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# Reactive Extraction of Itaconic Acid using Tri –n-Butyl Phosphate in 1-Butanol, 1-Octanol and 1-Dodecanol

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**ABSTRACT**: Reactive extraction of itaconic acid is one of the most promising methods of extraction when compared with other processes like distillation, membrane, dialysis, electro dialysis etc. The reactive extraction has been a highly successful method for recovery of carboxylic acids from dilute solutions. In present paper, reactive extraction of itaconic acid using tri-n-butyl phosphate (TBP) (extractant), an organophosphorus compound in 1-butanol, 1-octanol, and 1-do-decanol (diluents) has been studied. Results were presented in terms of distribution coefficients and degree of extraction. The data obtained are useful in understanding the equilibrium characteristics for efficient design of reactive extraction process for recovery of itaconic acids from fermentation broths.

KEYWORDS: Reactive extraction, TBP, Itaconic acid, 1-Butanol, 1-Octanol, 1-Do decanol

### I. INTRODUCTION

Unsaturated dibasic carboxylic acids like Itaconic acid (methylene succinic acid) can be efficiently used as an emulsifier in paints, as starter for polymerization and for making durable carpet fibers. Itaconic acid can also be used for making special lenses and synthetic cut stones as it imparts special lustre and transparency to the polymers. Now a day's Fermentation technology (green and clean) is viewed as promising alternative to produce itaconic acid from renewable materials. The recovery of products from fermentation broth is a major challenge in the commercialization so there is need to develop a separation technique which can make fermentation an efficient and effective alternative[1-2]. A review of literature was carried out to find out the various methods employed for recovery of organic acids (produced by fermentations) from aqueous solution [3-11]. Literature review revealed that unsaturated di basic carboxylic acids like itaconic acid can be extracted by using organophosphorus compounds (TBP, TOPO) and amines (Aliquat, TOA, Amberlite).

The objective of my work is to develop a new combination of extractant (TBP) and diluent (alcohols) system to extract itaconic acid from aqueous solution as no work has been found to be done on extraction of itaconic acid by TBP in 1-butanol, 1- octanol and 1-dodecanol.

### II. RELATED WORK

Few studies related to extraction of Itaconic Acid are summarized here

Hasan Uslu et .al [2] carried out experimental and theoretical investigations on reactive extraction of itaconic (2-Methylidenebutanedioic) acid using Trioctylamine (N,N-Dioctyloctan-1-amine). They reported maximum extraction efficiency of 94.80% and obtained  $K_D$  equal to 18.20 at 1.946 mol.kg-1 of TOA in octan-1-ol.



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G Kaur et al.[12]-studied exhaustively the extraction of itaconic acid with eight different amines with seventeen types of diluents (alcohols, esters, alkane)but only nine reactive extraction systems consisting of amine extractants (trioctylamine, dioctylamine and *N*methyldioctylamineas) dissolved in 1-octanol, pentylacetate and methyloctanoate were found to be most suitable. The results of this study will form the basis for future investigations for large scale reactive extraction systems and development of appropriate method for itaconic acid extraction.

Selim et al.[13]studied the reactive extraction of itaconic acid by using trioctylamine (TOA), tridodecylamine (TDA) and mixtures of Amines in di methyl phthalate ((DMP), methyl isobutyl ketone (MIBK), 2-octanone, 1-octanol, cyclohexyleacetate (CHA), and 1-decanol. The TOA–TDA mixture in DMP gave highest 98.39% extraction of itaconic acid.

Wasewar et al.[14] studied the Reactive extraction of itaconic acid using quaternary amine Aliquat 336 in ethyl acetate, toluene, hexane, and kerosene as diluents and described the values of equilibrium complexation constants, loading ratios, and distribution coefficients.

Dipaloy Datta et  $al.^2$  studied the reactive extraction of 2-methylidenebutanedioic acid (five initial acid concentrations in the range of 0.05 to 0.25mol. dm-1) from aqueous solution by N,N-dioctyloctan-1-amine (TOA, 0.115 and 0.229mol.dm-3) as extractants dissolved in six different diluents [heptane, kerosene, methylbenzene, decane-1-ol, 4-methylpentan-2-one (MIBK). They reported a highest value of the KD 32.478 at 0.05 mol. dm<sup>-3</sup>.

Wasewar et al.[15] studied the Reactive extraction of itaconic acid using tri-*n*-butylphosphate (TBP) (an organophosphorus compound) and Aliquat 336(a quaternary amine) in sunflower oil(non toxic diluent) and reported equilibrium complexation constant, *K*E, values of 1.789 and 2.385 m3 kmol-1, respectively.

George Kyuchoukov et al. [16] studied the extraction of itaconic acid from aqueous solution with Tributylphosphate dissolved in dodecane at different volume phase ratios and they predicted that the acid extraction was possible only for a limited range of acid concentrations. The experimental results also revealed that the concentration of the interaction product in the aqueous phase cannot be neglected.

Michiaki Matsumoto et al. [17] studied the Synergistic extraction equilibria of organic acid (itaconic acid) with trioctylamine (TOA) and or tri-n-butylphosphate (TBP). They showed that extraction equilibrium constants were roughly correlated with the hydrophobicity of the acid, except for L+-malic acid. They calculated results of the distribution ratio of organic acids based on the formation of three complexes.

Eyal Bressler et al. [1] studied the selective separation of citric and itaconic acids by amine extractants. They also discussed the possible roles of amine salt and the diluent on selectivity. They found that DEHPA & MTOA was selective towards itaconic acid where as TOA in 1-octanol towards citric acid.

Hano et al.[18] studied the extraction equilibria of organic acid(itaconic acid) with Tri-n-octyl phosphineoxide (TOPO). The salvations' numbers of the acids were the same as the numbers of the carboxyl groups on each acids molecule. The extraction equilibrium was constant was controlled by the hydrophobicity of the acid when hexane was used as a diluents.

#### III. EXPERIMENTAL

**Material and Methods:** TBP (99%) (Otto, India), a phosphorous bonded oxygen donor, is a light colourless liquid with the molecular weight of 266.32g/mol and density of 0.975 g/cm<sup>3</sup>. Itaconic acid was purchased as crystals. Diluents: Butanol, Octanol and Do decanol. For titration NaOH was used. Phenolphthalein solution (pH range 8.2 - 10.0) was used as an indicator. Low initial aqueous concentration of acid (0.05 to 0.2 kmol/m<sup>3</sup>) was used in view to find the effectiveness of the reactive extraction for recovery of acid present in dilute form and as the concentration is not expected to be greater than 0.2 kmol/m<sup>3</sup> in fermentation broths



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**General procedure:** The extraction experiments were performed using an orbital shaking incubator by maintaining a constant temperature of 303K. Equal volumes (30 ml) of aqueous and organic phases were taken in a 250 ml conical flask and were shaken for 12 hour. This could be considered as the appropriate time for attaining equilibrium. The solution was then made to settle for at least 2 hour at a fixed temperature (303K) and atmospheric pressure in a separating funnel. The aqueous phase was taken for analysis. The concentration of acid in organic phase was determined by material balance as initial concentration of acid in aqueous phase minus final acid concentration in aqueous phase. The experiments were repeated 3 times to check the consistency and average is taken for calculation to minimize the error

**Detection Method:** The itaconic acid concentration in equilibrium aqueous phases was determined by titration using NaOH solution. The standardization of NaOH with the Oxalic acid was carried out and freshly prepared NaOH was used before every titration for calculating concentration of itaconic acid. The acid concentration in the organic phase was obtained by the mass balance.

#### Theory:

The recovery of itaconic acid using TBP is by the formation of a complex via the interfacial reaction [4] and can be represented as:

$$HA_{aq} + pB_{org} \leftrightarrow (HA. B_p)_{org}$$
 (1)

This complex is rapidly extracted into the organic phase. The extraction constant and the number of reacting molecules of extractant are computed by applying law of mass action that is ratio between concentrations of reactant molecules and concentration of the product species, according to following equation as:  $K_{\rm E} = [({\rm HA}).({\rm B})_p]_{\rm org}/[{\rm HA}]_{\rm aq} [{\rm B}]^p_{\rm org}$  (2)

 $K_{\rm E}$  is expected to depend on the properties of the acid and the solvation efficiency of the diluent used. The dissociation of the acid in the aqueous phase is given as follows:

$$\mathrm{HA} \leftrightarrow \mathrm{H}^{+} + \mathrm{A}^{-} \qquad K_{\mathrm{HA}} = \frac{[\mathrm{H}^{+}][\mathrm{A}^{-}]}{[\mathrm{HA}]} \qquad (3)$$

Distribution ratio,  $K_D$  is defined as the ratio of total (analytical) concentration of acid in all its forms (by partition, dimmers and as complexes) in organic phase and total (analytical) concentration of all its existing forms (dissociated and undissociated) in aqueous raffinate.  $K_D$  includes the effects like ionic strength, nature of ion concentration of H<sup>+</sup> etc. of solution constituents.

$$K_{\rm D} = \frac{[\rm{HA}]_{\rm org}}{[\rm{HA}]_{\rm aq}} \tag{4}$$

Degree of extraction is defined as the ratio of itaconic acid concentration in organic phase to the sum of acid concentration in organic and aqueous phase and is defined in term of  $K_{\rm D}$  as,

$$E\% = \frac{K_{\rm D} \times 100}{1 + K_{\rm D}}$$
(5)



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#### IV. EXPERIMENTAL RESULTS

Table 1: Equilibrium results for the Physical extraction of itaconic acid using in 1- butanol, 1- octanol and 1- dodecanol at 303k

Pure	Average K <sub>D</sub>	Average %E
1- butanol	2.698	72.53
1- octanol	0.611	37.775
1- dodecanol	0.41	29.29

The physical extraction of itaconic acid using 1- butanol, 1-octanol, and 1-dodecanol was performed and data obtained is shown in Table 1. The degree of extraction (E %) of itaconic acid in respective extractants is found to be 72.53%, 37.7755% and 29.29%.

Table 2: Eq	uilibrium results	s for the extracti	on of itaconic	acid using TBF	dissolved in	1-butanol at 303K
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TBP kmol m <sup>-3</sup>	[HA] <sub>0</sub> kmol m <sup>-3</sup>	[HA] <sub>aq</sub> kmol m <sup>-3</sup>	[HA] <sub>org</sub> kmol m <sup>-3</sup>	K <sub>D</sub>	Avg.K <sub>D</sub>	Е%	Avg.E%
0.37	$0.05 \\ 0.1 \\ 0.15 \\ 0.2$	0.0136 0.0292 0.0448 0.0721	0.0363 0.0707 0.1051 0.1278	2.666 2.416 2.342 1.770	2.298	72.722 70.725 70.077 63.898	69.355
0.74	0.05 0.1 0.15 0.2	0.0140 0.0344 0.0520 0.0766	0.0360 0.0666 0.0980 0.1241	2.571 1.941 1.884 1.633	2.007	71.996 53.61 65.325 62.020	66.334
1.1	0.05 0.1 0.15 0.2	0.0160 0.0400 0.0580 0.0780	0.0340 0.0060 0.0920 0.1220	2.125 1.156 1.586 1.564	1.607	68.00 53.610 61.330 60.998	60.984
1.48	0.05 0.1 0.15 0.2	0.0095 0.0228 0.0400 0.0571	0.0404 0.0771 0.1100 0.1428	4.252 3.374 2.750 2.500	3.219	80.959 77.137 73.33 71.428	75.713
1.85	0.05 0.1 0.15 0.2	0.0055 0.0167 0.0297 0.0446	0.0444 0.0832 1.1202 0.1553	7.960 4.973 4.038 3.479	5.112	88.839 83.257 80.150 77.673	82.479

Chemical equilibrium data for extraction of itaconic acid using 0-50 % TBP in 1-butanol are presented in Table 2.  $K_D$  was obtained 2.698, 2.298, 2.007, 1.607, 3.219 and 5.112 with (0 – 50) % TBP in 1-butanol. Higher degree of extractions was obtained at lower concentrations of acid with various TBP percentages



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Table 3: Equilibrium results for the extraction of itaconic acid using TBP dissolved in 1-octanol at 303K

ТВР	[HA] <sub>0</sub> kmol m <sup>-3</sup>	[HA] <sub>aq</sub> kmol m <sup>-3</sup>	[HA] <sub>org</sub> kmol m <sup>-3</sup>	K <sub>D</sub>	Avg.K <sub>D</sub>	Е%	Avg.E%
0.37	0.05 0.1 0.15 0.2	0.0260 0.0576 0.0874 0.1209	0.0239 0.0423 0.0625 0.0790	0.9193 0.7339 0.7154 0.6539	0.755	47.897 42.326 41.704 39.537	42.866
0.74	0.05 0.1 0.15 0.2	0.0279 0.0595 0.0893 0.1227	0.0220 0.0404 0.0607 0.0772	0.7919 0.6797 0.6797 0.6292	0.695	44.193 40.465 39.722 38.620	40.750
1.1	0.05 0.1 0.15 0.2	0.0186 0.0446 0.0744 0.1041	0.0314 0.0553 0.0755 0.0958	1.6881 1.2396 1.0156 0.9197	1.215	62.799 55.349 50.386 47.908	54.110
1.48	0.05 0.1 0.15 0.2	0.0111 0.0297 0.0502 0.0707	0.0388 0.0702 0.0997 0.1293	3.4880 2.3592 1.9862 1.8289	2.415	77.718 70.231 66.512 64.650	69.777
1.85	0.05 0.1 0.15 0.2	0.0055 0.0167 0.0297 0.0446	0.0442 0.0832 0.1202 0.1553	7.9605 4.9735 4.0387 3.3408	5.078	88.839 83.259 80.153 76.962	82.303

The chemical equilibrium data for extraction of itaconic acid using 0- 50% TBP in 1-octanol are presented in Table 3.  $K_D$  was obtained 0.611, 0.755, 0.695, 1.215, 2.415 and 5.078 with (0 - 50) % TBP in 1-octanol. Higher degree of extractions was obtained at lower concentrations of acid



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Table 4: Equilibrium results for the extraction of itaconic acid using TBP dissolved in 1-dodecanol at 303K

TBP kmol m <sup>-3</sup>	[HA] <sub>o</sub> kmol m <sup>-3</sup>	[HA] <sub>aq</sub> kmol m <sup>-3</sup>	[HA] <sub>org</sub> kmol m <sup>-3</sup>	K <sub>D</sub>	Avg.K <sub>D</sub>	Е%	Avg.E%
0.37	0.05 0.1 0.15 0.2	0.028 0.061 0.100 0.137	0.021 0.038 0.049 0.062	0.74 0.62 0.49 0.45	0.57	42.52 38.27 32.88 31.03	36.17
0.74	0.05 0.1 0.15 0.2	0.024 0.055 0.090 0.121	0.025 0.044 0.059 0.078	1.03 0.80 0.66 0.65	0.78	50.73 44.44 39.75 39.39	43.57
1.11	0.05 0.1 0.15 0.2	0.018 0.045 0.077 0.102	0.031 0.054 0.072 0.097	1.70 1.21 0.92 0.95	1.19	62.96 54.75 47.91 48.71	53.58
1.48	0.05 0.1 0.15 0.2	0.010 0.036 0.061 0.088	0.039 0.063 0.088 0.111	3.87 1.70 1.43 1.26	2.06	79.46 62.96 58.84 55.75	64.25
1.85	0.05 0.1 0.15 0.2	0.006 0.028 0.051 0.071	0.043 0.071 0.098 0.128	7.13 2.48 1.92 1.78	3.31	87.69 71.26 65.75 64.02	72.18



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The chemical equilibrium data for extraction of itaconic acid using 0-50% TBP in 1-dodecanol are presented in Table 4.  $K_D$  was obtained as 0.41, 0.57, 0.78, 1.19, 2.06 and 3.31 with (0 - 50) % TBP in 1-dodecanol. Higher degree of extraction was obtained at lower concentrations of acid



Fig.1. %E (Degree of extraction of itaconic acid) Vs % TBP dissolved in 1- butanol, 1- octanol and 1- dodecanol at 303K

From the Fig.1, *E*% with 1- butanol was found to decrease from 72.537 % (0% TBP) to 60.984% (30% TBP) and then increased to 82.49% (50% TBP). In case of 1- butanol the degree of extraction of 72.53 % indicates that 1- butanol is good extractor but with the addition of TBP the % E first decreases and then increases indicates a possible interaction between functional OH group of alcohol and TBP. In the beginning when TBP concentration is less this interaction leads to decrease in the degree of extraction and once TBP concentration increases there is again increase in the degree of extraction.

Higher degree of extractions was obtained at lower concentrations of acid with and without TBP when 1-octanol was used. An unusual behaviour occurred at 20% TBP resulting in slight decrease in the degree of extraction. Here the alkyl group seems to interfere between the interaction between functional OH group of alcohol and TBP due to which the degree of extraction increases as the percentage of TBP.

Higher degree of extraction was obtained at lower concentrations of acid with and without TBP in 1 -dodecanol. E% was found to increase from 29.29 % (0% TBP) to 72.18 % (50% TBP). In case of 1- dodecanol the interference of large alkyl group was quite eminent from the fact the degree of extraction and  $K_D$  values increases with the increase in TBP concentration

#### V. CONCLUSION

Physical and reactive extraction of itaconic acid was studied using TBP in various diluents: 1- butanol, 1- octanol and do decanol. Results obtained showed an interaction between TBP and alcohols thus useful in designing of reactive extraction process for recovery of itaconic acid



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