Liquid-Liquid Equilibria for Water + 2,3-Butanediol + 3-Methyl-1-Butanol at Different Temperatures of 303.15, 313.15, and 323.15 K

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***Abstract*—Liquid-liquid equilibria of water + 2,3-butanediol+3-methyl-1-butanol at different temperatures of 303.15, 313.15, and 323.15 K were measured. Complete phase diagrams were obtained by evaluating the solubility and tie-line results for ternary system. The consistency of the tie-line was ascertained using Othmer-Tobias plot. The effect of temperature on distribution coefficient and selectivity of 2,3-butanediol were studied. It was observed that increase in temperature has a favorable effect on the distribution coefficient and selectivity.**

***Index Terms*—2,3-butanediol, Liquid-liquid extraction, ternary system, Othmer-Tobias plot.**

I. Introduction

2,3-butanediol is promising chemical which have a wide range of applications and can be biologically produced. The separation of this diol from fermentation broth makes more than 50% of the total costs in their microbial production. It is argued that separation technologies such as aqueous two-phase extraction with short chain alcohols deserve more attention. Sharma et. al (1) investigated extraction using 3-methyl-1-butanol, butane-1-ol, 2-methyl-2-pentanone, trybutyl phosphate and butyl acetate. It has been found that solvents having good extracting power for 2,3-butanediol have a low selectivity. Therefore an investigation was carried out to study the effect of different temperatures on the selectivity , extracting power of the solvent and tie lines . This work measurement of liquid-liquid equilibrium (LLE) data of water + 2,3-butanediol + 3-methyl-1-butanol under atmospheric pressure and at different temperature ranges of 303.15, 313.15, and 323.15 K and the consistency of the tie-line was ascertained using Othmer-Tobias plots.

II. Materials and Methods:

3-methyl-1-butanol was of analytical grade and was used without further purification. 2,3-butanediol was obtained from Merk with a purity of 98% and used as such. Double distilled water was sued through out the experimental study.

BD was determined by gas chromatography (Perkin Elmer Sigma 3b) using chromosorb 101 coated with 3% ffap.

III. Experimental Methods:

Experimental section**;**

The mutual solubilities for water + 2,3-butanediol + 3-methyl-1-butanol at different temperature ranges of 303.15, 313.15, and 323.15 K were determined by using cloud point titrator (2). Extractions were performed by placing 5ml of a solution containing 3% w/v 2,3-butanediol in water into a sample cell of the cloud point titrator (Fig.1) along with 5 ml of solvent. Water was circulated with the aid of peristaltic pump through the water jacket to maintain different temperature. The mixture was stirred for 30minutes. Thereafter the two layers were separated by centrifugation, and analyzed for 2,3-butanediol concentrations by GLC-PE Model Sigma 3B equipped with an FID, using a stainless steel column (2.5m x 1/8 in. O.D.) packed with chromosorb 101 (80/100 mesh) coated with 3% FFAP (1). The extractions were carried out in triplicate and standard deviation calculated was found to be less than +/- 0.0065 mass fraction of 2,3-butanediol.

Tie lines values were determined by making mixtures of the three components, water , 2,3-butanediol and 3-methyl-1-butanol whose compositions fell within the two-phase region of the solubility curve. The mixtures of a 3ml of 2,3-butnediol in water and 3 ml of 3-methyl-1-butanol were taken in sample bottles and stirred for 30 minutes to reach equilibrium at different temperatures. The mixtures were separated by centrifugation and the two layers were analyzed for 2,3-butanediol by GLC. The complete composition of the conjugated layers were obtained from the mutual solubility curve by locating the points on the curve. The composition determination was accurate to +/- 0.0026 mass fraction.

Result and Discussion:

The experimental esults, in mass fraction for the mutual solubility and tie lines of ternary system water + 2,3-butanediol + 3-methyl-1-butanol at different temperature ranges of 303.15, 313.15, and 323.15 K are given in TABLE I, TABLEII and TABLE III. Mutual solubility curves along with tie-lines and mixture composition for ternary system have been plotted on a triangular diagram and are shown in fig 2, fig. 3 and fig.4.

The effect of temperature on the distribution of 2,3-butanediol between water and 3-methyl-1-butanol is depicted in fig.6. It was observed that with increase in temperature more of the butanediol gets extracted into the solvent phase. The selectivity curves for the system water + 2,3-butanediol+3-methyl-1-butanol at different temperatures is shown in fig 7, which indicate increased selectivity with increase in temperature. This increase in selectivity is due to increase in the solubility of 2,3-butanediol in solvent phase. At 5% concentration of 2,3-butanediol, 20 K rise in temperature increases the distribution coefficient from 0.63 to 1.132 and selectivity from 6.38 to 8.2. The temperature also has an effect on rate of extraction, rise in temperature invariably increases

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the rate of extraction and also enhances the rate of coalescence or phase separation. The areas of two-phase region decrease with increasing temperature as observed by Jui-Tang Chen (5 & 6). From these Liquid-liquid extraction data, we can find that very small amounts of the organic compounds were found in the aqueous phase, while water dissolved appreciably in the organic-rich phase This is quite sensitive and a 10 K rise in temperature can easily halve the thickness of a dispersion band (3). Similar observations were made by Ghanadzadeh G.H (4) using 2-ethyl -1-hexanol as solvent.

IV. Conclusion

Thus it is observed that increase in temperature has a favorable effect on the distribution coefficient and selectivity. A 20 K rise in temperature increases the distribution coefficient from 0.63 to 1.132 and selectivity from 6.38 to 8.2. Increase in temperature also increase the rate of extraction and phase separation.

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Dr. Mrs. Sadhana Vishwakarma is born in Nagpur on 14thAugust 1967. She completed her undergraduation from Shri Shivaji Education Society, Nagpur, India, in 1988 and stood third in order of merit and received Gold medal in Zoology. She completed her post graduation in organic chemistry in 1990 from Science college, Nagpur, India and stood first in order of merit and received gold medal in Organic chemistry. In 1991 she has completed her degree in education from Nagpur university, India and diploma in pharmacy in 2005 from Karnataka Board, Karnataka, India. She has completed her MSCIT certificate course in 2005. She acquire Ph.D. from Nagpur University in 1996 and topic of the thesis was " Recovery of value added chemicals from fermentation broth".

She has 2 years research experience as junior project fellow under DBT sponsored project " Production of 2,3-butanediol from water hyacinth and 3 years experience as senior project fellow under the DBT sponsored project " Production of Hydrogen and chemicals from waste in National Environmental Engineering Research Institute (NEERI), Nagpur, India. She worked as Head of the Department of Medical Laboratory Technology in Gramin Polytechnique , Vishnupuri, Nanded, India successfully for 9 years. Then she worked as Assistant Professor of chemistry in All Saints' college, Bhopal, India for 1 year and now she is working as Professor of Engineering Chemistry in Technocrates Institute of Technology Bhopal, India till date. She worked as a coordinator of Jalswarajaya Project titled " 100 % Analysis of government drinking water sources of Latur and Washim districts".

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TABLE I: Mutual Solubility for Water(1) + 2,3-Butanediol(2) +3-Methyl-1-Butanol at303.15 K (W = Mass Fraction)

W_1	W_2	W_3
0.987	-	0.013
0.838	0.140	0.022
0.753	0.212	0.035
0.691	0.252	0.056
0.600	0.292	0.108
0.548	0.294	0.158
0.456	0.292	0.252
0.339	0.282	0.379
0.244	0.221	0.535
0.188	0.162	0.650
0.133	0.066	0.801
0.094	-	0.906
Tie Lines for Solubility for water(1) + 2,3-butanediol(2) +3-methyl-1-butanol at 303.15K		
Water rich layer		Solvent rich layer
W_{11}	W_{21} W_{31}	W_{13} W_{23} W_{33}
0.950	0.032 0.018	0.090 0.020 0.890
0.906	0.076 0.018	0.100 0.029 0.871
0.852	0.128 0.020	0.110 0.040 0.850
0.830	0.149 0.021	0.120 0.060 0.820
0.775	0.195 0.030	0.131 0.080 0.790

TABLE II: Mutual Solubility for Water(1) +2,3-Butanediol(2) +3-Methyl-1-butanol at 313.15 K (W = Mass Fraction)

W_1	W_2	W_3
0.977	-	0.023
0.831	0.138	0.031
0.710	0.237	0.053
0.548	0.274	0.178
0.554	0.279	0.168
0.498	0.295	0.223
0.458	0.276	0.266
0.413	0.267	0.320
0.334	0.256	0.409
0.258	0.218	0.524
0.185	0.140	0.674
0.139	0.066	0.795
0.110	-	0.890
Tie Lines for Solubility for water(1) + 2,3-butanediol(2) +3-methyl-1-butanol at 313.15K		
Water rich layer		Solvent rich layer
W_{11}	W_{21} W_{31}	W_{13} W_{23} W_{33}
0.945	0.029 0.025	0.113 0.023 0.864
0.910	0.062 0.028	0.127 0.048 0.825
0.860	0.104 0.029	0.149 0.082 0.769
0.850	0.119 0.031	0.165 0.112 0.723

TABLE III: Mutual Solubility for Water(1) + 2,3-Butanediol(2) +3-Methyl-1-Butanol at 323.15K (W = Mass Fraction)

W_1	W_2	W_3
0.962	-	0.031
0.827	0.138	0.035
0.699	0.233	0.067
0.562	0.260	0.178
0.522	0.265	0.207
0.487	0.262	0.251
0.429	0.260	0.311
0.348	0.251	0.401
0.261	0.218	0.521
0.177	0.112	0.712
0.118	-	0.883
Tie Lines for Solubility for water(1) + 2,3-butanediol(2) +3-methyl-1-butanol at 323.15K		
Water rich layer		Solvent rich layer
W_{11}	W_{21} W_{31}	W_{13} W_{23} W_{33}
0.940	0.025 0.035	0.130 0.028 0.842
0.905	0.059 0.037	0.141 0.051 0.808
0.860	0.101 0.039	0.152 0.086 0.762
0.840	0.119 0.041	0.175 0.113 0.712