iso-octylamine) in iso-decanol/low aromatic kerosene mixtures was studied as a function of acid, amine and iso-decanol concentrations at 25°C by Popaska *et al.* [47]. Matsumoto *et al.* (2001) studied the extraction of organic acids using both amine based extractant – (tri-n-octylamine) and phosphorus based extractant – (tri-n-butyl phosphate) [48]. Reactive extraction of succinic acid from aqueous solutions with various tertiary amines dissolved in 1- octanol and in n-heptane has been studied as a function of the chain length of the tertiary amine. In the tertiary amine extractants in 1-octanol, the extractabilities of tertiary amines were proportional to their chain length [49]. Cascaval *et al.* studied the reactive extraction of mono-, di- and polycarboxylic acids using Amberlite LA-2 dissolved in diluents [50, 51]. The design of an amine extraction process requires equilibrium data for the acid-amine (solvent) system used. Equilibria for propionic acid extraction by trioctyl amine in various solvents hexanol, butyl acetate, and petroleum ether have been determined by Wasewar and Pangarkar [52].

Phosphorus based solvating extractants has also opened new avenues in process development and reduced energy and reagent consumption and several studies using these extractants have been made. extraction equilibria of a number of organic acids, like acetic acid, glycolic acid, lactic acid, pyruvic acid, butyric acid, succinic acid, fumaric acid, maleic acid, itaconic acid, tartaric acid, citric acid and isocitric acid with tri-*n*-octylphosphine oxide in hexane has been investigated by Hano *et al.* [53]. Al-Mudhaf *et al.* [54] and Cai *et al.* [55] using phosphate based extractants showed that tri-*n*-octylphosphine oxide (TOPO) had a higher distribution coefficient than tri-*n*-butyl

phosphate (TBP). Wang *et al.* [56] has used Cyanex® 923 in kerosene to extract monocarboxylic acids and found that the distribution ratios increased with an increase in the trialkylphosphine oxide concentration but decreased with the carboxylic acid concentration in the aqueous phase. The study has been made to examine the use of the organophosphine oxides, (Cyanex@921 and Cyanex@923) for the extraction of formic, acetic and propionic acids from aqueous solutions by Wisniewski and Piezchalska [57]. Kumar *et al.* [58, 59] has intensified the recovery of nicotinic acid and propionic acid using reactive extraction. The conventional extractants, such as simple hydrocarbon, ketone, kerosene oil and alcohol with or/and without tri-n-butyl phosphate (TBP) and Aliquat 336 are used to recover the acids from aqueous solution using reactive extraction. The comparative study of the reactive extraction of nicotinic acid with Amberlite LA-2 (lauryl-trialkyl-methylamine) and di-(2-ethylhexyl)-phosphoric acid (D2EHPA) has been reported by Cascaval *et al* [60].

Conclusion

The growing demand of carboxylic acids draws attention to the improvement of a conventional recovery process for carboxylic acid production. It is important to have an efficient and sustainable process for the separation of these acids from the fermentation broth. Although commercial processes for acid recovery are based on the classical method of separation, this separation and final purification stages account for up to 50% of the production costs. The productivity of these fermentation processes can be significantly increased by *in-situ* recovery of carboxylic acids from fermentation broths by process intensification using reactive extraction. Organophosphoric derivatives and long-chain, aliphatic amines have also opened up new avenues in recovery process development and reduced energy and reagent consumption using reactive extraction.

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