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Intensification of picolinic acid extraction with tri-*n*-butylphosphate and tri-*n*-octylamine in three different diluents

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ABSTRACT

In this paper, the equilibrium study on reactive extraction of picolinic acid from aqueous solution is studied by individual and mixed extractant of tri-*n*-butyl phosphate (TBP) and tri-*n*-octylamine (TOA) in three different diluents at a constant temperature, 298 K. A better synergism is observed (80% extraction efficiency) with a TBP:TOA ratio of 1:1 for all acid concentration with IAA. Mathematical modeling is also carried out to find insight of mechanism exist between acid and extractant molecules. The results of present study will be useful to intensify the recovery of picolinic acid from fermentation broth (bio-separation) and other aqueous solutions.
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1. Introduction

Picolinic acid, a pyridine carboxylic acid is detected in a variety of biological mediums [cell culture supernatants, blood serum cerebrospinal fluid (CSF), human milk, pancreatic juice and intestinal homogenates]. It is an endogenous metabolite of L-tryptophan and possesses a wide range of neuroprotective, immunological, and anti-proliferative affects within the body (Grant et al., 2009). The acid shows antiviral, cytotoxic and apoptotic effects against human immunodeficiency virus-1 (HIV-1) and human herpes simplex virus-2 (HSV-2) infected cells (Fernandez-Pol et al., 2001). Therefore, it is an importance and necessary approved food supplement in the body. Picolinic acid acts as a chelating agent of elements such as chromium, zinc, manganese, copper, iron, and molybdenum in the human body. The acid is believed to form a complex with zinc that may facilitate the passage of zinc through the gastrointestinal wall and into the circulatory system (Evans and Johnson, 1981). Capitalizing on its chelation properties HPc–metal complexes are now widely used as a means of bioactive metals into biological systems (Suzuki et al., 1957; Broadhurst and Domenico, 2006).

Currently, pyridine carboxylic acids are produced by chemical oxidation of alkyl pyridines under severe operating conditions (temperature and pressure) with high cost and modest productivity. However, the enzyme catalyzed reactions based on fermentation technology have started to replace the conventional synthesis route of these chemicals in the industry (Kumar and Babu, 2009a; Kaplan et al., 2006). The enzymes work in a mild environmental condition and are suitable for the production of labile organic molecules. They are also very specific to a particular product synthesis. Nitrilase enzymes have been identified as valuable biocatalysts for the synthesis of pyridine carboxylic acids from the low cost and readily available nitriles (Malandra et al., 2009). Generally, downstream processing (recovery) of bio-products accounts for a huge amount (near about 60%) of the total cost of the production

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Nomenclature

—	overbar to denote species in the organic phase, [–]
$\overline{C}_{11}$	1:1 acid–extractant complex concentration, [kmol m^{-3}]
$\overline{C}_{1m}$	1:*m* acid–extractant complex concentration, [–]
$C_{HPc}^{Diluent}$	concentration of the acid in aqueous phase with diluent only, [kmol m^{-3}]
$\overline{C}_{HPc}^{Diluent}$	concentration of the acid in organic phase with diluent only, [kmol m^{-3}]
C_{HPc}	aqueous phase acid concentration at equilibrium, [kmol m^{-3}]
$\overline{C}_{HPc}$	organic phase acid concentration at equilibrium, [kmol m^{-3}]
D	dimerization constant, [kmol^{-1} m^3]
E	degree of extraction, [–]
H^+	hydrogen ion, [–]
HPc	picolinic acid, [–]
[HPc]	undissociated acid concentration, [kmol m^{-3}]
$(\overline{HPc})_2$	acid dimer, [–]
H_2^+Pc	charged cations, [–]
K_1, K_2, K_3, K_E	equilibrium constants, [–]
K_{a1}, K_{a2}	dissociation constants, [–]
K_D^{Chem}	distribution coefficient in chemical extraction, [–]
$K_D^{Diluent}$	distribution coefficient in physical extraction, [–]
$K_{D,TBP–TOA}$	distribution coefficient with mixed extractant, [–]
K_D^{Total}	total distribution coefficient, [–]
m	number of extractant molecules reacting with one acid molecule, [–]
N	number of data points, [–]
P	partition coefficient, [–]
Pc^-	dissociated ions, [–]
s	loading of acid in organic phase by diluent alone, [–]
T	extractant, [–]
$[\overline{T}]$	extractant concentration in the organic phase, [kmol m^{-3}]
$[\overline{TBP}]_o$, $[\overline{TOA}]_o$, $[\overline{T}]_o$	initial extractant concentration in the organic phase, [kmol m^{-3}]
Z	loading ratio, [–]
$Z_{TBP–TOA}$	loading ratio with mixed extractant, [–]

Greek

ν	volume fraction of diluent in the organic phase, [–]

Subscripts

aq	aqueous, [–]
org	organic, [–]
o	initial, [–]

Abbreviations

IAA	iso-amyl alcohol, [–]
MIBK	methyl-iso-butyl ketone, [–]
NaOH	sodium hydroxide, [–]
PE	petroleum ether, [–]
rmsd	root mean square deviation, [–]
TBP	tri-*n*-butyl phosphate, [–]
TOA	tri-*n*-octylamine, [–]
vol.%	volume percentage, [–]

process (Chen et al., 2007). When the bio-product is required in the concentrated form, the existing fermentation technology cannot compete with the chemical synthesis route for the large-scale production. Therefore, it is necessary to focus on the development of an efficient recovery method which will improve the bio-synthesis path of pyridine carboxylic acids and their derivatives by fermentation (Tuyun and Uslu, 2011).

The reactive extraction is proposed to be an efficient and effective separation technique to recover carboxylic acids from a dilute aqueous solution like fermentation broth. Several equilibrium studies are available in the literature on the recovery of pyridine carboxylic acids by reactive extraction (Kumar and Babu, 2009a,b; Tuyun and Uslu, 2011; Hong et al., 2002; Kertes and King, 1986; Kumar and Babu, 2008; Jarvinen et al., 2000; Uslu, 2007; Wasewar et al., 2002; Kumar et al., 2008; Waghmare et al., 2011; Datta et al., 2012; Datta and Kumar, 2013; Senol, 2001). Investigators have studied the effect of different parameters such as composition of the phases, types of diluents and extractants, types of acids, pH of the aqueous phase, temperature of the recovery system, etc. on the extraction efficiency of the reactive extraction. Though, substantial work on the equilibrium study of several acid–amine systems is available in the literature, very limited information pertaining to equilibrium and kinetics of pyridine carboxylic acids is available. In the present study, the equilibrium reactive extraction of picolinic with individual and mixed extractants [tri-*n*-butyl phosphate (TBP) and tri-*n*-octylamine (TOA)] dissolved in three different solvents [petroleum ether (PE), methyl-iso-butyl ketone (MIBK) and iso-amyl alcohol (IAA)] at an equilibrium temperature of 298 K has been carried out. The equilibrium results (experimental and theoretical) of the reactive extraction of acid will be useful to design the extraction process for the intensification of recovery of picolinic acid from fermentation broth (bio-separation) and other aqueous solutions.

2. Experimental

2.1. *Reagents*

Picolinic acid is a white crystalline powder of purity 99.5% and is purchased from Himedia, India. Tri-*n*-butyl phosphate (purity = 98%, molecular weight = 266.32 kg kmol^{-1}, density = 977 kg m^{-3}, Spectrochem Pvt. Ltd., India) and tri-*n*-octylamine (purity = 98%, molecular weight = 353.68 kg kmol^{-1}, density = 811 kg m^{-3}, Spectrochem Pvt. Ltd., India) are used as extractants. Petroleum ether (purity = 99.5%, molecular weight = 78.11 kg kmol^{-1}, density = 878.6 kg m^{-3}, SISCO Res. Lab. Pvt. Ltd. India), methyl iso butyl ketone (purity = 99.5%, molecular weight = 78.11 kg kmol^{-1}, density = 878.6 kg m^{-3}, SISCO Res. Lab. Pvt. Ltd. India), and iso-amyl alcohol (purity = 98%, molecular weight = 158.28 kg kmol^{-1}, density = 830 kg m^{-3}, Spectrochem Pvt. Ltd., India) of technical grade are used as diluents to prepare the organic solution. Sodium hydroxide used for titration is supplied by Merck, India and phenolphthalein solution (pH range of 8.2–10.0, CDH, India) is used as an indicator for titration.

2.2. *Procedure*

0.12 kmol m^{-3} stock solutions of picolinic acid was prepared and then diluted using distilled water to get the required concentrations (0.02–0.12 kmol m^{-3}) of acid in the aqueous phase. The organic solutions consisted of TBP (0.365 kmol m^{-3}) and

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