Liquid- Liquid Extraction of 2,3-Butanediol: Performance of Packed Extraction Column Using Continuous Countercurrent

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Abstract— **Liquid- liquid countercurrent extraction of 3% 2,3-butanediol from aqueous solution was carried out using 3-methyl-1-butanol as extracting solvent in presence of 10% sodium carbonate. Study of effect of dispersed phase flow rate and continuous phase flow rate was carried out. Overall mass transfer coefficient was found to increase with dispersed phase flow rate but there is practically no effect of continuous phase flow rate on it. For 3% BD in feed 90% extraction was achieved at solvent to feed ratio of 5, Number of theoretical stages of 1.102 was found to be established during extraction. A run was made using 15% of BD containing 10% sodium carbonate at continuous phase velocity of 0.531 cm/sec and dispersed phase velocity of 2.655 cm/sec corresponding to solvent to feed ratio of 5. Overall mass transfer coefficient of 1.060×10^{-3} Sec^{-1} was obtained corresponding to 94.16% extraction. Number of theoretical stages was found to be 1.292. Hence it is more economical to work with a feed containing up to 15% BD**

Index Terms—**2,3-butanediol, butylene glycol, solvent extraction, countercurrent extraction, mass transfer coefficient, 3-methyl -1-butanol.**

I. INTRODUCTION

Though fermentation of renewable resources has the potential to produce large fraction of oxychemicals and their derivatives that constitute the bulk of feed stock chemicals, ethanol and food grade acetic acid are the only fermentation oxychemicals that are presently economically competitive with the corresponding petrochemical derived compounds (1). Significant advances in research and development are necessary for the potential of fermentation to be realized. The main constraint in its commercialization is economic recovery of chemicals from fermentation broth especially chemicals having high boiling point and having great affinity with water such as 2,3-butanediol, 1,4-butanediol, 1,3-propanediol, glycerol, acetoin etc. If the biotechnological production of these chemicals is to be considered as a commercially viable proposition, it is essential to develop simple and inexpensive methods of their recovery from dilute fermentation broth. Also effective scale up of both difficulties encountered in the design of the separation or recovery system. Thus efficient large scale recovery processes with potential for continuous operation

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should be considered and developed. Liquid- liquid countercurrent extraction of 2,3-butanediol from aqueous solution was undertaken in this study to test the efficiency of method developed by the author (2).

II. MATERIALS AND METHODS:

All the solvents and salts used in the study is of analytical grade. BD was determined by gas chromatography (Perkin Elmer Sigma 3b) using chromosorb 101 coated with 3% FFAP. [3].

III. EXPERIMENTAL METHODS

a) Experimental set up: The essential parts of the equipment used for continuous countercurrent extraction consisted of the extraction column, two feed reservoirs, and two peristaltic pumps. The column was made of 4.0 cm internal diameter and 47 cm long glass tube. The column had a packed length of 28.2 cm and two disengaging spaces at the top and bottom where from the light and heavy phases respectively were withdrawn. The dispersed phase (3-methyl-1-butanol) was fed from a 3 litre flask to the column through a distributor at the bottom of the column. The distributor consists of a diffuser having pore size of 200 to 300 micron. The diffuser tube as connected to a silicon tubing which was attached to a peristaltic pump so that the flow of the dispersed phase could be regulated. Solvent left the column from the top through a silicon tube by gravity. A complete set up of the experiment is shown in fig. 1.

The continuous phase (aqueous phase) was fed to the top of the packing through a tube with a nozzle of 1.5 mm internal diameter which was connected to a peristaltic pump though silicon tube to control the flow rate of the aqueous phase. The column was randomly packed with ceramic rasching rings having dimensions (table1).

b) Experimental procedure: The rasching rings used for packing the column were first washed thoroughly with water to remove all dirt and then cleaned with 3-methyl-1-butanol saturated with water and finally washed thoroughly with distilled water. The column was packed by dropping one piece of packing at a time into the column filled with distilled water. Whenever necessary, the packing was gently moved to eliminate larger voids. Once the desired height of column was packed, the column was drained off. Before making run, a mixture of 3% 2,3-butanediol and 10% sodium carbonate was prepared and the mixture was saturated with 3-methyl-1-butanol. Similarly 3-methyl-1-butanol phase

was saturated with water. The pumps were set properly to deliver these mixtures at the desired flow rates.

c) Mass transfer experiment: For experiments involving mass transfer from the continuous (aqueous) phase to dispersed phase (solvent), the BD concentration in the aqueous phase was held at about 3%, and in the next run at15%. The column was first filled with aqueous phase saturated with solvent phase, followed by regulating the water flow to a desired rate. The dispersed phase saturated with water was then introduced at a predetermined rate. The zone of separation of the two phases was maintained at about 1cm above the level of the packing. The level of the solvent phase was maintained at 3.4cm above the level of the separation zone.

d) Effect of dispersed phase flow rate: To observe the effect of dispersed phase flow rate on mass transfer, the flow rate of the aqueous phase was held constant at 4ml/min and the dispersed phase flow rate was varied. Initially the column was started with dispersed phase flow rate of 4ml/min. Flow rates of aqueous and solvent phases were checked by noting the amount of each liquid collected in a suitable interval. The samples from the outlet streams were taken out at regular intervals of 10 min and were analyzed for BD concentration by GLC. The operation was continued till the steady state was reached. It has been observed that a run time of 80-90mins was required to achieve the steady state.

After achieving the steady state at a particular flow rate of dispersed phase, the column was run for another 10min and samples were then collected from the outlet streams and analyzed for BD concentration. After the run was over, flow rate of the dispersed phase was increased to 8ml/min so as to obtain solvent to feed ratio of 2. The procedure was repeated at flow rates of 12, 16, 20 and 24 ml/min.

e) Effect of continuous phase flow rate: To study the effect of continuous phase flow rate on the mass transfer, experiments were repeated as described above. Only the flow rate of the dispersed phase was held constant at 4 ml/min and that of the continuous phase was varied. The BD concentrations in the effluent streams, after steady state, were analyzed.

f) Evaluation of mass transfer coefficient: The overall mass transfer coefficient ($K_d a$) was calculated based on dispersed phase flow rate as given by Treybal at.el. (4)
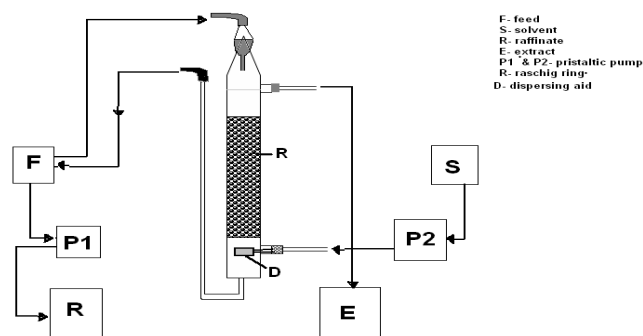

Fig. 1: Continuous Countercurrent Extration Column

IV. RESULT AND DISCUSSION

a) Number of theoretical stages: The number of theoretical stages calculated from Kremser equation (5) is summarized in Table:2. The calculations assumes 3% and

15% BD in feed for getting 95% and 99% recovery. It is observed that when the solvent to feed ratio is 3 or greater than 3, the number of theoretical stages is 3 at 99% recovery and less than 3 at 95% recovery. On the basis of these results, packed column was selected for the recover of BD by continuous countercurrent extraction using 3-methyl-1-butanol as extracting solvent.

TABLE: I DIMENSIONS OF RASCHIG RINGS

Outer Diameter	5.5mm
Internal Diameter	2.0mm
Height	4.5mm
No. the raschig rings used	1500

b) Effect of dispersed phase flow rate: Table 3 shows the summary of the results obtained during continuous countercurrent extraction of BD from aqueous phase to the solvent phase using packed column. It has been observed that at low flow rates large portion of the packing surface are not utilized by the dispersed phase. At increased flow rate of the dispersed phase, for a given rate of the continuous phase, the surface are of packing units seemed to be effectively utilized by the dispersed phase. The dispersed phase flow rate was varied from 0.55×10^{-2} to 2.655×10^{-2} cm per sec. The results clearly indicate that there is increase in $K_d a$ with increase dispersed phase flow rate this is due to the increase in drop surface. This increase can also be attributed to the increased hold up as the dispersed phase flow rate is increased.

Percent extraction was calculated by equation given by Morrison et. Al. (6) for 3% BD in feed. 90% extraction was achieved at solvent to feed ratio of 5, using continuous countercurrent extraction in presence of 10% sodium carbonate. Number of theoretical stages of 1.102 was found to be established during extraction at solvent to feed ratio of 5 corresponding to 90% recovery of BD.

d) Effect of continuous phase flow rate: Results shows that there is practically no effect of continuous phase flow rate on the $K_d a$. It might be due to the reason that the size of the droplets of the dispersed phase is practically independent of the rate of the continuous phase (7). Under such condition, it is reasonably safe to assume that the velocity of one phase relative to the other will be independent of the rate of continuous phase, since the other factors influencing this velocity are not influence by the rate of the continuous phase. This explains why there has been no change in the efficiency of the extraction column with the change in the rate of continuous phase.

An attempt has also been made to carry out extraction using higher concentration of BD in feed solution, as it has been observed that a dilute solution of extract was obtained when the feed solution contains lower concentration of BD. This will necessitate the use of a large quantity of solvent which will create problem in handling. Hence a run was made using 15% (17.322 gmol/cm^3 X 10^{-4}) of BD containing 10% sodium carbonate at continuous phase velocity of 0.531 cm/sec and dispersed phase velocity of 2.655 cm/sec corresponding to solvent to feed ratio of 5. The results are shown in run no. 11 of Table 3. Overall mass

transfer coefficient of 1.060×10^{-3} Sec^{-1} was obtained corresponding to 94.16% extraction. Number of theoretical stages was found to be 1.292. Thus, overall mass transfer coefficient and number of theoretical stages are found to be close to that achieved when 3% of BD was used as feed. On the contrary, a higher percent extraction has been achieved. Extract phase was found to contain 3.262×10^{-4} gmol/cm^3 of BD as compared to 0.513×10^{-4} gmol/cm^3. Thus concentrated extract was obtained which helps in reducing the energy required to concentrate the extract before BD recovery by distillation. Hence it is more economical to work with a feed containing up to 15% BD. This requires the concentration of BD in the feed by simple distillation. It has been found by Othmer et. al. (8) that BD does not form azeotrope with water at any given pressure and there is no loss of BD during distillation. Thus concentration of BD in the feed can be achieved easily by distillation or ripple effective evaporator.

V. CONCLUSION

Number of theoretical stages required for 95% or 99% recovery using solvent to feed ratio of 3 or 5, were found to be less than 3. Overall mass transfer coefficient was found to increase with increase in the flow rate of disperse phase. Continuous phase flow rate has found to have no effect on the overall mass transfer coefficient. Extraction of 90% was achieved using 3-methyl-1-butanol as solvent to feed ratio of 5 in presence of 10% sodium carbonate. It has been found to be more economical to work with high BD concentration (15%) resulting in the necessity to concentrate the feed before subjecting to extraction.

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TABLE 3. SUMMARY OF CALCULATED RESULTS FOR CONTINUOUS COUNTERCURRENT EXTRACTION OF BD IN PACKED COLUMN FOR AQUEOUS SOLUTION

No. Of Runs	Flow Rates, cm/sec		Nav Gmol/sec $X10^{-7}$	Concentration, gmol/cm^3 $X10^{-4}$			Cd Mole/ Cm^{3}x10^{-4}	Mass Transfer Coefficient K_da Sec^{-1} $X10^{-3}$
	U_cx10^{-2}	U_dx10^{-2}		C_{c1} Water In	C_{c2} Water Out	C_{d1} Solvent Out		
1	0.531	0.557	116.78	3.31	1.61	1.72	2.82	0.117
2	0.531	1.114	129.30	3.31	0.86	1.17	2.32	0.158
3	0.531	1.671	126.12	3.31	0.57	0.91	2.06	0.173
4	0.531	2.188	121.83	3.31	0.36	0.71	1.81	0.190
5	0.531	2.655	120.14	3.31	0.33	0.60	1.78	0.191
6	0.531	3.183	119.75	3.31	0.23	0.51	1.61	0.210
7	0.557	0.729	102.41	2.98	1.71	1.27	2.88	0.100
8	1.074	0.729	127.00	2.98	1.86	1.12	3.06	0.120
9	1.592	0.729	72.92	3.18	2.68	0.50	4.14	0.051
10	2.149	0.729	60.23	3.18	2.84	0.33	4.35	0.040
11	0.531	2.655	65.30	17.32	1.01	3.26	1.78	1.060

U_c – Superficial Velocity Of Continuous Phase, Cm/Sec

U_d - Superficial Velocity Of Disperse Phase, Cm/Sec

Nav - Aerage Rate Of Solute Transfer, Gmol/Sec

C_{c1} , C_{c2} And C_{d1} Solute Concentration In Continuous And Disperse Phase At 1 And 2 Ends Of Column Where Solutions Are Concentrated And Dilute Resp.

Cd – Solute Concentration In Disperse Phase

K_da- Overall Mass Transfer Coefficient, Sec^{-1}