# CHAPTER-(6)

# **RESULTS AND DISCUSSION**

# LIQUID-LIQUID EXTRACTION OF AROMATICS IN A PACKED COLUMN

#### **CHAPTER-6**

# RESULTS AND DISCUSSION FOR LIQUID-LIQUID EXTRACTION OF AROMATICS IN A PACKED COLUMN. 6.0.0 ABSTRACT:-

Liquid- Liquid extrication of aromatics has been carried out using two mixed solvents- (Dmf + W and Dmso + W). Packed column could be used as the convenient contacting device due to its simplicity. Since interfacial tension for two systems consisting of aromatics + aliphatics + mixed solvent – (Dmf + W) & (Dmso + W) is very low in comparison to solvents DEG/ TEG, it is expected that values of effective interfacial area and the corresponding rates of extraction are likely to be very high even in a packed column. Hence packed column has been used as a contacting device for mass transfer in this investigation.

Various mass transfer aspects like hold up of the dispersed phase, %aromatics extracted have been considered. The values of NTU, HTU, koc.a and kod.a etc. have been determined and performance of both the solvents has been compared.

Multistage operation has also been carried out. It is observed that values of percentage aromatic extracted can be increased considerably by carrying out multistage operation.

Exhaustive experimental data has been obtained in a packed column of 5 cms. in diameter. However it has been also confirmed that experimental data obtained is free from End effects and Wall effects by performing liquid-liquid extraction in a packed column of 15 cms. in diameter. However only very limited data and experimental runs were taken in this column due to financial constraints.

#### 6.1.0 Preliminary considerations and data Tables:

Preliminary consideration includes fixing the ranges of dispersed phase flow rate (Vd) and continuous phase flow rate (Vc) for operation of a packed column, range of S/F ratio, feed composition, composition of mix solvent and column characteristics like packed column height, size of packing etc. to be used. After fixing the range of

operating variables, accordingly liquid-liquid extraction was carried out in a packed column.

#### **6.1.1 Ranges of different variables:**

(i) Quaternary liquid-liquid phase equilibrium data obtained for mixed solvent – (Dmf + W) which has been reported in previous Chapter-(5) indicates that to have percentage purity of extract of the order of 90% and above, the concentration of anti solvent water has to be of the order of 20% by weight and the temperature of operation of packed column should be of the order of 30°C for liquid- liquid extraction of aromatics.

Under these conditions reasonably good value of percentage aromatics extracted (% AE) and percentage purity of extract (%PE) could be achieved.

If the temperature is lower than  $30^{\circ}$ Ci.e.  $20^{\circ}$ C, the limiting values of percentage AE could be on lower side and if temperature is higher than  $30^{\circ}$ Ci.e.  $40^{\circ}$ C, the range of limiting values of % AE is high; however range of % PE values (purity of extract) is very low i.e. of the order of 70% to 80% for anti solvent concentration of water being of the order of 20% by weight.

Hence to have reasonably good value of both i.e. % AE and %PE, temperature of extraction of the order of 30  $^{0}$ C and concentration of antisolvent in solvent- Dmf of the order of 20% by weight appear to be desirable.

Hence advantage of seasonal changes in temperature at Baroda was taken and all the experiments were performed in Winter season when temperature remains in the range of  $30^{\circ}$ C.Thus for liquid- liquid extraction of aromatics using mixed solvent-(Dmf + W) all the experimental runs in a packed columns were taken during Winter season.

(ii) Quaternary liquid- liquid phase equilibrium data obtained for mixed solvent-(Dmso + W) which has been reported in previous Chapter-(5) indicates that if one requires % purity of extracts of the order of 90% and above the concentration of anti solvent in mixed solvent- Dmso + W has to be of the order of 20% by weight. Further if one operates Extraction column at 30°C, the value of % AE are expected to be on lower side. Hence to have reasonably good values of % AE and %PE, the operation of packed column has to be carried out at higher temperature i.e. at 40°C.Hence to have reasonably good value of. %AE as well as %PE, the temperature of extraction of the order of 40°C and the value of anti solvent concentration of water of the order of 20% by wt. appear to be desirable.

Hence advantage of seasonal changes in temperature in Baroda was taken and all the experimental runs in packed column were performed in Summer season only when temperature remains of the order of  $40^{\circ}$ C.Thus for liquid-liquid extraction of aromatics for the mixed solvent-(Dmso + W), all the experimental runs in a packed column were taken during summer season only.

(iii) Typical representative Benzene (B) of aromatics & typical representative
 Hexane (H) of aliphatics was taken and feed for liquid- liquid extraction consisted of
 50% Benzene + 50% Hexane for all experimental observations.

(iv) Flooding velocities were calculated by Crawford and Wilke <sup>10</sup> equation for systems B- H- Dmf containing 20% water by weight and also for system : B- H- Dmso containing 20% water by weight and accordingly the ranges of flow rates of Vc and Vd were fixed for performing liquid- liquid extraction of aromatics.

For system : B-H- Dmf + W, based on values of flooding velocities, the range of Vc was fixed as: 0.2 cms. / min to 2.0 cms. / min. and the range of Vd was fixed as: 0.1 cms./min. to 0.7 cms./min.

For system : B-H- Dmso + W, based on values of flooding velocities, the range of Vc was fixed as: 0.25 cms. / min to 1.0 cms. / min. and the range of Vd was fixed as: 0.2 cms./min. to 2.0 cms./min.

To have reasonably good value of %AE, S/F ratio of the order 5 by weight appears to be a suitable value based on limiting value of % AE which have estimated from quaternary liquid- liquid phase equilibrium data available for system : B-H- 80% Dmf + 20% W at 30°C and B-H-80% Dmso + 20 % W at 40°CHence S/F ratios have been varied from 1 by wt. to 7 by wt. in this investigation.

(v) Critical size of packing was estimated by the equation proposed by Lewis & Pratt <sup>(161)</sup>. Accordingly to have droplet diameter of dispersed phase independent of packing size of Raschig rings the packing size utilized was 0.7 cms. for 5 cm. column

diameter and for 15 cms diameter column, packing size of Raschig rings utilized was 1.5 cms.

(vi)To avoid End effects and Wall effects, all the considerations discussed in detail by Sharma and Puranik <sup>127</sup> were considered. Accordingly packed bed height of 118.5 cms. was utilized for a column diameter of 5 cms. and packed bed height of 188.5 cms. was utilized for a column diameter of 15 cms.

As has been already pointed out, some selected runs were taken in a column of 15 cms. diameter to ensure that the values of %AE are independent of column diameter, However, performing these experimental runs in a bigger diameter column required large quantity of solvents and both the solvents being costly, due to financial constrains, only limited experiments were performed in a column diameter of 15 cms.

(vii) Liquid-Liquid extraction of aromatics was also carried out in a stage wise manner by performing the operation in three stages.

However, in actual practice while performing stagewise operation, the same packed column was operated as such thrice. Accordingly raffinate phase, coming out of column in a single stage operation was utilized as a feed for Stage-II operation in the same column. Similarly raffinate phase coming out of column during Stage-II, after the same column being operated as a column-for Stage-II, was utilized as feed for stage-III wherein column utilized was a single stage operation packed column.

#### 6.1.2 Summery of Data Tables utilized for Discussion:-

Experimental Data Tables from Table-I to Table -XVIII reported in Chapter-(4): Experimental, have been utilized to prepare derived tables from Table- 1 to Table - 36.

Derived Tables for hold up: Data Tables: 1 to 5, derived tables for % AE and %PE values: Data Tables: 6 to 14 & derived tables for mass transfer: Data Tables- 15 to 22 were prepared based on Experimental Data Tables- I to VIII. Derived tables for multi stage liquid-liquid extraction: Data Tables- 23 to 29 for Multi Stage Operation were prepared from Experimental Data Tables- IX to XIV.

Derived tables from 30 to 36 for liquid-liquid extraction in a bigger column were prepared from Experimental Data Tables- XV to XVIII.

#### 6.2.0 HOLD UP OF DISPERSED PHASE:

In liquid liquid extraction, hold up of the dispersed phase plays the most important role. In case of a packed column, rate of mass transfer is always governed by effective interfacial area available for mass transfer. Rate of mass transfer is directly proportional to effective interfacial area and effective interfacial area is directly proportional to hold up of the dispersed phase. Hence data on values of dispersed phase hold up (% X) appears to be of prime importance.

Hence, values of dispersed phase hold up measured by displacement technique during Liquid-Liquid extraction of aromatics under different sets of conditions of dispersed phase flow rate (Vd) and continuous phase flow rate (Vc) obtained in the present investigation for the following two cases under consideration.

Case-I : B-H-80% Dmf+20% Water

Case-II: B-H-80% Dmso+20% Water

The values of dispersed phase hold up (% X) for the system B-H-80%Dmf+20%Water and for the system. B-H-80%Dmso + 20%Water are reported in Tables – 1(a) & 1(b) and 2(a) & 2(b) respectively.

#### 6.2.1 Dispersed phase hold up for system B+H+80% Dmf+20%W:

The value of dispersed phase hold up reported in Table -1(a) are plotted in Fig-1(a) which represents the plot of fractional hold up (X) Vs. dispersed phase flow rate (Vd) with continuous phase flow rate (Vc) as a parameter. Fig-1. (b) also represents a plot of fractional hold up X Vs. Vc with Vd as a parameter.

Following are the important conclusions from Fig. 1(a) and Fig. 1(b)

(i) Effect of dispersed phase flow rates on Values of Hold up for system B + H +80% Dmf+ 20 % W:

It is observed that as Vd increases, dispersed phase hold up also increases under other wise constant value of Vc. Thus, for example for a fixed value of Vc of the order of 0.3 cm/min with increase in Vd from 1.2 cm/min to 1.8 cm/min., the value of fractional dispersed phase hold up increases from 0.048 to 0.072. Further for a fixed value of Vc =

Q., N.,	*7	***	
Sr.ivo.	V cavg cm/min	Vdavg cm/min	<u> </u>
- 1	0.300	0.161	0.013
- 2	0.325	1.100	0.041
3	0.330	1.200	0.048
4	0.325	1.410	0.056
5	0.350	1.500	0.060
6	0.354	1.620	0.064
7	0.332	1.700	0.068
8	0.345	1.830	0.072
9	0.370	1.900	0.076
10	0.618	0.500	0 020
11	0.620	0.500	0.028
12	0.619	1.220	0.056
13	0.653	0.310	0.011
14	0.645	0.400	0.024
15	0.668	0.500	0.028
16	0.628	1.070	0.048
17	0.625	1.320	0.060
18	0.680	1.815	0.071
19	0.642	1.612	0.072
20	0.632	1.705	0.076
21	1.832	0'700	0.026
22	1.825	0.780	0.030
23	1 830	0.700	0.041
24	1.842	0.000	0.041
25	1.835	1 420	0.040
26	1 837	1.720	0.008
27	1.832	1.720	0.071
28	1 867	1.220	0.071
~~~	1.007	1.520	0.072

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# Table-1 (a)Data Table for Hold up forsystem B-H-80%Dmf -20%W at 30 ° C

# Table-1(b)

## Data Table for Hold up for system B-H-80%Dmf-20%W at 40 ° C with Vd as a parameter

	Sr.No.	Vdavg (cm/min)	Vcavg (cm/min)	$\mathbf{X}$
	1	0.161	0.300	0.013
	2	0.310	0.653	0.011
	3	0.400	0.645	0.024
	4	0.500	0.618	0.028
	5	0.500	0.668	0.028
	6	0.700	1.832	0.036
.	7	0.780	1.825	0.041
1	8	0.800	1.839	0.041
	9	0.870	0.620	0.041
	10	0.970	1.842	0.048
	11	107	0.628	0.048
ſ	12	1.100	0.325	0.041
ľ	13	1.200	0.330	0.048
	14	1.220	0.619	0.056
I	15	1.320	0.625	0.060
		· .		· .
I	16	1.410	0.325	0.056
	17	1.420	1.835	0.068
l	18	1.500	0.350	0.060
	19	1.520	1.867	0.072
	20	1.612	0.642	0.072
	21	1.620	0.354	0.064
	22	1.700	0.332	0.068
ŀ	23	1.705	0.632	0.076
	24	1.720	1.832	0.071
	25	1.815	0.680	0.071
	26	1.830	.0.345	0.072
	27'	1.900	<b>`0.370</b>	0.076
	28	1.990	1.832	0.071



Fig.1 (a) The plot of % Hold up Vs. Vd with Vc as a parameter for tl : B-H-80%Dmf+20%W at 30 ° C





#### : B-H-80%Dmf+20%W at 30 ° C

1.8 cm/min., with increase in Vd from 0.7 cm/min. to 1.5 cm/min. the value fractional hold up (X) increases from 0.036 to 0.072 respectively. Thus, with an increase in Vd the value of dispersed Phase hold up increases substantially.

(ii) Effect of continuous phase flow rates on Values of Hold up for system B+H+80%Dmf+20%W.

For a fixed value of Vd of the order of 1.5 cm/min, with an increase Vc from 0.35 cm/min to 1.85 cm/min., the value of fractional dispersed phase hold up increases from 0.06 to 0.072. Further, for a fixed value of Vd = 0.9 cm/min with an increase in Vc from 0.06 cm/min to 1.85 cm/min, the values of holdup increases from 0.041 to 0.048. Thus increase in Vc appears to have very marginal effect on the values of dispersed phase hold up (X).

From the above discussion, it can be concluded that dispersed phase hold up appears to be a strong function of Vd and at the same time a very weak function of Vc.

#### (iii) Correlating hold up data:

The well-known Laddha's hold-up data correlation can be utilized to correlate hold up data terms of Vd and Vc which also reflects the above-mentioned observations made in the preceding paragraphs.

The Equation is  $Vd + Vc(X/1-X) = \in$ . Vo X(1-X). Thus a plot of Vd + (X/1-X) Vc Vs X(1-X) is expected to be a straight line and the slope is expected to be evaluated for obtaining the value of characteristic droplet velocity (Vo).

The relevant data requiring for plotting for correlation is reported in Table -3 and corresponding plot based on this data is presented in Fig-3. The straight-line nature of the plot clearly indicated that the holdup data obtain for the system:B-H-Dmf+W can be correlated satisfactorily by Laddha's correlation. The values of characteristic droplet velocity (Vo) can be obtained from the slope of the straight line. The slope of straight line for the present system involving solvent Dmf is 31. Hence, the value of characteristic droplet velocity is expected to be 52 cm/min.

The value of  $V_0$  obtained experimentally can also be estimated by generalized correlations available in the literature. Out of various correlations available in the literature, the correlation proposed by Laddha and co workers can be utilized satisfactorily to obtain the value of Vo. The said correlation is as under.

#### $\in V_0 = C/(a_p \rho_c/\epsilon^3 g \Delta \rho)^{1/2}$

Thus, from physico chemical properties of the system fractional voidage of packing and surface area of packing, one can also estimate the value of Vo. The value of Vo predicted by the above equation is 62.2 cm/min (theoretical). The experimental value of Vo calculated by Laddha's hold up data correlation is 52 cm/min (experimental). The agreement between these two values of  $V_0$  experimental and  $V_0$  predicated is reasonably good.

#### 6.2.2 Dispersed phase hold up for system B+H+80% Dmso+20%W:

The value of dispersed Phase hold up reported in Table -2(a) are plotted in Fig-2(a), which represents the plot of fractional dispersed phase hold up (X) Vs. dispersed phase flow rate (Vd) with continuous phase flow rate (Vc) as a parameter. Fig-2(b) also represents a plot of fractional hold up (X) Vs. Vc with Vd as a parameter.

Following are the important conclusions from Fig. 2(a) & 2(b)

(i) Effect of dispersed phase flow rates on Values of Hold up for system B + H +80% Dmso+ 20 % W:

It is observed that as Vd increases, dispersed phase hold up increases under other wise constant value of Vc. Thus for example for a fixed value of Vc of the order of 0.6 cm/min, with increase in Vd from 0.13 to 1.5 cm/min, the values of fractional dispersed phase hold up increases from 0.013 to 0.05. Further for a fixed value of Vc = 0.9 cm/min, with increase in Vd from 1 cm/min. to 1.8 cm/min., the value fractional hold up (X) increase from 0.053 to 0.07 respectively. Thus with an increase in Vd, the value of dispersed Phase hold up increases substantially.

(ii) Effect of continuous phase flow rates on Values of Hold up for system B + H +80% Dmso+ 20 % W:

system B-H-80%Dmso-20%W at 40 0C								
Sr.No.	Vcavg (cm/min)	Vdavg (cm/min)	X					
1	0.100	1.000	0.030					
2	0.100	1.800	0.080					
3	0.300	0.161	0.013					
.4	0.488	0.212	0.014					
5	0.513	1.429	0.086					
6	0.527	0.261	0.018					
7	0.531	1.100	0.069					
8	0.600	0.131	0.013					
9	0.600	0.255	0.018					
10	0.600	1.000	0.040					
11	0.600	1.500	0.050					
12	0.618	0.500	0.028					
13	0.619	1.220	0.056					
14	0.620	0.200	0.015					
15	0.620	0.870	0.041					
16	0.620	0.207	0.013					
17	0.628	1.070	0.048					
18	0.640	1.760	0.108					
19	0.640	0.300	0.020					
20	0.645	0.400	0.024					
21	0.653	0.730	0.036					
22	0.663	0.690	0.033					
23	0.668	0.500	0.028					
24	.0.900	1.000	0.053					
25	0.900	1.500	0.070					

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# Table-2(a) Data Table for Hold up for system B-H-80%Dmso-20%W at 40 00

# Table-2(b) Data Table for Hold up for system B-H-80%Dmso-20%W at 40 0C with Vd as a parameter

Sr.No.	Vdavg (cm/min)	Vcavg (cm/min)	X		
.1 .	0.131	0.600	0.013		
2	0.161	0.300	0.013		
3	0.212	0.488	0.014		
4	0.255	0.527	0.018		
5	0.255	0.600	0.018		
6	0.400	0.645	0.024		
7	0.500	0.668	0.028		
8	0.500	0.618	0.028		
9	1.000	0.900	0.053		
10	1.000	0.600	0.040		
11	1.000	0.100	0.030		
12	1.070	0.628	0.048		
13	1.220	0.619	0.056		
14	1.429	0.513	0.086		
15	1.500	0.900	0.070		
16	1.760	0.640	0.108		
17	1.800	0.100	0.080		



Fig. 2 (a) The plot of % Hold up Vs. Vd with Vc as a parameter for the system







: B-H-80%Dmso+20%W at 40 ° C

The relevant data required for plotting is reported in Table-2(b) and the corresponding plot based on this data is presented in Fig.-2(b)

For a fixed value of Vd of the order of 0.255 cm/min, with an increase in Vc from 0.527 cm/min to 0.6 cm/min., the value of fractional dispersed phase hold up increases from 0.018 to 0.019, i.e. the value of (X) remains practically the same. Further, for a fixed value of Vd = 1 cm/min with an increase in Vc from 0.1 cm/min to 0.9 cm/min, when Vc is increased by a factor of nine, the values of fractional holdup increases just from 0.03 to 0.053. Thus an increase in Vc appears to have very marginal effect on the Values of X.

Further, from the above discussion it can be concluded that dispersed phase hold up appears to be a strong function of Vd and at the same time a very weak function of Vc.

#### (iii) Correlating hold up data:

The well-known Loddha's hold-up data correlation can also be utilized to correlate hold up data for system involving solvent-Dmso.

The relevant data required for plotting for correlation is reported in Table -4 and corresponding plot based on this data is presented in Fig-4. The straight-line nature of the plot clearly indicated that the holdup data obtained for the system:B-H-Dmso+W can be correlated satisfactorily by Laddha's correlation. The values of characteristic droplet velocity can be obtained from the slope of the straight line. The slope of straight line for present system involving solvent Dmso is 23. Hence, the value of characteristic droplet velocity is expected to be 38 cm/min.

The value of  $V_0$  obtained experimentally can also be estimated by generalized correlation available in the literature. Out of various correlations available in the literature the correlation proposed by Laddha and co workers can be utilized satisfactorily to obtain the value of Vo. The said correlation is as under.

#### $\in V_0 = C/(a_p \rho_c/\epsilon^3 g \Delta \rho)^{1/2}$

Thus, from physico chemical properties of the system, fractional voidage of packing and surface area of packing, one can estimate the value of Vo. Value of Vo predicted by above equation is 55 cm/min (theoretical). The experimental value of Vo calculated by Laddha's hold up data correlation is 38 cm/min (experimental). The agreement between these two values of  $V_0$  experimental and  $V_0$  predicated is reasonably good.

for solvent Dmf. Hence it is expected that value rate of mass transfer for liquid-extraction of aromatics are expected to be higher for the system involving solvent Dmso than the system involving solvent Dmf.

Hold up data correlations for the two solvents under consideration are as under:-

For Solvent Dmf:-

Vd+Vc(X/1-X)=[31] X(1-X) From figure-(3)

For Solvent Dmso-

Vd+Vc(X/1-X)=[23] X(1-X) From figure-(4)

Since, the values of slope of st. lines are lower for Solvent Dmso than for Solvent Dmf the values of fractional dispersed phase holdup (X) under otherwise identical conditions are expected to be higher for Dmso than for Solvent Dmf.

It is also known that the value of slope of st line plot of Vd+Vc(X/1-X) Vs X(1-X) can be predicted from physicochemical properties of dispersed phase and continuous phase and column characteristics. These predicted values of ( $\in$ Vo) for Solvents Dmf and Dmso are 37 cm/min and 29 cm/min respectively. Thus, without performing any experimentation, the values of dispersed phase holdup can also be predicted and the comparison of dispersed phase also can be made.

In summary, the values of dispersed phase holdup under otherwise identical conditions of Vc and Vd, are expected to be higher for the system involving Solvent-Dmso than for the system involving Solvent-Dmf.

# Table- 3Data Table for Hold up correlationfor system B-H-80%Dmf-20%W at 30 0C

1 $0.013$ $0.013$ $0.300$ $0.161$ $0.165$ 2 $0.041$ $0.039$ $0.325$ $1.100$ $1.114$ 3 $0.048$ $0.046$ $0.330$ $1.200$ $1.217$ 4 $0.056$ $0.053$ $0.325$ $1.410$ $1.429$ 5 $0.060$ $0.056$ $0.350$ $1.500$ $1.522$ 6 $0.064$ $0.060$ $0.354$ $1.620$ $1.644$ 7 $0.068$ $0.063$ $0.332$ $1.700$ $1.724$ 8 $0.072$ $0.067$ $0.345$ $1.830$ $1.857$ 9 $0.076$ $0.070$ $0.370$ $1.900$ $1.930$ 10 $0.028$ $0.027$ $0.618$ $0.500$ $0.518$ 11 $0.041$ $0.039$ $0.620$ $0.870$ $0.897$ 12 $0.056$ $0.619$ $1.220$ $1.257$ 13 $0.011$ $0.653$ $0.310$	+ve	avg-	g+	+v	/ca	ivg	(X	<b>[/1</b>	-X	)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	0		•	0	0:1	65				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1			1	1.1	14				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	. 1			. 1	1.2	217				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1			1	1.4	29				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1			1	1.5	22				
7 $0.068$ $0.063$ $0.332$ $1.700$ $1.724$ 8 $0.072$ $0.067$ $0.345$ $1.830$ $1.857$ 9 $0.076$ $0.070$ $0.370$ $1.900$ $1.930$ 10 $0.028$ $0.027$ $0.618$ $0.500$ $0.518$ 11 $0.041$ $0.039$ $0.620$ $0.870$ $0.897$ 12 $0.056$ $0.056$ $0.619$ $1.220$ $1.257$ 13 $0.011$ $0.011$ $0.653$ $0.310$ $0.318$ 14 $0.024$ $0.023$ $0.645$ $0.400$ $0.416$ 15 $0.028$ $0.027$ $0.668$ $0.500$ $0.519$ 16 $0.048$ $0.070$ $0.628$ $1.070$ $1.102$ 17 $0.060$ $0.027$ $0.625$ $1.320$ $1.360$ 18 $0.071$ $0.066$ $0.680$ $1.815$ $1.867$ 19 $0.072$ $0.067$ $0.642$ $1.612$ $1.662$ 20 $0.076$ $0.070$ $0.632$ $1.705$ $1.757$ 21 $0.036$ $0.035$ $1.832$ $0.700$ $0.768$ 23 $0.041$ $0.039$ $1.839$ $0.800$ $0.879$ 24 $0.048$ $0.046$ $1.842$ $0.970$ $1.063$ 25 $0.068$ $0.063$ $1.835$ $1.420$ $1.554$ 26 $0.071$ $0.066$ $1.832$ $1.720$ $1.861$ 27 $0.071$ $0.066$ $1.832$ $1.990$ $2.131$	1			1	1.6	44				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1			1	1.7	24				•
9 $0.076$ $0.070$ $0.370$ $1.900$ $1.930$ 10 $0.028$ $0.027$ $0.618$ $0.500$ $0.518$ 11 $0.041$ $0.039$ $0.620$ $0.870$ $0.897$ 12 $0.056$ $0.056$ $0.619$ $1.220$ $1.257$ 13 $0.011$ $0.011$ $0.653$ $0.310$ $0.318$ 14 $0.024$ $0.023$ $0.645$ $0.400$ $0.416$ 15 $0.028$ $0.027$ $0.668$ $0.500$ $0.519$ 16 $0.048$ $0.070$ $0.628$ $1.070$ $1.102$ 17 $0.060$ $0.027$ $0.625$ $1.320$ $1.360$ 18 $0.071$ $0.066$ $0.680$ $1.815$ $1.867$ 19 $0.072$ $0.067$ $0.642$ $1.612$ $1.662$ 20 $0.076$ $0.070$ $0.632$ $1.705$ $1.757$ 21 $0.036$ $0.035$ $1.832$ $0.700$ $0.768$ 23 $0.041$ $0.039$ $1.839$ $0.800$ $0.879$ 24 $0.048$ $0.046$ $1.842$ $0.970$ $1.063$ 25 $0.068$ $0.063$ $1.835$ $1.420$ $1.554$ 26 $0.071$ $0.066$ $1.832$ $1.790$ $2.131$	1			1	1.8	57				
10 $0.028$ $0.027$ $0.618$ $0.500$ $0.518$ $11$ $0.041$ $0.039$ $0.620$ $0.870$ $0.897$ $12$ $0.056$ $0.056$ $0.619$ $1.220$ $1.257$ $13$ $0.011$ $0.011$ $0.653$ $0.310$ $0.318$ $14$ $0.024$ $0.023$ $0.645$ $0.400$ $0.416$ $15$ $0.028$ $0.027$ $0.668$ $0.500$ $0.519$ $16$ $0.048$ $0.070$ $0.628$ $1.070$ $1.102$ $17$ $0.060$ $0.027$ $0.625$ $1.320$ $1.360$ $18$ $0.071$ $0.066$ $0.680$ $1.815$ $1.867$ $19$ $0.072$ $0.067$ $0.642$ $1.612$ $1.662$ $20$ $0.076$ $0.070$ $0.632$ $1.705$ $1.757$ $21$ $0.036$ $0.035$ $1.832$ $0.700$ $0.768$ $22$ $0.041$ $0.039$ $1.825$ $0.780$ $0.858$ $23$ $0.041$ $0.039$ $1.832$ $0.970$ $1.063$ $24$ $0.048$ $0.046$ $1.842$ $0.970$ $1.063$ $25$ $0.068$ $0.063$ $1.835$ $1.420$ $1.554$ $26$ $0.071$ $0.066$ $1.832$ $1.720$ $1.861$ $27$ $0.071$ $0.066$ $1.832$ $1.990$ $2.131$	1			1	1.9	30				
11 $0.041$ $0.039$ $0.620$ $0.870$ $0.897$ $12$ $0.056$ $0.056$ $0.619$ $1.220$ $1.257$ $13$ $0.011$ $0.011$ $0.653$ $0.310$ $0.318$ $14$ $0.024$ $0.023$ $0.645$ $0.400$ $0.416$ $15$ $0.028$ $0.027$ $0.668$ $0.500$ $0.519$ $16$ $0.048$ $0.070$ $0.628$ $1.070$ $1.102$ $17$ $0.060$ $0.027$ $0.625$ $1.320$ $1.360$ $18$ $0.071$ $0.066$ $0.680$ $1.815$ $1.867$ $19$ $0.072$ $0.067$ $0.642$ $1.612$ $1.662$ $20$ $0.076$ $0.070$ $0.632$ $1.705$ $1.757$ $21$ $0.036$ $0.035$ $1.832$ $0.700$ $0.768$ $22$ $0.041$ $0.039$ $1.839$ $0.800$ $0.879$ $24$ $0.048$ $0.046$ $1.842$ $0.970$ $1.063$ $25$ $0.068$ $0.063$ $1.835$ $1.420$ $1.554$ $26$ $0.071$ $0.066$ $1.832$ $1.720$ $1.861$ $27$ $0.071$ $0.066$ $1.832$ $1.990$ $2.131$	0			0	0.5	18				
12 $0.056$ $0.056$ $0.619$ $1.220$ $1.257$ $13$ $0.011$ $0.011$ $0.653$ $0.310$ $0.318$ $14$ $0.024$ $0.023$ $0.645$ $0.400$ $0.416$ $15$ $0.028$ $0.027$ $0.668$ $0.500$ $0.519$ $16$ $0.048$ $0.070$ $0.628$ $1.070$ $1.102$ $17$ $0.060$ $0.027$ $0.625$ $1.320$ $1.360$ $18$ $0.071$ $0.066$ $0.680$ $1.815$ $1.867$ $19$ $0.072$ $0.067$ $0.642$ $1.612$ $1.662$ $20$ $0.076$ $0.070$ $0.632$ $1.705$ $1.757$ $21$ $0.036$ $0.035$ $1.832$ $0.700$ $0.768$ $22$ $0.041$ $0.039$ $1.825$ $0.780$ $0.858$ $23$ $0.041$ $0.039$ $1.839$ $0.800$ $0.879$ $24$ $0.048$ $0.046$ $1.842$ $0.970$ $1.063$ $25$ $0.068$ $0.063$ $1.835$ $1.420$ $1.554$ $26$ $0.071$ $0.066$ $1.832$ $1.720$ $1.861$ $27$ $0.071$ $0.066$ $1.832$ $1.990$ $2.131$	0		•	0	0.8	97				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1			1	1.2:	57				
14 $0.024$ $0.023$ $0.645$ $0.400$ $0.416$ $15$ $0.028$ $0.027$ $0.668$ $0.500$ $0.519$ $16$ $0.048$ $0.070$ $0.628$ $1.070$ $1.102$ $17$ $0.060$ $0.027$ $0.625$ $1.320$ $1.360$ $18$ $0.071$ $0.066$ $0.680$ $1.815$ $1.867$ $19$ $0.072$ $0.067$ $0.642$ $1.612$ $1.662$ $20$ $0.076$ $0.070$ $0.632$ $1.705$ $1.757$ $21$ $0.036$ $0.035$ $1.832$ $0.700$ $0.768$ $22$ $0.041$ $0.039$ $1.825$ $0.780$ $0.858$ $23$ $0.041$ $0.039$ $1.839$ $0.800$ $0.879$ $24$ $0.048$ $0.046$ $1.842$ $0.970$ $1.063$ $25$ $0.068$ $0.063$ $1.835$ $1.420$ $1.554$ $26$ $0.071$ $0.066$ $1.832$ $1.720$ $1.861$ $27$ $0.071$ $0.066$ $1.832$ $1.990$ $2.131$	0	1. S. 1		0	0 <b>.3</b> 2	18	. •	•		
15 $0.028$ $0.027$ $0.668$ $0.500$ $0.519$ $16$ $0.048$ $0.070$ $0.628$ $1.070$ $1.102$ $17$ $0.060$ $0.027$ $0.625$ $1.320$ $1.360$ $18$ $0.071$ $0.066$ $0.680$ $1.815$ $1.867$ $19$ $0.072$ $0.067$ $0.642$ $1.612$ $1.662$ $20$ $0.076$ $0.070$ $0.632$ $1.705$ $1.757$ $21$ $0.036$ $0.035$ $1.832$ $0.700$ $0.768$ $22$ $0.041$ $0.039$ $1.825$ $0.780$ $0.858$ $23$ $0.041$ $0.039$ $1.839$ $0.800$ $0.879$ $24$ $0.048$ $0.046$ $1.842$ $0.970$ $1.063$ $25$ $0.068$ $0.063$ $1.835$ $1.420$ $1.554$ $26$ $0.071$ $0.066$ $1.832$ $1.720$ $1.861$ $27$ $0.071$ $0.066$ $1.832$ $1.990$ $2.131$	0			0	0.4	16	•			
16 $0.048$ $0.070$ $0.628$ $1.070$ $1.102$ $17$ $0.060$ $0.027$ $0.625$ $1.320$ $1.360$ $18$ $0.071$ $0.066$ $0.680$ $1.815$ $1.867$ $19$ $0.072$ $0.067$ $0.642$ $1.612$ $1.662$ $20$ $0.076$ $0.070$ $0.632$ $1.705$ $1.757$ $21$ $0.036$ $0.035$ $1.832$ $0.700$ $0.768$ $22$ $0.041$ $0.039$ $1.825$ $0.780$ $0.858$ $23$ $0.041$ $0.039$ $1.839$ $0.800$ $0.879$ $24$ $0.048$ $0.046$ $1.842$ $0.970$ $1.063$ $25$ $0.068$ $0.063$ $1.835$ $1.420$ $1.554$ $26$ $0.071$ $0.066$ $1.832$ $1.720$ $1.861$ $27$ $0.071$ $0.066$ $1.832$ $1.990$ $2.131$	0		•	0	0.5	19				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1			1	1.1(	02				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1			1	1.30	60	. '			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1			1	1.80	67				
20       0.076       0.070       0.632       1.705       1.757         21       0.036       0.035       1.832       0.700       0.768         22       0.041       0.039       1.825       0.780       0.858         23       0.041       0.039       1.839       0.800       0.879         24       0.048       0.046       1.842       0.970       1.063         25       0.068       0.063       1.835       1.420       1.554         26       0.071       0.066       1.832       1.720       1.861         27       0.071       0.066       1.832       1.990       2.131	· 1.			· 1	1.60	62	'			
21       0.036       0.035       1.832       0.700       0.768         22       0.041       0.039       1.825       0.780       0.858         23       0.041       0.039       1.839       0.800       0.879         24       0.048       0.046       1.842       0.970       1.063         25       0.068       0.063       1.835       1.420       1.554         26       0.071       0.066       1.832       1.720       1.861         27       0.071       0.066       1.832       1.990       2.131	<b>, 1</b> .	. *		<sub>2</sub> 1	1.75	57			· .	
22       0.041       0.039       1.825       0.780       0.858         23       0.041       0.039       1.839       0.800       0.879         24       0.048       0.046       1.842       0.970       1.063         25       0.068       0.063       1.835       1.420       1.554         26       0.071       0.066       1.832       1.720       1.861         27       0.071       0.066       1.832       1.990       2.131	0.			0	0.76	68			·	
23       0.041       0.039       1.839       0.800       0.879         24       0.048       0.046       1.842       0.970       1.063         25       0.068       0.063       1.835       1.420       1.554         26       0.071       0.066       1.832       1.720       1.861         27       0.071       0.066       1.832       1.990       2.131	0.			0	0.85	58				
24         0.048         0.046         1.842         0.970         1.063           25         0.068         0.063         1.835         1.420         1.554           26         0.071         0.066         1.832         1.720         1.861           27         0.071         0.066         1.832         1.990         2.131	0.			0	0.87	79				
25         0.068         0.063         1.835         1.420         1.554           26         0.071         0.066         1.832         1.720         1.861           27         0.071         0.066         1.832         1.990         2.131	. 1.			1	1.06	63				
26         0.071         0.066         1.832         1.720         1.861           27         0.071         0.066         1.832         1.990         2.131	1.			1	1.55	54	•			
	1		•	1	1.86	61		•		
	2.			2	2.13	31				
28 0.072 0.067 1.867 1.520 1.665	1.			1	1.66	65				

#### Table-4

# Data Table for Hold up correlation for system B-H-80%Dmso-20%W at 40 <sup>0</sup>C

Sr.No.	X	X(1-X)	Vcavg	Vdavg	Vdavg+ Vcavg (X /1-X)
• 1	0.030	0.029	0.100	1.000	1.003
2	0.080	0.074	0.100	1.800	1.809
3	0.013	0.013	0.300	0.161	0.165
1					
4	0.014	0.014	0.488	0.212	0.219
5	0.086	0.079	0.513	1.429	1.477
6	0.018	0.018	0.527	0.261	0.271
7	0.069	0.064	0.531	1.100	1.139
8	0.013	0.012	0.600	0.131	0.139
9	0.018	0.018	0.600	0.255	0.266
10	0.040	0.038	0.600	1.000	1.025
11	0.050	0.048	0.600	1.500	1.532
12	0.028	0.027	0.618	0.500	0.518
13	0.056	0.053	0.619	1.220	1.257
14	0.015	0.015	0.620	0.200	` 0.209
15	0.041	0.039	0.620	0.870	0.897
16	0.013	0.013	0.620	0.207	0.215
17	0.048	0.046	0.628	1.070	1.102
18	0.108	0.096	0.640	1.760	1.837
19	0.020	0.020	0.640	0.300	0.313
· .					
20	0.024	0.023	0.645	0.400	0.416
21	0.036	0.035	0.653	0.730	0.754
22	0.033	0.032	0.663	0.690	0.712
23	0.028	0.027	0.668	0.500	0.519
24	0.053	0.050	0.900	1.000	1.050
25	0.070	0.065	0.900	1.500	1.568
	•	•			









# 6.2.3 Comparison of Hold up of dispersed phase for solvent Dmf and Dmso:

Based on the values of dispersed phase hold up reported in Tables-1 and 2 a comparison table has been prepared for comparing the values of dispersed phase hold up under otherwise similar conditions for the two systems under considerations namely

System-I: B-H-80%Dmf-20%W at 30 °C

System-II: B-H-80%Dmso-20%W at 40 °C

The relevant data on hold up of dispersed phase(X) with corresponding Vc and Vd values is reported in Table-5. From the fourteen different values of fractional dispersed phase hold up reported in Table-(5) under otherwise similar conditions, it is observed that values of dispersed phase hold up for solvent Dmso are higher than that with solvent Dmf.

Thus for example, under otherwise identical conditions Vc=0.68 cm/min and Vd in the range of 1.76 cm/min to1.81cm/min, the values of dispersed phase hold up are 0.108 and 0.071 for solvents Dmso and Dmf respectively.

Thus, the values of Hold for solvent Dmso are higher than for solvent Dmf.

Further for example, under otherwise identical conditions Vc=0.6 cm/min and Vd in the range of 1.1cm/min to 1.2cm/min, the values of dispersed phase hold up are 0.069 and 0.056 for solvents Dmso and Dmf respectively.

For solvent Dmso at Vc=0.65 cm/min and Vd=0.2 cm/min, the value of dispersed phase hold up is 0.015.Further for solvent Dmf at Vc=0.65 cm/min and Vd= 0.31 cm/min, the value of dispersed phase hold up is 0.011. It is known that as Vd decreases from 0.31 cm/min to 0.2 cm/min, the value of dispersed phase flow is expected to decrease. Hence by extrapolation at Vc=0.65 cm/min. and Vd=0.2 cm/min., the value of dispersed phase hold up for solvent Dmf is expected to be 0.009. Thus this observation also confirms that dispersed phase Hold up value for the system involving solvent Dmso are higher than solvent Dmf.

Hence it is expected that value of effective interfacial area available for solvent Dmso are expected to be substantial higher than the value of effective interfacial area available

		Table-5				
Comparison	of Hold	upof disp	persed	phase	<b>(X)</b>	for
	solvents	Dmf and	l Dms	0	•	

Sr.No.	Vc Cm/min	Vd Cm/min	<b>X</b>	Solvent
. 1	0.5	1.1	0.069	Dmso
2	0.6	1.2	0.056	Dmf
3	0.64	1.76	0.108	Dmso
4	0.68	1.815	0.071	Dmf
5	0.6	0.255	0.018	Dmso
6	0.65	0.31*	0.011	Dmf
	0.65	0.255*	0.01	*Expected
				by extrapolation
7	0.51	1.429	0.086	Dmso
8	0.625	1.32	0.06	Dmf
9	0.1	1.8	0.08	Dmso
10	0.33	1.7	0:068	Dmf
11	0.62	0.2	0.015	Dmso
12	0.65	0.31*	0.011	Dmf
13	0.65	0.2*	0.009	*Expected
				by extrapolation
13	0.9	1	0.053	Dmso
14	1.84*	0.97	0.048	Dmf
	0.9*	0.97	0.043	*Expected
	i.			by extrapolation

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# 6.3.0 PERCENTAGE AROMATIC EXTRACTED AND PERCENTAGE PURITY OF EXTRACT:

The values of percentage aromatic extracted (%AE) and Percentage purity of extract for system B-H-Dmf-W and B-H-Dmso-W under different sets of conditions are reported in Table 8 and 9 respectively. Continuous phase flow rate (Vcavg) has been varied from 0.3 cm/min to 1.8 cm/min and Vdavg has been varied from 0.1 cm/min to 0.6 cm/min for system B-H-Dmf-W. Further, continuous phase flow rate (Vcavg) has been varied from 0.1 cm/min to-0.9-cm/min and Vdavg has been varied from 0.13 to 1.8 cm/min for system B-H-Dmso-W

6.3.1 Percentage aromatic extracted and Percentage purity extract for system B-H-Dmf-W:

(i) Effect of dispersed phase flow rate (Vdavg) on percentage aromatic extracted (%AE):

The values of percentage aromatic extracted reported in Table 8 are plotted in Fig 8 which is a plot of %AE Vs Vdavg with Vcavg as a parameter. As could be seen from graph with an increase in Vdavg at constant value of Vcavg, the value of percentage aromatic extracted increases. Thus, for example for a fixed value of Vcavg=0.3 cm/min as Vdavg is increased from 0.15 cm/min to 0.35 cm/min, the value of percentage aromatic extracted increases from 37% to 42% however marginally only. Further for a constant value of Vcavg =1.9 cm/min as value of Vdavg is increased from 0.2 cm/min to 0.35 cm/min, the value of percentage aromatic extracted increases from 49% to 59%. Thus with an increase in Vdavg value of percentage aromatic extracted increases marginally .As such with an increase in Vdavg under otherwise identical conditions for a constant value of Vcavg, S/F ratio decreases. Hence value of percentage aromatic extracted is expected to decrease. However with an increase in Vdavg, the value of dispersed phase hold up increases. Hence effective interfacial area available, mass transfer increases, consequently rate of extraction also increases. Hence percentage aromatic extracted increases, however very marginally only.

(ii) Effect of continuous phase flow rate (Vcavg) on percentage aromatic extracted (%AE):

# Table-6Limiting values of %AE

# Table-7Limiting values of %AE

for L-L extraction of Aromatics

for L-L extraction of Aromatics

System: B-H-80Dmf-20%W

System: B-H-80Dmso-20%W

Sr.No.	S/F	%AE limiting	Sr.No.	S/F	%AE limiting	.,, .
1	0.50	30.00				
2	1.00	40.00	1	0.50	16.00	v
3	1.20	. 41.00	2	0,75	21.00	· .
4	1.40	42.00	3	1.00	22.00	
5	1.60	43.00	4	1.25	24.00	, .
6	1.80	44.00	5	1.50	28.00	
7	2.00	45.00	6	2.50	40.00	۰.
8	2.50	46.00	7	3.00	43.00	
8	3.00	47.00	8	3.20	45.00	
9	3.20	52.00	9	4.00	51.00	
10	3.40	55.00	10	4.40	58.00	
11	5.10	70.00	11	5.00	70.00	
12	5.40	73.00	12	6.00	81.00	
13	6.00	76.00	13	6.50	86.00	
14	6.50	78.00	14	6.80	88.00	
15	7.00	80.00	15	7.00	90.00	×
16	7.50	84.00	16	8.00	96.00	
17	8.00	88.00	17	9.00	100.00	
18	8.50	90.00	18	10.00	100.00	
19	9.00	92.00	19	11.00	100.00	
20	9.50	94.00				:
21	10.00	90.00			•	
22	11.00	100.00			· · ·	
23	12.00	100.00			•	



Fig.6 A Plot of limiting value of %AEVs.S/F ratio for system B-H-80%Dmf-20%W at 30 <sup>0</sup>C



Fig.7 A Plot of limiting value of %AEVs.S/F ratio for system B-H-80%Dmso-20%W at 40  $^{6}$ C

	,					
	Table	8		÷.	•	
Data Table for	Values of % arom	atic ex	ctracte	d unde	r diffe	rent
sets of conditio	ns System: B-H-80	)%Dm	f+20%	W at 3	0°C	

Sr.No.	Vcavg	Vdavg	S/F	S/F	% A <sub>Eexp</sub> % A <sub>E limiting</sub>
	cm / min	cm / min	vol	wt	
1	0.248	0.125	1.984	2.401	33.201 45.700
. 2	0.299	0.161	1.857	2.247	37.807 45.300
3	0.299	0.200	1.495	1.809	35.000 43.800
4	0.299	0.260	1.150	1.392	40.000 42.00
• 5•	0.299	0.300	· 0.997	1.206	41.500 41.00
6	0.299	0.340	0.879	1.064	42.000 40.00
7	0.299	0.370	0.808	0.978	42.000 38.000
. 8	0.360	0.128	2.813	3.404	40.500 55.00
9	0.422	0.205	2.059	2.491	39.062 46.000
10	0.422	0.250	1.688	2.043	43.000 45.00
11	0.422	0.300	1.407	1.702	47.100 43.500
12	0.422	0.270	1.563	1.891	43.000 44.50
13	0.422	0.340	1.241	1.502	46.000 42.500
14	0.422	0.370	1.141	1.380	46.000 41.700
15	0.422	0.170	2.482	3.004	38.000 47.000
16	0.580	0.100	5.800	7.019	36.000 80.00
. 17	0.596	0.200	2.980	3.606	41.500 60.000
18	0.596	0.310	1.923	2.327	40.327 45.500
19	0.596	0.260	2.292	2.774	48.000 46.500
20	0.596	0.340	1.753	2.121	49.000 45.200
21	0.596	0.370	1.611	1.949	49.000 44.700
22	1.899	0.631	3.010	3.642	34.547 60.000
23	1.899	0.100	18.990	22.982	38.000 100.000
24	1.899	0.200	9.495	11.491	49.000 100.000
25	1.899	0.300	6.330	, 7.661	60.000 84.00
26	1.899	0.340	5.585	6.759	59.000 78.00
27	1.899	0.370	5.132	6.211	59.000 77.000

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# Table

Effect of Cotinuous phase flow rate (Vc) on percentage Aromatics Extracted (%AE)with Vd as a parameter System B-H-80%Dmf+20%W at 30 ° C

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Sr.No.	V <sub>davg</sub>	V <sub>CAVG</sub>	S/F	S/F	% A <sub>Eexp</sub>	%A <sub>E limiting</sub>
	cm / min	cm / min	vol	wt		
1	0.631	1.899	3.010	3.642	34.547	60.000
2	0.100	0.580	5.800	7.019	36.000	80.00
3	0.100	1.899	18.990	22.982	38.000	100.000
. 4	0.125	. 0.248	1.984	2.401	33.201	45.700
5	0.128	0.360	2.813	3.404	40.500	55.00
6	0.161	0.299	1.857	2.247	37.807	45.300
- 7	0.170	0.422	2.482	3.004	38.000	47.000
8	0.200	0.299	1.495	1.809	35.000	43.800
9	0.200	0.596	2.980	3.606	41.500	60.000
10	0.200	1.899	9.495	11.491	49.000	100.000
,11	0.205	0.422	2.059	2.491	39.062	46.000
12	0.250	0.422	1.688	2.043	43.000	45.00
13	0.260	0.299	1.150	1.392	40.000	42.00
14	0.260	0.596	2.292	2.774	48.000	46.500
15	0.270	0.422	1.563	1.891	43.000	44.50
16	0.300	0.299	0.997	1.206	41.500	41.000
- 17	0.300	0.422	1.407	1.702	47.100	43.500
18	0.300	1.899	6.330	7.661	60.000	84.00
19	0.310	0.596	1.923	2.327	40.327	44.000
20	0.340	0.299	0.879	1.064	42.000	40.00
21	0.340	0.422	1.241	1.502	46.000	42.500
22	0.340	0.596	1.753	2.121	49.000	45.200
23	0.340	1.899	5.585	6.759	59.000	78.00



Fig.8 The plot of % AE Vs. Vd with Vc as a parameter for the system



#### : B-H-80%Dmf+20%W at 30 ° C



#### B-H-80%Dmf+20%W at 30 ° C

Values of percentage aromatic extracted reported in Table 9 are plotted in Fig 9 which is the plot of %AE Vs Vcavg avg, with Vdavg as a parameter. As could be seen from graph with an increase in Vcavg under other wise constant value of Vdavg the value of percentage aromatic extracted increases substantially. Thus for example, for a constant value of Vdavg =0.2 cm/min as value of Vcavg increases from 0.3 cm/min, 0.4 cm/min, 0.6 cm/min to 1.9 cm/min, the value of % aromatic extracted increases from 35%, 39%, 42% to 49% respectively. Further for example for a constant value of Vdavg = 0.34 cm/min as value of Vcavg increases from 0.3 cm/min, 0.4 cm/min, 0.6 cm/min to 1.9 cm/min, the value percentage aromatic extracted increases from 42%, 46%, 49% to 59% respectively. Thus it is crystal clear that with increase in Vcavg for a fixed value of Vdavg the value of percentage aromatic extracted increases substantially. Thus for fixed value of Vdavg when Vcavg increases, the value of S/F ratio also increases. It is known that as S/F ratio increases value of percentage aromatic extracted also increases. Similar conclusions can be drawn from phase equilibrium data of quaternary system under consideration wherein with an increase in [S/F] ratio, the limiting values of %AE also increase under other wise identical conditions. . Hence in Table (9), the corresponding limiting values of %AE are also reported.

6.3.2 Percentage aromatic extracted and Percentage purity extract for system B-H-Dmso-W:

(i) Effect of dispersed phase flow rate (Vdavg) on percentage aromatic extracted (%AE):

The values of percentage aromatic extracted reported in Table 10 are plotted in Fig 10 which is a plot of %AE Vs Vdavg with Vcavg as a parameter. As could be seen from the graph with an increase in Vdavg at constant value of Vcavg the value of percentage aromatic extracted decreases. Thus for example for a fixed value of Vcavg=0.64 cm/min as Vdavg increases from 0.2 cm/min to 0.4 cm/min the value of percentage aromatic extracted decreases from 46.5% to 23%. Further for a fixed value of Vcavg=0.53 cm/min as value of Vdavg is increased from 0.2 cm/min, 1.1 cm/min to 1.4 cm/min the value of percentage aromatic extracted decreases from 47%,18% to 16 % respectively. Thus with an increases in Vdavg under constant value of Vc, the value of percentage aromatic extracted decreases.

As Vdavg increases under constant value of Vcavg, the value of S/F ratio decreases. It is known from phase equilibrium data study that as S/F ratio decreases, the limiting value of % AE also decrease (Refer Table-7) Since S/F ratio decreases, values of % AE also decreases. Hence, with an increase in Vd avg. under otherwise constant value of Vc avg. Value of % aromatic extracted decreases.

Similar trend is observed for other values of Vc, avg. which is a parameter in graph of % AE Vs Vd avg. This is in contrast to behaviour observed for system B-H-Dmf-W where in with an increase in Vd avg. under otherwise constant value of Vc avg. values of % AE were increasing, however marginally.

Hence, it appears that when dispersed phase flow rate increases. dispersed phase holdup value for system. B-H-Dmso-W increases very marginally. Thus effect of decrease in S/F ratio appears to be more pronounced than the effect of increase in holdup when value of Vd avg. increases under other wise constant value of Vc.

(ii) Effect of continuous phase flow rate (Vcavg) on percentage aromatic extracted:

Values of percentage aromatic extracted reported in Table 11 are plotted in Fig. 11 which is the plot of %AE Vs Vcavg with Vdavg as a parameter. As could be seen from graph with an increase in Vcavg under other wise constant value of Vdavg, the value of percentage aromatic extracted increases substantially. Thus for example, for a constant value of Vdavg =0.23 cm/min as value of Vcavg increase from 0.488 cm/min, 0.53 cm/min, to 0.62 cm/min., the value of % aromatic extracted increases from 32, 43% to 46% respectively. Further for example for a constant value of Vdavg = 1 cm/min, as value of Vcavg increases from 0.53 cm/min to 0.6 cm/min, the value percentage aromatic extracted increases from 11.64 to 19%. Thus it is crystal clear that with increase in Vcavg for a fixed value of Vdavg, the value of Vdavg when Vcavg increases, the value of S/F ratio also increases, It is known that as S/F ratio increases value of percentage aromatic extracted also increases.

### Table-10

### Data Table for Values of % aromatic extracted under different sets of conditions for system B-H-80%Dmso-20%W at 40 ° C

Sr.No.	Vcavg	Vdavg	S/F	S/F	% A <sub>Eexp</sub>	$% A_{E \text{ limiting}}$
	cm/min	cm/min	vol	wt		• * <sup>*</sup>
					·	
1	0.300	0.161	1.863	2.601	42.673	40.000
2	0.447	0.138	3.235	4.516	40.000	60.000
3	0.488	0.212	2.302	3.213	32.040	44.500
4	0.513	1.429	0.359	0.501	7.920	16.00
5	0.527	0.261	2.019	2.818	29.726	42.000
6	0.531	1.100	0.483	0.674	11.640	18.000
7	0.534	0.200	2.671	3.728	43.000	47.000
			• •	-		
8	0.600	0.255	2.353	3.284	41.500	45.500
9	0.600	1.000	0.600	0.838	19.000	22.000
10	0.600	1.800	0.333	0.465	14.000	14.000
11	0.618	0.500	1.236	1.725	22.000	29.000
12	0.620	0.207	2.996	4.182	46.520	52.000
13	0.628	1.070	0.587	0.819	17.000	21.000
14	0.631	0.316	1.999	2.790	24.000	41.000
15	0.640	0.300	2.133	2.978	24.140	43.000
16	0.645	0.400	1.613	2.251	23.000	38.000
17	0.653	0.730	0.895	1.249	20.000	25.000
18	0.663	0.690	0.961	1.341	21.000	26.000
19 '	0.668	0.500	1.336	1.865	22.000	30.000
20	0.670	0.640	1.047	1.461	21.000	27.000
21	0.900	1.000	0.900	1.256	15.500	24.00
22	0.900	1.500	0.600	0.838	11.000	22.000

#### Table-11

Effect of continuous phase flow rate on Percentage Aromatics in Extract Vd as parameter for system B-H-80%Dmso-20%W at 40 ° C

Sr.No.	Vdavg . cm/min	Vcavg cm/min	S/F vol	S/F wt	% A <sub>Eexp</sub> %	A <sub>E limitin</sub>
1	0.138	0.447	3.235	4.516	40.000	60.000
2	0.161	0.300	1.863	2.601	42.673	40.000
3	0.212	0.488	2.302	3.213	32.040	44.500
4	0.261	0.527	2.019	2.818	29.726	42.000
5	0.200	0.534	2.671	3.728	43.000	50.000
6	0.255	0.600	2.353	3.285	41.500	45.500
7	0.207	0.620	2.376	2.665	46.520	40.500
8	0.316	0.631	1.999	2.790	24.000	41.000
9	0.300	0.640	2.133	2.978	24.140	43.000
10	0.400	0.645	1.613	2.251	23.000	38.000
. 11	0.500	0.618	1.236	1.725	22.000	29.000
12	0.730	0.653	0.895	1.249	20.000	25.000
13	0.690	0.663	0.961	1.341	21.000	26:000
14	0.500	0.668	1.336	1.865	22.000	30.000
15	0.640	0.670	1.047	1.461	21.000	27.000
16	1.100	0.531	0.483	0.674	11.640	18.000
17	1.000	0.600	0.600	0.838	19.000	22.000
18	1.070	0.628	0.587	0.819	17.000	21.000
19	1.000	0.900	0.900	1.256	15.500	24.00
20	1.429	0.513	0.359	0.501	7.920	16.00
21	1.500	0.900	0.600	0.838	11.000	22.000
22	1.800	0.600	0.333	0.465	14.000	14.000
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Similar conclusions can be drawn from phase equilibrium data of quaternary system under consideration where in with an increase in [S/F] ratio, the limiting values of %AE also increase under other wise identical conditions. Hence in Table (11), the corresponding limiting values of %AE are also reported.

6.3.3 Comparison of values of % aromatic extracted (% AE) for solvents Dmf and Dmso:

Values of aromatic extracted (% AE) for liquid-liquid extraction of aromatic using two solvents namely Dmf and Dmso, have been compared under otherwise identical conditions in Table-12.

In general it is observed that the values of % aromatic extracted (% AE) are higher for solvent Dmf than that for solvent Dmso under otherwise identical conditions. However, for higher values of S/F ratio of the order of 3.5 by wt. and above, the values of % AE for both solvents appear to be comparable.

Thus for example, for a fixed value of Vd=0.26 cm/min, Vc=0.96 cm/min and S/F ratio by Wt.=2.77, the value of % AE for system B-H-Dmf-W is 48%. Further for a fixed value of Vd=0.31 cm/min, Vc=0.63 and S/F ratio by Wt. 2.79, the value of % AE for system B-H-Dmso-W is 24% only which is lower than the value of 48% for the system B-H-Dmf-W.

Further for a fixed value of Vc=0.59 cm/min, Vd=0.26 cm/min and S/F ratio by Wt. 2.7 the value of % AE for system involving solvent Dmf is 48%

For Vc=0.6 cm/min, Vd=0.55 cm/min and S/F ratio by Wt. 3.28 for system involving solvent Dmso, the value of % AE is 41.5 %. If S/F ratio value for system is decreased to 2.7, the value of % AE by extrapolation is expected to decrease to 36%.

Similar observation can be made for other S/F ratio value like 1, 1.5, 2 and 2.2 where in value of %AE for solvent Dmf are higher than that of solvent Dmso.

However, for values of S/F ratio of the order of 3.5 by Wt. and above, the trend observed is different.

L-L extraction of aromatics	solvents Dmf and Dmso	Vc S/F %AE Solver	cm/min wt.	0.59 1.5 23 Dmf	0.65 1.2 20 Dmsc	0.422 . 1.5 46 Dmf	0.67 1.46 21 Dmsc	0.422* 1.5 30 Dmsc	0.596 2.77 48 Dmf	0.63 2.79 24 Dmsc	0.596 3.6 41.5 Dmf	0.534 3.7 43 Dms	0.596 2.7 48 Dmf	0.6 3.28 41.5 Dms	0.596 1.94 44 Dml	0.645 2.25 23 Dms	0.596 2.3 40 Dm	0.631 2.79 24 Dms
		٧d	cm/min	0.68	0.73	0.34	0.64	0.34*	0.26	0.31	0.2	0.2	0.26	0.255	0.37	0.4	0.31	0.316
		Sr.No.		  - 	7	m	4		Ś	9	٢	00	6	10	11	12	13	14

Trable 13

\* expected by extrapolation

However, when S/F ratio reaches to higher value like 3.5, it is observed that values of % AE for both solvents-Dmf as well as Dmso under consideration are comparable. For example, for a fixed value of Vd-0.2 cm/min, Vc=0.59 cm/min and S/F=3.6 the value of % AE for system involving solvent Dmf is 41.5%.Under otherwise comparable operating conditions for a fixed value of Vd=0.2 cm/min, Vc=0.53 cm/min and S/F=3.7 the value of % AE for system involving for Dmso is 43%.Thus for higher values of S/F ratio, the value of % AE for both the solvents appear to be comparable.

#### 6.3.4 Effect of contact time on values of % Aromatic Extracted:

The values of %  $A_E$  under different sets of conditions for two systems under consideration namely B-H-Dmf-W at 30°C and B-H- Dmso- w at 40°C along with corresponding values of Vd, Vc, S/F, and contact time (Ø1 and Ø2) are reported in Tables 13 and 14 respectively. The corresponding plots of %A<sub>E</sub>, Vs. Ø1 or Ø2 with S/F ratio as a parameter are depicted in Figs. (12.a, 12.b) and Figs (13.a, 13.b) for solvents Dmf and Dmso respectively.

#### (i) Effect of $\emptyset$ on values of % A<sub>E</sub> for system B-H-Dmf-W:-

Under otherwise constant value of S/F ratio in the range of 1 to 1.3 as the value of  $\emptyset$ 1 increases from 33 min, 45 min, 67 min to 71 min, the values of % AE increase from 23%, 30%, 42% and 41.5% respectively. As contact time increases, it is understandable that values of %A<sub>E</sub> also increase. However with further increase in contact time from 67 min to 71 min, the value of %A<sub>E</sub> remain practically same because limiting value of %A<sub>E</sub> is reached under this conditions. Further increase in contact time appears to have, thus, no effect on the values of %A<sub>E</sub>. Thus contact time of the order of 60 min appears to be desirable. In the above observation contact time<sup>40</sup> has been based on volumetric dispersed phase flow rate as well as volumetric continuous phase flow rate i.e.(Vd+Vc).

If contact time is based on only volumetric dispersed phase flow rate and the said contact time is labeled as  $\emptyset 2$ , its effect on  $\% A_E$  is very similar to that observed for the effect of  $\emptyset 1$  on  $\% A_E$ .

Thus for example when contact time  $\emptyset 2$  is increased from 63 min, 80 min, 126 min, to 146 min, the values of  $\%A_E$  increase from 23%, 30%, 42% and 41.5 % respectively. Hence contact time  $\emptyset 2$  of the order of 120 min appears to be desirable.
	System : B-H-Dmf- W at 30 <sup>o</sup> C										
Sr.No.	$V_{davg}$	V <sub>CAVG</sub>	S/F	01	02	% A <sub>Eexp</sub>	% A <sub>E limiting</sub>				
	cm / min	cm / min	wt	min	min		v				
. 1	0.260	0.200	1 200	76 975	165 174	40.000	40.00				
	0.200	0.299	1.392	70.823	103.174	40.000	42.00				
2	0.300	0.299	1.200	/1.095	143.151	41.500	41.00				
3	0.340	0.299	1.064	67.207	126.309	42.000	40.200				
4.	0.510	0.450	1.074	45.101	80.101	30.000	40.300				
5	0.680	0.598	1.054	33.604	63.160	23.000	40.100				
							e				
6	0.270	0.422	1.891	62.060	159.056	43.000	42.800				
7	0.300	0.422	1.702	59.481	143.151	47.100	42.700				
8	0.340	0.422	1.502	56.359	126.309	46.000	42.50				
							· · ·				
		· · ·									
9	0.205	0.422	2.491	68.493	209.489	39.062	46.00				
10	0.250	0.422	2.043	63 <b>.</b> 907	171.781	43.000	45.00				
11	0.260	0.596	2.774	50.170	165.174	48.000	46.500				
12	0.340	0.596	2.121	45.882	126.309	49.000	45.200				
13	0.310	0.596	2.327	47.401	138.533	40.327	45.300				
14	0.620	1.200	2.330	24.200	69.266	25.310	45.400				
• .							1				
15	0.085	0.240	3.405	132.000	500.500	58.500	55.30				
16	0.128	0.360	3.404	88.003	335.510	40.500	55.00				
· 17	0.170	0.422	3.004	72.543	252.619	38.000	47.00				
18	0.200	<b></b> .596	3.606	53.951	214.726	41.500	55.50				
L							,				

Table 13

Effect of contact time(O) on the value of % aromatic extracted (%AE)

	Table-14	
Effect of a	ontact time(O) on the value of % aromatic e	xtracted (%AE)

Sr.No.	Vdavg cm/min	Vcavg cm/min	S/F wt	Ø1 min	Ø2 min	% A <sub>Eexp</sub>	% $A_{E \text{ limiting}}$
· 1	1 000	0.600	0.838	26 841	42 945	19.000	21'50
2	1.000	0.000	0.050	25 292	40 136	17 000	21.30
3	1.500	0.900	0.838	17.894	28.630	11.000	21.50
4	0.730	0.653	1.249	31.052	58.829	20.000	24.00
5	0.690	0.663	1.341	31.741	62.239	21.000	25.000
6	0.640	0.670	1.461	32.783	67.102	21.000	27.000
7	1.000	0.900	1.256	22.603	42.945	15.500	24.300
8	0.161	0.300	2.601	93.157	266.741	42.673	40.500
9	0.207	0.620	2.665	51.929	207.465	46.520	41.000
10	0.316	0.631	2.790	45.357	136.019	24.000	42.000
11	0.300	0.640	2.978	45.686	143.151	24.140	43.00
12	0.400	0.645	2.251	41.096	107.363	23.000	39.000
13	0.212	0.488	3.213	61.350	, 202.572	48.040	45.00
14	0.200	0.534	3.728	58.499	214.726	43.000	50.000
15	0.255	0.600	3.285	50.228	168.413	41.500	45.50
16	0.375	0.905	3.281	33.221	112.312	30.110	45.40

system B-H-80%Dmso-20%W at 40 ° C



Fig.12(a) A plot of%AE VsØ1 for solvent Dmf



Fig.12 (b) A plot of %AE VsØ2

for solvent Dmf

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#### Fig.13 (a) A plot of %AE VsO1 for solvent Dmso









Fig.14(a) Comparison of %AE Vs. O1 for solvents-Dmf and Dmso



Fig.14(b) Comparison of %AE Vs. O2 for solvents-Dmf and Dmso

From plots of  $\%A_E$  Vs Ø1 or  $\%A_E$  Vs Ø2 drawn in Figs. 12 (a) and 12(b), Similar observations can be observed for other S/F ratio values being 1.7, 2 and 3.5by wt. under otherwise similar condition of Ø1. For all these observations also Ø 1 of the order of 60 min appears to be favorable condition for liquid-liquid extraction of aromatic using solvent Dmf.

Hence for system consistent of B-H-80% Dmf-20%w for contact time = 60 min, S/F ratio of the order of 3.5 by wt., value of  $%A_E$  is expected to be 58%.

#### (ii) Effect of O as values of % A<sub>E</sub> for system B-H-Dmso-W:-

Under otherwise constant value of S/F ratio in the range of 2.25 to 2.6, as the value of  $\emptyset$ 1 increases from 41 min, 45 min, 51 min, to 93 min, the values of % AE increase from 23%, 24%, 46% and 42.6% respectively. As contact time increases, it is understandable that values of %A<sub>E</sub> also increase. However with further increase in contact time from 51 min. to 93 min ,the value of %A<sub>E</sub> remain practically same because limiting value of %A<sub>E</sub> is reached under this conditions. Further increase in contact time <sup>40</sup> appears to have thus no effect on the values of %A<sub>E</sub>. Thus contact time of the order of 50 min appears to be desirable. In the above observation contact time has been based on volumetric dispersed phase flow rate as well as volumetric continuous phase flow rate i.e.(Vd+Vc).

If contact time is based only on volumetric dispersed phase flow rate (40 ) continuous phase flow rate and the said contact time is labeled as  $\emptyset 2$ , its effect on  $\%A_E$  is very similar to that observed for the effect of  $\emptyset 1$  on  $\%A_E$ 

Thus for example when contact time  $\emptyset 2$  is increased from 107 min, 143 min, 207 min, to 266 min the value of  $\%A_E$  increase from 23% ,24% ,46% and 42.6 %respectively. Hence contact time  $\emptyset 2$  of the order of 200 min appears to be desirable.

From plots of  $\%A_E$  Vs Ø1 or  $\%A_E$  Vs Ø2 drawn in Figs. 13 (a) and 13(b), similar observations can be observed for other S/F ratio values being 1.7, 2 and 3.5 by wt. under otherwise similar condition of Ø1. For all these observations Ø1 of the order of 50-60 min appears to be favorable condition for liquid-liquid extraction of aromatic using solvent Dmso. **602**  Hence for system consisting of B-H-80% Dmso-20%W for contact time = 50-60 min, S/F ratio of the order of 3.5 by wt., value of  $%A_E$  is expected to be 48%

(iii) Favourable operating conditions for %  $A_E$  for Single Stage Extraction:-

Favorable operating condition for liquid-liquid extraction of Aromatic using solvent either Dmf or Dmso are expected to be the following for single stage extraction:-

Contact Time  $\emptyset = 50$  to 60 min, S/F by wt. = 3.5

Feed composition = 50% Benzene + 50% Hexane

Under these conditions the values of Aromatic extracted for solvents Dmf and Dmso is expected to be 58% and 48% respectively.

It is known from quaternary liquid-liquid phase equilibrium data that by adding anti solvent water to solvent Dmf/Dmso,% purity of extract %PE can be increased considerably

Both solvents-Dmf/Dmso contain anti solvent water and its composition in solvent either Dmf or Dmso is of the order of 20% by wt. The values of % purity of extract (%P<sub>E</sub>) obtained in liquid-liquid extraction in packed column under these conditions are of the order of 97-98% which agrees fairly well with the values obtained from quaternary liquid-liquid phase equilibrium data.

#### 6.4.0 MASS TRANSFER ASPECTS DURING LIQUID-LIQUID

#### **EXTRACTION**

As discussed already in previous sections, aromatic can be extracted successfully using newer solvents:- Dmf and Dmso . Purity of extract is expected to be of the order of 95% to 98 % and the values of %AE extracted are expected to be in the range of 42 to 60 % for Dmf and 20 to 40 % using S/F ratio by wt. in the range of 1 to 4 for both solvents.

By either increasing solvent to feed ratio or by carrying L-L extraction unit operation in multistage manner under fixed value of S/F ratio, one can increase value of % AE extracted. The above mentioned solvents are being costly, stage wise operation is preferable. Relevant details about above aspects have been discussed elsewhere.

Hence, liquid- liquid extraction of aromatics was carried out in a paked column under some selected values of flow rates(Vc, Vd) and S/F ratios to study various mass transfer parameters like the following.:

(i) Number of transfer units on dispersed side as well as continuous side.(NTUod, NTUoc)

(ii) Over all Height of transfer unit on dispersed side as well as continuous side.(HTUod, HTUoc).

(iii) Height of transfer unit on dispersed sideas well as on continuos side (HTUd,HTUc).

(iv) Overall volumetric mass transfer coefficient based on dispersed side as well as continuous side(Kod.a, Koc.a).

(v) Individual mass transfer coefficient based on dispersed side as well as continuous side (Koc., Kod).

All the above mentioned mass transfer aspects have been studied for the two systems under considerations

(i) B-H-80% Dmf-20%W at 30 °C

#### (ii) B-H-80% Dmso-20%W at 40 °C

Experimental observations with reference to inlet and outlet flow rates and extract phse as well as raffinate phase compositions etc. along with results obtained for NTU,

HTU,Kod.a and Koc.a are recorded in Tables-I to IV for solvent Dmf and Tables-V to VIII for solvent Dmso.

These tables include Vdi, Vdo values, also Vci, Vco values and the corresponding values of S/F ratio along with extract phase compositions (XBE,XHE and XSE) and raffinate phase composition (XBR,XHR and XSR).

6.4.1 Mass Transfer Data Tables for System :B-H-80%Dmf +20%Wat 30 °C:-

(i) Determination of NTUod and NTUoc for the system B-H-80%Dmf-20%W at 30 °C :-

Values of NTU can be determined conveniently by calculating area under the curve .To calculate NTUod, one has to make a plot of HB1 Vs. 1/(HB1-H'B\*) Further Values of NTUoc can be determined by plotting HB1 Vs. 1/(HB\*-HB1) .For calculating area under the curve by above plots ,Equations- 73 and 74 mentioned in chapter – "Theoretical considerations" have been utilized.

The values required for determining area under the curve for NTUod are reported in Table-15 and the values required for determining area under the curve for NTUoc are reported in Table-16. The corresponding plots are shown in Figs.15-(I-VIII). From these . 11 plots the NTUod and corresponding 11 plots for NTUoc, the values of area under the curve have been determined and the values of NTUod and NTUocthus estimated are reported in main Table-IV and also in Table-17(a) and 17(b).

(ii) Determination of HTUod and HTUoc for the system B-H-80%Dmf-20%W at 30 °C :-

Based on the values of NTUod and NTUoc reported in main Table-IV the values of HTUod and HTUoc have been calculated using equations 75(a) and 75(b) mentioned in the chapter" Theoretical considerations".

These calculated values of NTUod and HTUod are reported in main Table- IV and also in Tables 17(a) and 17(b).

(iii) Determination of Kod.a and Koc.a for the system B-H-80%Dmf-20%W at 30 °C :- Table- 15

Data processing table for determination of area under the curve for NTUod for system B-H- 80% Dmf- 20% w at 30 0C

		101 595	cm D-11	- 00 /0 D	mn- 20 /0	waist		•
Sr.No.	Gcavg	Gdavg	H'B*	H'B*	H'B*	H'B*	H'B*	1/(HB1-H'B*)
1	10.167	2.435	0.603	0.700	0.023	0.130	0.570	1.754
	10.167	2.435	0.603	0.800	0:047	0.330	0.470	2.128
	10.167	2.435	0.603	0.900	0.071	0.600	0.300	3.333
	10.167	2.435	0.603	1.000	0.095	0.800	0.200	5.000
	10.167	2.435	0.603	0.603	0.000	0.000	0.603	1.657
2	5.318	1.350	0.625	0.700	0.019	0.100	0.600	1.667
	5.318	1.350	0.625	0.800	0.045	0.320	0.480	2.083
	5.318	1.350	0.625	0.900	0.070	0.570	0.330	3.030
ļ	5.318	1.350	0.625	1.000	0.095	0.800	0.200	5.000
· ·	5.318	1.350	0.625	0.625	0.000	0.000	0.625	1.601
.3	4.420	1.010	0.669	0.700	0.007	0.000	0.700	1.429
	4.420	1.010	0.669	0.800	0.030	0.210	0.590	1.695
1	4.196	1.009	0.669	0.900	0.056	0.420	0.480	2.083
· ·	4.196	1.009	0.669	1.000	0.080	0.680	0.320	3.125
<b>1</b>	4.196	1.009	0.669	0.669	0.000	0.000	0.669	1.495
. 4	10.527	1.139	-0.364	0.700	0.036	0.250	0.450	2.222
	10.527	1.139	0.364	0.800	0.047	0.340	0.460	2.174
	10.527	1.139	0.364	0.900	0.058	0.460	0.440	2.273
	10.527	1.139	0.364	1.000	0.069	0.540	0.460	2.174
	33.982	5.224	0.364	0.364	0.000	0.000	0.364	2.748
5	33.982	5.224	0.614	0.700	0.013	0.050	0.650	1.538
	33.982	5.224	0.614	0.800	0.029	0.170	0.630	1.587
·	33.982	5.224	0.614	0.900	0.044	0.320	0.580	1.724
	33.982	5.224	0.614	1.000	0.059	0.460	0.540	1.852
	33.982	5.224	0.614	0.614	0.000	0.000	0.614	1.628
6	10.280	2.401	0.540	0.700	0.037	0.270	0.430	2.326
	10.280	2.401	0.540	0.800	0.061	0.470	0.330	3.030
	10.280	2.401	0.540	0.900	0.084	0.700	0.200	5.000
	10.280	2.401	0.540	1.000	0.107	0.900	0.100	10.000
•	10.280	2.401	0.540	0.540	0.000	0.000 ,	0.540	1.851
7	7.354	0.983	0.404	0.700	0.040	0.280	0.420	2.381
	7.354	0.983	0.404	0.800	0.053	0.420	0.380	2.632
	7.354	0.983	0.404	0.900	0.066	0.520	0.380	2.632
	7.354	0.983	0.404	1.000	0.080	0.660	0.340	2.941
	7.354	0.983	0.404	0.404	0.000	0.000	0.404	2.475

Table-	15
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Data processing table for determination of area under the curve for NTUod for system B-H- 80% Dmf- 20% w at 30 0C

Sr.No.	Gcavg	Gdavg	H'B*	H'B*	H'B*	H'B*	H'B*	1/(HB1-H'B*)		
8	5.666	1.073	0.600	0.700	0.019	0.100	0.600	1.667		
	5.666	1.073	0.600	0.800	0.038	0.250	0.550	1.818		
	5.666	1.073	0.600	0.900	0.057	0.430	0.470	2.128		
	5.666	1.073	0.600	1.000	0.076	0.630	0.370	2.703	-   ·	
	5.666	1.073	0.600	0.600	0.000	0.000	0.600	1.667		
9	7.982	1.183	0.495	0.700	0.030	0.200	0.500	2.000		
	7.982	1.183	0.495	0.800	0.045	0.320	0.480	2.083		
	7.982	1.183	0.495	0.900	0.060	0.470	0.430	2.326		
ļ	7.982	1.183	0.495	1.000	0.075	0.600	0.400	2.500	ľ	
1	7.982	1.183	0.495	0.495	0.000	0.000	0.495	2.022		
10	16.425	2.604	0.611	0.700	0.014	0.080	0.620	1.613		
1	16.425	• 2.604	0.611	0.800	0.030	0.200	0.600	1.667		
· ·	16.425	2.604	0.611	0.900	0.046	0.330	0.570	1.754		
	16.425	2.604	0.611	1.000	0.062	0.480	0.520	1.923	• •	
<b>i</b> .	16.425	2.604	0.611	0.611	0.000	0.000	0.611	1.637		
11	7.784	1.330	0.556	0.700	0.025	0.150	0.550	1.818		
	7.784	1.330	0.556	0.800	0.042	0.270	0.530	1.887		
v	7,784	1.330	0.556	0.900	0.059	0.450	0.450	2.222	· ].	
	7.784	1.330	0.556	1.000	0.076	0.620	0.380	2.632	- <b>}</b> .	
1	7.784	1.330 ·	0.556	0.556	0.000	0.000	0.556	1.797		

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Fig.15-I Plot of 1/(HB1-H'B\*) Vs. HB1 for determination of Area under the curve.



Fig.15-II Plot of 1/(HB1-H'B\*) Vs. HB1 for determination of Area under the curve.









## Fig.15-IV Plot of 1/(HB1-H'B\*) Vs. HB1 for determination of Area under the curve.



Fig.15-V Plot of 1/(H'B\* - HB1) Vs. HB1 for determination of Area under the curve.



Fig.15-VI Plot of 1/(H'B\* - HB1) Vs. HB1 for determination of Area under the curve.





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Table-

16

Data processing table for determination of area under the curve for NTUoc for system B-H- 80% Dmf- 20% w at 30 0C

	101 System D-H- 60 76 Dm1- 20 76 W at 50 0C								
Sr.No.	Gcavg	Gdavg	H'B*	H'B*	H'B*	H'B*	H'B*	1/ (H'B*-HB1)	
1	10.167	2.435	0.000	0.113	0.691	0.082	0.082	12.195	
	10.167	2.435	0.040	. 0.113	0.858	0.106	0.066	15.152	
1	10.167	2.435	0.060	0.113	0.942	0.110	0.050	20.000	
· ·	10.167	2.435	0.100	0.113	1.109	0.130	0.030	33.333	
2	5.318	1.350	0.000	0.097	0.622	0.075	0.075	13.333	
	5.318	1.350	0.040	0.097	0.779	0.092	0.052	19.231	
	5.318	1.350	0.060	0.097	0.858	0.102	0.042	23.810	
	5.318	1,350	0.080	0.097	0.937	0.111	0.031	32.258	
<b>.</b> .	5.318	1.350	0.100	0.097	1.016	0.119	0.019	52.632	
3	4.420	1.010	0.000	0.197	0.302	0.041	0.041	24.390	
	4.420	1.010	0.040	0.197	0.477	0.060	0.020	50.000	
	4.420	1.010	0.060	0.197	0.552	. 0.069	0.009	111.111	
	4.420	1.010	0.100	0.197	0.719	0.088	-0.012	-83.333	
	4.420	1.010	0.197	0.197	1.121	0.100	-0.097	-10.326	
4	10.527	1.139	0.000	0.076	0.417	0.051	0.051	19.608	
·	10.527	1.139	0.040	0.076	0.787	0.093	0.053	18.868	
	10.527	1.139	··0.060	0.076	0.972	0.114	0.054	<sup>-</sup> 18.519	
• *	10.527	1.139	0.076	0.076	1.121	0.130	0.054	18.573	
5	33.982	5.224	0.000	0.054	0,664	0.080	0.080	12.500	
·	33.982	5.224	0:040	0.054	0.924	0.110	0.070	14.286	
	33.982	-5.224	0.054	0.054	1.016	0.118	0.064	15.638	
6	10.280	2.401	0.000	0.099	0.503	0.066	0.066	15.152	
	10.280	2.401	0.040	0.099	0.674	0.082	0.042	23.810	
	10.280	2.401	0.060	0.099	0.760	0.091	0.031	32.258	
	10.280	2.401	0.099	0.099	0.926	0.115	0.016	62.116	
7	7.354	0.983	0.000	0.064	0.317	0.043	0.043	23.256	
	7.354	0.983	0.040	0.064	0.616	0.077	0.037	27.027	
	7.354	0.983	0.060	0.064	0.766	0.091	0.031	32.258	
	7.354	0.983	0.064	0.064	0.796	0.094	0.030	33.353	
8	5.666	1.073	0.000	0.081	0.603	0.074	0.074	13.514	
	5.666	1.073	0.040	0.081	0.814	0.096	0.056	17.857	
	5.666	1.073	0.060	0.081	0.920	0.108	0.048	20.833	
	5.666	1.073	0.081	0.081	1.033	0.120	0.039	25.913	
9	7.982	1.183	0.000	0:071	0.451	0.058	0.058	17.241	
	7.982,	1.183	0.040	0.071	0.721	0.087	0.047	21.277	
	7.982	1.183	0.060	0.071	0.856	0.100	0.040	25.000	
	7.982	1.183	0.071	0.071	0.930	0.110	0.039	25.658	
10	16.425	2.604	0.000	0.064	0.633	0.078	0.078	12.821	
	16.425	2.604	0.040	0.064	0.885	0.105	0.065	15.385	
	16.425	2.604	0.064	0.064	1.035	0.120	0.056	17.779	
11	7.784	1.330	0.000	0.071	0.516	0.067	0.067	14.925	
	7.784	1.330	0.040	0.071	0.750	0.090	0.050	20.000	
	7.784	1.330	0.060	0.071	0.867	0.103	0.043	23.256	
	7.784	1.330	0.071	0.071	0.934	0.110	0.039	25.925	







Fig.16(b) The plot of NTUoc Vs. Vc with Vd as parameter for the system :B-H-80% Dmf+20%W at 30<sup>o</sup> C

The values of volumetric overall masstransfer coefficients- Koc.a and Kod.a can be determined using equations- 76(a) and 76(b) mentioned in the chapter -- "Theoretical considerations".

The values of Kod.a and Koc.a so determined using above equations are reported in main Table- IV and also in Tables- 18(a) and 18(b).

6.4.2 Results and discussions for Mass Transfer Aspects System:

#### B-H-80%Dmf +20%W at 30 °C:-

#### (i) Results and discussions for NTUod and NTUoc :

The values of NTUod and NTUoc reported in Tables 17(a) and 17(b) are plotted in Fig.16(a) and 16(b) which is a plot of NTUod Vs. Vd with Vc as a parameter and

NTUoc Vs. Vc with Vd as a parameter respectively. Under otherwise identical conditions of constant value of Vc as Vd increases , the value of NTUod initially decreases . Later on with a further increase in Vd under constant value of Vc, the value of NTUod remains in the range of 0.7 to 0.8 Thus for example , for constant value of Vc of the order of 0.9 cm/min as Vd is increased from 0.315 to 0.51 cm/min ,the value of NTUod remain in the range of 0.68 . Effect of Vc on the value of NTUoc under other wise constant value of Vd appears to be marginal. Thus for example, for constant value of Vd=0.125cm/min as Vc increases from 0.25 cm/min to 0.45 cm/min , the value of NTUoc remains practically same - slightly decreasing from 1.46 to 1.4. Thus, range of NTUoc value happens to be 1.0 to 1.6.

#### (ii) Results and discussions for HTUod and HTUoc :

The values of HTUod and HTUoc reported in Tables 17(a) and 17(b) along with corresponding values of NTUod and NTUoc.

Fig.17(a) is the plot of HTUod Vs. Vd with Vc as a parameter. The parameter Vc has been varied from 0.25 cm/min to 1.8 cm/min. Even though variation takes place by the factor of 7 the value of HTUod follow practically a single straight line. With an increase in Vd values of HTUod initially increases . However , with a further increase in Vd , an increase in value of HTUod is marginal. Thus for example under otherwise constant value of Vc=0.58 cm/min when Vd is increased from 0.13 cm/min to 0.27 cm/min., HTUod increases from 75 to 135 cm. However, with a further increase in Vd from 0.27 to 0.31

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· ·			Table 1/	(a)							
Effect of Vd on HTUod and NTU od with Vc as parameter											
system: <b>B-H-80%Dmf -20%W</b>											
Sr.No.	Vcavg	Vdavg	NTUod	HTUod							
	cm/min	cm/min		cm							
1	0.594	0.131	1.570	75.478							
2	0.580	0.270	0.875	135.429	•						
3	0.580	0.310	0.832	142.428							
4	0.420	0.100	1.530	77.451							
5	0.460	0.132	1.050	112.857							
6	0.450	0.150	0.870	136.207							
7	0.248	0.205	0.760	155.000							
8	0.299	0.161	0.830	142.771	, *						
9	0.900	0.315	0.670	176.866							
10	0.910	0.510	0.680	175.800							
11	1.899	0.631	0.750	158.000							
		·									

#### Table 17(a)

Table.17(b) Effect of Vc on HTUoc and NTU oc with Vd as parameter system: B-H-80%Dmf -20%W

Sr.No.	Vcavg cm/min	Vdavg cm/min	•	NTUoc	HTUoc cm
1	0.248	0.125	·····	1.460	80.100
2	0.360	0.128		1.425	83.158
3	0.420	0.100		1.664	71.214
4	0.460	0.132		1.548	76.550
5	0.450	0.150		1.400	84.643
6	0.594	0.131		1.650	71.818
7	0.580	0.310		1.925	61.558
8.	0.900	0.315	4	1.000	118.500
9	1.510	0.305			120.100
10	-1.899	0.631		0.780	151.923
			na sana ana ani ani ani ani ani		



:B-H-80%Dmf+20%Wat 30<sup>0</sup> C



Fig.17(b) The plot of HTUoc Vs. Vc with Vd as a parameter for the system B-H-80% Dmf+20%W at 30<sup>0</sup> C

cm/min the value of HTUod increases marginally from 135 cm to 142 cm. Thus, with an additional increase in Vd from 0.31 cm/min to 0.63 cm/min, the value of HTUod increases very marginally from 142 cm to 158 cm even though Vd has been varied by factor of 2.

Fig. 17(b) is a plot of HTUoc Vs. Vc with Vd as a parameter. It can be observed from plot that as Vc increases, the value of HTUoc increases initially. With a further increase in the value of Vc, HTUoc increases marginally. Thus for example, when Vc is increased from 0.58 cm/min to 0.9 cm/min under other wise constant value of Vd=0.31 cm/min ,the value of HTUoc increases from 61.5 cm. to 118.5 cm. As Vc is increased from 0.9 to 1.5 cm/min at a constant value of Vd=0.31 cm/min ,the value HTUoc increases very marginally from 118.5 cm to 120.1 cm as if it remains practically constant. Based on the above discussion, it appears that initially HTUoc happens to be function of continuous phase flow rate(Vc). However in the higher range of continuous phase flow rate ,it is expected that HTUocis practically independant of Vc.

#### (iii) Results and discussion for HTUd and HTUc :-

Based on the values of HTUod and HTUoc reported in Tables 17(a) and 17(b) and the discussion held in preceeding paragraph, range of values of HTUod is slightly higher than the rang of HTUoc. Thus for the present system under considerations, resistance to mass transfer for transfer of aromatics from dispersed phase to continuous phase is in both the phases.

Hence it is of utmost importance to estimate value of HTUd and HTUc for liquid -liquid extraction of aromatics where in system investigated for mass transfer aspects is "B-H-80% Dmf-20%W at 30 °C."

The value of HTU<sub>0</sub>c can be plotted against Gc/Gd and the nature of plot is expected to be a straight line. The value of intercept on Y-axis for a value of Gc/Gd =0 is the value of individual height of transfer unit based on continuous phase. i.e. HTUc. From the slope of the straight line plot and utilizing equilibrium curve value i.e. Distribution coefficient, one can estimate value of individual height of transfer unit based on dispersed phase i.e. HTUd. Refer Fig.17(c).

The value of HTUod can be plotted against Gd/Gc and the nature of plot is expected to be a straight line. The value of intercept on Y-axis for a value of Gd/Gc =0 is the value of individual height of transfer unit based on dispersed phase. i.e. HTUd. From

the slope of the straight line plot and utilizing inverse of equilibrium curve value i.e. Distribution coefficient, one can obtain the value of individual height of transfer unit based on continuous phase i.e. HTUc. Refer Fig.17(d).

Based on these two plots-Fig.17(c). and Fig.17(d)- one for HTUoc and other for HTUod, the values of HTUc and HTUd are expected to be 50 cm and 40 cm respectively.

The values of (HTU)c and (HTU)d can also be evaluated from the slope of these two straight line plots.

From a plot of (HTU)oc Vs. Gc/Gd, the slope of the straight line happens to be 5.0 and the slope of equilibrium diagram under consideration Fig.17(c). is 0.1 Thus, the value of HTU)d from the slope of plot is expected to be 50 cm. which compares very well with the intercept value obtained from a plot of HTU)od Vs. Gd/Gc which is being 40 cm.

From a plot of (HTU)od Vs. Gd/Gc the slope of the straight line happens to be 330. . The inverse of slope of equilibrium diagram under consideration is 10. Thus, the value of HTU)c from the slope of plot is expected to be 33 cm. which compares reasonably well with the intercept value obtained from a plot of HTU)oc Vs. Gc/Gd which is being 50 cm.

Thus average value of HTU)c avg and HTU)d avg. are expected to be 42 cm and 45 cm respectively.

The above data clearly indicates that during liquid -liquid extraction of aromatics when benzene gets transferred from dispersed phase to continuous phase (80%Dmf + 20% water )the resistance to mass transfer lies in both the phases. Tare and coworkers<sup>3\*</sup> have studied extraction of furfural from its aqueous solution in a packed column wherein they have used benzene and ethyl acetate as solvent. Their observation indicates that resistance to mass transfer of furfural from aqueous phase to solvent phase appears to be is negligible because value of HTUc are substantially smalle than the value of HTUd. Hence, resistance to mass transfer lies in dispersed phase only and not in continuous phase. Despande and coworkers<sup>§6</sup> have studied liquid -liquid extraction of aromatics using solvent Dmf where in "Resistance to mass transfer" lies in continuous phase and not in dispersed phase.

In the present case under investigation, resistance to mass transfer lies in both the phases i.e. dispersed phase as well as continuous phase.

(iv) Results and Discussion for Kod.a and Koc.a:



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Fig.-17(d) Plot of (HTU)od Vs.Gd/Gc



Fig.17(e) Equilibrium Curve for B-H-80%Dmf-20%W at 30° C

#### (iv) Results and Discussion for Kod.a and Koc.a:

The values of data on over all (volumetric) mass transfer coefficients-Dispersed side-(Kod.a) reported in Table.18(a) are plotted in Fig.-18(a).) Which is the plot of Kod.a Vs Vd with Vc as a parameter. The parameter Vc has been varied from 0.3 cm/min to 1.8 cm/min- by a factor of six.

As could be seen from the graph, the values of over all mass transfer coefficient (dispersed side) appears to have very little effect of continuous phase flow rate (Vc). Thus, Kod. a is a strong function of Vd and weak function of Vc.

Thus for example, for a constant value of Vc of the order of 0.95 cm/min with an increase in Vd from 0.315 cm/min to 0.51 cm/min, the value of Kod. a increases from  $1.7 \times 10^{-3}$  min<sup>-1</sup> to  $3.1 \times 10^{-3}$  min<sup>-1</sup> respectively.

Thus when Vd increases by a factor of 1.5, increase in Kod.a is of the order of 1.8 Further, for a constant value of Vd in the range of 0.5 to 0.6 cm/min , with an increase in Vc from 0.98 cm/min to 1.9 cm/min , the value of Kod. a increases very marginally from  $3.1 \times 10^{-3}$  min<sup>-1</sup> to  $4 \times 10^{-3}$  min<sup>-1</sup> respectively. Thus when Vc increases by a factor of approximately 2, increase in Kod. a happen to be of the order of facor of 1.25 only.

Hence, it can be concluded that overall volumetric dispersed side mass transfer coefficient depends mainly on the values of dispersed phase flow rate  $(V_d)$ .

The values of data on overall (volumetric) mass transfer coefficient – continuous side – (Koc. a) also reported in Table 18(b) are plotted in Fig. 18(b) which is the plot of Koc. a Vs. Vc with Vd as a parameter.

The parameter  $V_d$  has been varied from 0.12 cms/min to 0.63 cms/min by a factor of 5(five).

As could be seen from the graph the values of over all mass transfer coefficient continuous side ,appear to have little effect of dispersed phase flow rate (V<sub>d</sub>). Thus  $K_{oc.a}$  is a strong function of Vc and a weak function of V<sub>d</sub>.

Thus for example, when  $V_d$  is in the range of 0.5 to 0.6 cm/min with an increase in Vc from 0.88 cms/min to 1.89 cms/min, the values of Koc. a increase form  $0.88 \times 10^{-3}$  min<sup>-1</sup> to  $12.5 \times 10^{-3}$  min<sup>-1</sup>.

Table 18(a)

Data Table for Over all Mass Transfer Coefficients -Dispersed side- (Kod.a) along with Koc.a in a packed column

Sr.No.	Vcavg cm/min	system Vdavg cm/min	B-H-80%Dmf -20%W at 30 °C Kod.a min <sup>-1</sup> x10 <sup>3</sup>	Koc.a min <sup>-1</sup> x10 <sup>3</sup>
1	0.420	0.100	1.291	5.898
2	0.450	0.150	1.101	5.316
3	0.460	0.132	1.170	6.009
4	0.580	0.270	1.994	13.705
5	0.580	0.310	2.177	9.422
6	0.900	0.315	1.781	7.595
7	0.860	0.410	2.510	8.410
. 8	0.980	0.510	3.112	8.810
9	1.899	0.631	3.994	12.500
10	0.299	0.161	1.128	5.551
11	0.311	0.210	1.882	5.701

Table.18(b)

# Data Table for Over all Mass Transfer Coefficients -Continuous side- (Koc.a along with Kod.a in a packed column

system B-H-80%Dmf -20%W at 30 °

Sr.No.	VCavg	Vdavg	Koc.a	Kod.a
<b></b>	em/min	em/min	min <sup>-1</sup>	min <sup>-1</sup>
			·	
1	0.360	0.128	4.329	0.772
2	0.420	0.100	5.898	1.291
3	0.460	0.132	6.009	1.170
4	0.299	0.161	5.551	1.128
5	0.450	<b>'</b> 0.150	5.316	1.101
6	0.580	0.310	9.422	2.177
7	0.900	0.315	7.595	1.781
8	0.860	0.415	8.410	2.510
9.	0.880	0.510	8.810	3.112
10	1.899	0.631	12.500	3.994



System B-H-80%Dmf-20%W at 30 °C

Further, when Vc is of the order of 0.45 cm/min when V<sub>d</sub> is increased from 0.1 cm/min to 0.13 cm/min, the value of Koc. a increases marginally from  $0.59 \times 10^{-3}$  min<sup>-1</sup> to  $6 \times 10^{-3}$  min<sup>-1</sup>.

Hence, it can be concluded that over all volumetric continuous side mass transfer coefficient depends mainly on the values of continuous flow rate ( $V_c$ ).

Thus the values of Kod. a appears to be in the range of  $1 \times 10^{-3}$  min<sup>-1</sup> to  $4 \times 10^{-3}$  min<sup>-1</sup>. <sup>1</sup>. Further range of Koc. a values appears to be from  $4 \times 10^{-3}$  min<sup>-1</sup> to  $12 \times 10^{-3}$  min<sup>-1</sup>. The values of Koc. a appear to be higher than the values of Kod. a approximately by a factor of four (4).

It has been already concluded that resistance to mass transfer for the transfer of aromatic – Benzene – from dispersed phase to continuous phase lies in the film of both the phases i.e. dispersed phase film as well as continuous phase film. As a result both the values of mass transfer coefficient Kod. a as well as  $K_{oc}$  a has to be considered while calculating rates of mass transfer during liquid-liquid extraction of aromatics in a packed column using solvent Dmf.

6.4.3 Mass Transfer Data Tables for System:

B-H-80%Dmso +20%W at 40 °C:-

(i) Determination of NTUod and NTUoc for the system B-H-80%Dmso-20%W at 40 °C :-

Values of NTU can be determined conveniently by calculating area under the curve .To calculate NTUod, one has to make a plot of HB1 Vs. 1/(HB1-H'B\*) Further Values of NTUoc can be determined by plotting HB1 Vs. 1/(HB\*-HB1) .For calculating area under the curve by above plots ,Equations- 73 and 74 mentioned in chapter – "Theoretical considerations" have been utilized.

The values required for determining area under the curve for NTUod are reported in Table-19 and the values required for determining area under the curve for NTUoc are reported in Table-20. The corresponding plots are shown in Figs 19-(I-VIII). From these 11 plots the NTUod and corresponding 11 plots for NTUoc, the values of area under the curve have been determined and the values of NTUod and NTUocthus estimated are reported in main Table-VIII and also in Tables-21(a) and 21(b).

### Table- 19

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Data processing table for determination of area under the curve for NTUod

Sr.No. Gd Gc HB'1 HB1 HB HB1-H'B' 1/(HB1-H'B*)   1 12.837 13.192 0.958 0.960 0.002 0.860 1.163   12.837 13.192 0.958 0.970 0.012 0.770 1.299	H'B* 0.100 0.200
1 12.837 13.192 0.958 0.960 0.002 0.860 1.163 12.837 13.192 0.958 0.970 0.012 0.770 1.299	0.100
12.837 13.192 0.958 0.970 0.012 0.770 1.299	0.200
12.837 13.192 0.958 0.980 0.021 0.610 1.639	0.370
12.837 13.192 0.958 0.990 0.031 0.590 1.695	0.400
12.837 13.192 0.958 1.000 0.041 0.500 2.200	0.560
2 8.160 10.610 0.884 0.900 0.013 0.690 1.449	0.210
8.160 10.610 0.884 0.940 0.043 0.390 2.564	0.550
8.160 10.610 0.884 0.980 0.074 0.060 16.667	0.920
8.160 10.610 0.884 1.000 0.090 0.030 33.333	0.970
3 2.485 12.966 0.758 1.000 0.046 0.430 2.326	0.570
4 1.986 10.196 0.670 0.700 0.006 0.540 1.852	0.160
1.986 10.196 0.661 0.800 0.027 0.430 2.326	0.370
1.986 10.196 0.661 0.900 0.047 0.320 3.125	0.580
1.986 10.196 0.661 1.000 0.066 0.200 5.000	0.800
5 1.651 9.998 0.680 0.700 0.003 0.580 1.724	0.120
1.651 9.998 0.680 0.800 0.020 0.510 1.961	0.290
1.651 9.998 0.680 0.900 0.036 0.460 2.174	0.440
1.651 9.998 0.680 1.000 0.053 0.360 2.778	0.640
6 1.160 9.195 0.600 0.700 0.013 0.480 2.083	0.220
1.160 9.195 0.600 0.800 0.025 0.440 2.273	0.360
1.160 9.195 0.600 0.900 0.038 0.400 2.500	0.500
7 2 424 42 228 0 585 0 700 0 020 0 380 2 632	0 320
2 121 12,230 0,000 0,700 0,020 0,000 2,002 2 121 12,230 0,595 0,800 0,037 0,300 3,333	0.020 
2.121 12.230 0.303 0.000 0.037 0.300 3.000 2.121 12.230 0.585 1.000 0.037 0.300 10.000	0.000
2.121 12.200 0.000 1.000 0.012 0.100 10.000 9 4 764 42 604 0.535 0.600 0.000 0.435 0.200	0.000
	0.100
1.701 12.001 0.000 0.700 0.020 0.000 2.770 1.761 12.601 0.525 0.800 0.037 0.320 3.125	0.040
	0.400
1.701 12.001 0.000 0.000 0.001 0.200 0.000 1.761 12.601 0.635 1.000 0.065 0.180 5.556	0.070
0 10 525 10 277 0 021 0 040 0 010 0 650 1 517	0.020
9 10.020 10.077 0.021 0.040 0.019 0.000 1.017 10.525 10.277 0.021 0.060 0.010 0.000 2.273	0.201
	0.020
10.020 10.077 0.021 0.000 0.000 0.100 0.120	0.700
	0.300
	0.310
	0.070
	0.000
	0.100
	0.200
1.591 10.812 0.573 0.000 0.055 0.550 2.65 <i>1</i>	0.450
1.591 10.812 0.573 1.000 0.063 0.210 4.762	-0.790











Fig.19-.II Plot of 1/(HB1-HB\*) Vs. HB1 for determination of Area under the curve.


























Fig.19-.VI Plot of 1/(HB\* - HB1) Vs. HB1 for determination of Area under the curve.















Table- 20

		for syste	em B-H-	80% Dr	nso- 20	0% w at 4(	D OC	•
Sr.No.	Gd	Ge	<b>HB'1</b>	HB1	HB	H'B* -HB1	1/ (H'B*-HB1)	H'B*
1	12.837	13.192	0.045	0.000	1.040	0.081	12.346	0.081
<i>,</i> ``	12.837	13.192	0.045	0.010	1.051	0.072	13.889	0.082
	12.837	13.192	0.045	0.020	1.061	0.063	15.873	0.083
	12.837	13.192	0.045	0.045	1.087	0.039	25.550	0.084
2	8.160	10.610	0.096	0.000	0.950	0.076	13.158	0.076
	8.160	10.610	0.096	0.030	0.989	0.049	20.408	0.079
	8.160	10.610	0.096	0.060	1.028	0.020	50.000	0.080
· · ·	8.160	10.610	0.096	0.080	1.054	0.004	285.714	0.084
3	2.485	12.966	0.049	0.000	0.802	0.062	16.129	0.062
	2.485	12.966	0.049	0.010	0.854	0.055	18.182	0.065
4	1.986	10.196	0.072	0.030	0.907	0.043	23.256	0.073
	1.986	10.196	0.072	0.060	1.061	0.025	40.000	0.085
	1.986	10.196	0.072	0.072	1.125	0.023	44.317	0.095
5	1.651	9.998	0.060	0.000	0.764	0.059	16.949	0.059
	1.651	9.998	0.060	0.020	0.885	0.050	20.000	0.070
	1.651	9.998	0.060	0.040	1.006	0.040	25.000	0.080
,	1.651	9.998	0.060	0.060	1.125	0.036	27.513	0.096
6	1.160	9.195	0.051	0.000	0.602	0:045	22.222	0.045
· .	1.160	9.195	0.051	0.010	0.681	0.042	23.810	0.052
	1.160	9.195	0.051	0.020	0.760	0.039	25.641	0.059
	1.160	9.195	0.051	0.051	1.009	0.017	59.984	0.068
7	2.121	12.238	0.075	0.000	0.605	0.046	21.739	0.046
	2'121	10 028	0.075	0.075	1 028	0.008	125 000	0.083
Q	1 761	12.230	0.075	0.070	1.000	0.000	223.000	0.005
0	1 761	12.071	0.000	0.000	0.555	0.050	20.210 ·	0.030
	1.761	12.091	0.000	0.010	0.000	0.050	27.758	0.040
	1 761	12.071	0.000	0.020	1.002	0.031	00 000	0.031
Q	1.761	12.071	0.000	0.000	0 988	0:079	12 658	0.070
2	10.525	10 377	0.007	0.000	1 027	0.042	23 810	0.072
	10:525	10.377	0.007	0.040	1.027	0.042	166 667	0.086
	10.525	10.377	0.007	0.000 0.007	1.007	0.000	151 600	0.000
10	10.525	10.377	0.007	0.007	0 701	0.002	451.000	0.007
10	20.525	10.377	0.000	0.000	0./71 A 202	0.002	10.147	0.002
	2.013	10.772	0.000	0.020	1 005	0.070	14.200	0.090
•	2.013	10.775	0.003	0.040	1.005	0.004	11.905	0.124
11	2.013	10.775	0.003	0.003	1.120	0.034	29.053	0.097
11	1.391	10.812	0.089	0.000	0.810	0.063	15.873	0.063
	1.591	10.812	0.089	0.040	1.088	0.048	20.833	0.088
	1.391	10.812	0.089	0.089	1.421	0.024	41.007	0.113

Data processing table for determination of area under the curve for NTUoc

(ii) Determination of HTUod and HTUoc for the system B-H-80%Dmso-20%W at 40 °C :-

Based on the values of NTUod and NTUoc reported in main Table-VIII, the values of HTUod and HTUoc have been calculated using equations- 75(a) and 75(b) mentioned in the chapter" Theoretical considerations".

These calculated values of NTUod and HTUod are reported in main Table- VIII and also in Tables 21(a) and 21(b).

(iii) Determination of Kod.a and Koc.a for the system B-H-80%Dmso-20%W at 40 °C :-

The values of volumetric overall mass transfer coefficients- Koc.a and Kod.a can be determined using equations- 76(a) and 76(b) mentioned in the chapter –"Theoretical considerations".

The values of Kod.a and Koc.a so determined using above equations are reported in main Table- VIII and also in Tables- 22(a) and 22(b).

6.4.4 Results and discussions for Mass Transfer Aspects for System : B-H-80%Dmso +20%W at 40 <sup>0</sup>C:-

#### (i) Results and discussions for NTUod and NTUoc :

The values of NTUod and NTUoc reported in Tables 21(a) and 21(b) are plotted in Fig.20(a) and20(b) which are plots of NTUod Vs. Vd with Vc as a parameter and

NTUoc Vs. Vc with Vd as a parameter respectively. As could be seen from the  $\dot{F}$ ig.20(a) under otherwise identical conditions of constant value of Vc as dispersed phase flow rate (Vd) increases, the value of NTUod initially decreases, Later on with a further increase in Vd under constant value of Vc, the value of NTUod remains in the range of 0.7 to 0.8. Thus for example, for constant value of Vc =0.53 cm/min initially as Vd is increased from 0.20 to 0.25 cm/min ,the value of NTUod decreases from 1.2 to 0.8. However, with a further increase in the value of Vd, decrease in NTUod is practically marginal. Thus for example for a constant value of Vc of the order of 0.6 cm/min as Vd is increased from 0.2 cm/min to 1.7 cm/min, the value of NTUod remains in the range of 0.7

to 0.72. Thus in the higher range of values of Vd, the value of NTUod remains practically the same.

As could be seen from Fig.20(b), effect of Vc on the value of NTUoc under other wise constant values of Vd appears to be practically marginal. Thus for example, for constant value of Vd=0.2 cm/min as Vc increases from 0.534 cm/min to 0.62 cm/min, the value of NTUoc remains practically the same - marginally decreasing from 2.5 to 2.2. Thus, range of NTUoc value happens to be 2.0 to 2.5 when Vd is of the order of 0.25 cm/min.

However as the parameter Vd is increased from 0.25 cm/min to 1.7 cm/min, the range of NTUoc value increases substantially from the initial range of (2.2-2.5) to (4.5-5) range. Thus range of NTUod values and range of NTUoc values differ substantially. Hence it is expected that values of HTUod and HTUoc are also likely to differ substantially.

### (ii) Results and discussions for HTUod and HTUoc :

The values of HTUod and HTUoc reported in Tables 21(a) and 21(b) along with corresponding values of NTUod and NTUoc . Fig.21(a) is the plot of HTUod Vs. Vd with Vc as a parameter. As could be seen from the graph, with an increase in Vd values of HTUod increases . When the value of Vd are in the range of 2 cm/min and above an increase in the value of HTUod is expected to be marginal. Thus for example under otherwise constant value of Vc=0.53 cm/min when Vd is increased from 0.2 cm/min to 1.1 cm/min., HTUod increases from 97.1 cm to 141.0 cm. With a further increase in Vd from 1.1 cm/min to 1.5 cm/min ,the value of HTUod increases further from 141 cm to 246 cm. Thus, the range of HTUod value is expected to be of the order of 250 cm.

Fig. 21(b) is a plot of HTUoc Vs. Vc with Vd as a parameter. As could be seen from the plot with an increase in the values of Vc, the value of HTUoc increases marginally only. Thus for example, when Vc is increased from 0.48 cm/min to 0.6 cm/min under other wise constant value of Vd=0.2 cm/min ,the value of HTUoc remains in the range of 53.86 cm to 57 cm. Further for higher values of constant Vd=1.1 cm/min to 1.4 cm/min,the value HTUoc remain in the range of 21 to 28 cm only, even though Vc is increased substantially.Thus the values of HTUoc are expected to be substantially smaller than the values of HTUod.

(iii) Results and discussion for HTUd and HTUc :-



Fig.-20(a) The plot of NTUod Vs Vd with Vc as parameter for the system: B-H-80%Dmso -20%W at 40<sup>o</sup>C



Fig.-20(b) The plot of NTUod Vs Vc with Vd as parameter for the system: B-H-80%Dmso -20%W at 40<sup>0</sup>C

	System	m: B-H-80	)%Dmso -20%W	at 40 0C	
Sr.No.	Vcavg	Vdavg	NTUod	HTUod	
	cm/min	cm/min		cm	
1	0.447	0.138	0.744	159.274	
2	0.534	0.200	1.220	97.131	· .
3	0.488	0.212	0.630	188.095	
4	0.500	0.255	0.840	141.071	
5	0.527	0.261	0.912	129.934	
6	0.531	1.100	0.840	141.071	
· <b>7</b>	0.513	1.429	0.480	246.875	• •
* •				•	
8	0.620	0.207	0.700	169.286	
9	0.600	0.255	0.850	139.412	
10	0.640	1.760	0.720	164.583	
,		]	Table-21(b)	······································	· ·
F	Effect of V	c on HTUc	oc and NTUoc with Vd a	s parameter	r
•	Syster	n: B-H-80	%Dmso -20%W	at 40 0C	
Sr.No.	Vdavg	Vcavg		NTUoc	HTUoc
	cm/min	cm/min			cm
1	0.100	0.447		1 1 5 1	00.010

Table-21(a)

Sr.No.	Vdavg	Vcavg	NTUoc	HTUoc
	cm/min	cm/min		cm
1	0.138	0.447	1.464	80.943
2	0.212	0.488	2.070	57.246
3	0.200	0.534	2.520	47.024
4.	0.207	0.620	2.200	53.864
5	0.255	0.500	2.544	46.580
6	0.255	0.600	1.464	80.943
7	0.316	0.631	4.890	24.233
- 8	1.429	0.513	5.600	21.161
9	1.100	0.531	4.200	28.214
10	1.760	0.640	1.208	98.096

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Based on the values of HTUod and HTUoc reported in Tables 21(a) and 21(b) and the discussion held in preceeding paragraph, range of values of HTUod is substantially higher than the rang of HTUoc. Thus for the present system under considerations, major resistance to mass transfer for transfer of aromatics from dispersed phase to continuous phase is expected to lie in dispersed phase only. And resistance to mass transfer in continuous phase is expected to be low.

Hence it is of utmost importance to estimate value of HTUd and HTUc for liquid -liquid extraction of aromatics where in system investigated for mass transfer aspects is "B-H-80% Dmso-20%W at 40 °C."

The value of HTUoc can be plotted against Gc/Gd and the nature of plot is expected to be a straight line. The value of intercept on Y-axis for a value of Gc/Gd =0 is the value of individual height of transfer unit based on continuous phase. i.e. HTUc. From the slope of the straight line plot and utilizing equilibrium curve value i.e. Distribution coefficient, one can estimate value of individual height of transfer unit based on dispersed phase i.e. HTUd.Refer Fig.-21(c).

The value of HTUod can be plotted against Gd/Gc and the nature of plot is expected to be a straight line. The value of intercept on Y-axis for a value of Gd/Gc =0 is the value of individual height of transfer unit based on dispersed phase. i.e. HTUd. From the slope of the straight line plot and utilizing inverse of equilibrium curve value i.e. Distribution coefficient, one can obtain the value of individual height of transfer unit based on continuous phase i.e. HTUc. Refer Fig.-21(d).

Based on these two plots Fig.-21(c) and 21(d) one for HTUoc and other for HTUod, the values of HTUc and HTUd are expected to be 10 cm and 125 cm respectively.

Based on intercepts of these accompanied plots, the values of (HTU)c and (HTU)d can also be evaluated from the slope of these two lines.

From a plot of (HTU)oc Vs. Gc/Gd, the slope of the straight line happens to be 11.35 and the slope of equilibrium diagram under consideration [Fig.-21(e)] is 0.08. Thus, the value of HTUd from the slope of plot is expected to be 140 cm which compares very well with the intercept value obtained from a plot of HTUod Vs. Gd/Gc which is being 125 cm. as per Fig.-21(d).



Fig.-21(a) The plot of HTUod Vs Vd with Vc as parameter for the system: B-H-80%Dmso -20%W at 40<sup>0</sup>C



Fig.-21(b) The plot of HTUoc Vs Vc with Vd as parameter for the system: B-H-80%Dmso -20%W at 40<sup>0</sup>C







Fig.-21(d) Plot of (HTU)od Vs. Gc/Gd



Fig.21(e) Equilibrium Curve forB-H-80%Dmso-20%W at 40° C

From a plot of (HTU)od Vs. Gd/Gc the slope of the straight line happens to be 75. The inverse of slope of equilibrium diagram under consideration is 12.3. Thus, the value of HTUc from the slope of plot is expected to be 6.5 cm which compares reasonably well with the intercept value obtained from a plot of HTUoc Vs. Gc/Gd which is being 10 cm. as per Fig.-21(c).

Thus the average value of HTUc avg and HTUd avg. are expected to be 8.25 cm and 82.5 cm respectively. Thus the values of HTUc are expected to be 10 times lower than the HTUd values.

The above data clearly indicates that for the system B-H-80%Dmso-20%W when Benzene gets transferred from dispersed phase to continuous phase following are the interesting observations. :-

Major resistance to mass transfer lies in dispersed phase film and not in continuous phase film because of the fact that the value of HTUd is substantially higher than HTUc. Tare and coworkers<sup>()</sup> have studied extraction of furfural from its aqueous solution in a packed column wherein they have used two solvents benzene and ethyl acetate respectively. It is interesting to observe that resistance to mass transfer of furfural from aqueous phase to solvent phase appears to be negligible because value of HTUc is substantially smaller than the value of HTUd. Hence, resistance to mass transfer lies in dispersed phase film only and not in continuous phase film.

Hence, mass transfer aspect in a packed column for transfer of benzene from dispersed phase to continuous phase using solvent dimethyle sulfoxide (Dmso) are expected to be totally different than from the mass transfer aspect in a packed column for transfer of benzene from dispersed phase to continuous phase wherein solvent used is dimethyle formamide (Dmf).

#### (iv) Results and Discussion for Kod.a and Koc.a:

The Values of data on over all (volumetric) mass transfer coefficients- based on dispersed phase film-(Kod. a) reported in Table-22(a) are plotted in Fig-22(a).)The values of Kod. a and the corresponding values of dispersed phase flow rate (Vd) are plotted in the above mentioned fig; where in continuous phase flow rate (Vc) is the parameter. The dispersed phase flow rate (Vd) has been varied from 0.15 cm/min to 1.8 cm/min and the parameter continuous phase flow rate (Vc) has been varied from 0.4 cm/min to 0.65 cm/min

Table 22(a)

Data Table for Over all Mass Transfer Coefficients -Dispersed side- (Kod.a) along with Koc.a in a packed column. System B-H-80% Dmso-20%W at 40 ° C

		System D-	11-00 /0Dm30-20 /0	mattu C	
Sr.No.	Vcavg	Vdavg	Kodxax10 <sup>3</sup>	Kocxax10 <sup>3</sup>	• •
· .	cm/min	cm/min	min <sup>-1</sup>	min <sup>-1</sup>	:
1	0.447	0.138	0.860	5.523	· .
2	0.488	0.212	1.127	8.525	
3	0.534	0.200	2.286	11.359	•
4	0.500	0.255	1.808	10.734	u.
5	0.527	0.261	2.005	4.445	•
6	0.531	1.100	7,773	18.810	
7	0.513	1.429	8.788	24.224	
8	0.620	0.207	1.220	11.517	
9	0.600	0.255	2.000	7.413	· ,
10	0.640	1.760	10.620	6.520	
			1		-

Table 22(b)

Data Table for Over all Mass Transfer Coefficients -Continuous side- (Koc.a) along with Kod.a in a packed column.

Sr.No.	Vdavg cm/min	Vcavg cm/min	Kocxax10 <sup>3</sup> min <sup>-1</sup>	Kodxax10 <sup>.</sup> min <sup>-1</sup>
1.000	0.138	0.447	5.523	0.860
2.000	0.132	0.547	8.125	0.960
3.000	0.212	0.488	8.525	1.127
4.000	0.255	0.500	10.734	1.808
5.000	0.200	0.534	11.359	2.286
6.000	0.207	0.620	11.517	1.220
7.000	1.429	0.513	24.224	8.788
8.000	1.100	0.531	18.810	7.773
9.000	0.316	0.631	26.043	0.552

System B-H-80%Dmso-20%W at 40 ° C







Fig.22(b) Plot for Effect of Vc on Kocxa with Vd as parameter for system B-H-80%Dmso-20%W at 40 ° C

It is observed from the graph that the values of over all mass transfer coefficient (dispersed side) appear to have very little effect of continuous phase flow rate (Vc). Thus, Kod. a appears to be a strong function of Vd and a very weak function of Vc.

Thus for example, for a constant value of Vc of 0.525 cm/min as Vd is increased from 0.25 cm/min to 1.4 cm/min, the value of Kod.a increases from  $2\times10^{-3}$  min<sup>-1</sup> to  $8.8\times10^{-3}$  min<sup>-1</sup>.

Thus when Vd is increased by a factor of 5.5, the value of Kod.a also increases by a factor of 4.4. Thus, Kod. a appears to be a strong function of Vd.

Further, for a constant value of Vd of 0.2 cm/min, as Vc is increased from 0.48 cm/min to 0.65 cm/min, the value of Kod. a remains in the range of  $1.1 \times 10^{-1}$  min<sup>-1</sup> to  $1.2 \times 10^{-1}$  min<sup>-1</sup>. Thus effect of Vc on the values of Kod.a appears to be practically negligible.

The values of the data on overall (volumetric) mass transfer coefficient – continuous side – (Koc. a) are also reported in Table 22(b) The values of Koc. a have been plotted against continuous flow rate (Vc) with dispersed phase flow rate (Vd) as a parameter in Fig. 22(b).

As could be seen from the graph, continuous phase flow rate exercises very little effect on the value of  $K_{oc.}$  a The parameter Vd is having distinct effect as could be seen from three distinct lines corresponding to three distinct values of Vd namely 0.1 cm/min 0.2 cm/min and 1.2 cm/min wherein for a fixed value of Vc of 0.5 cm/min, the value of Koc. a are  $1.0 \times 10^{-3} \text{ min}^{-1}$ ,  $2.2 \times 10^{-3} \text{ min}^{-1}$  and  $8 \times 10^{-3} \text{ min}^{-1}$  respectively.

Thus the value of Kod. a appears in the range of  $1 \times 10^{-3}$  min<sup>-1</sup> to  $10 \times 10^{-3}$  min<sup>-1</sup> Further, the range of Koc. a value appears to be from  $4 \times 10^{-3}$  min<sup>-1</sup> to  $25 \times 10^{-3}$  min<sup>-1</sup>. Thus, the range of Koć. a value appears to be substantially higher than the value of Kod.a

It has been already concluded that the values of HTUc are expected to be 10 times lower than the values of HTUd. Hence resistance to mass transfer is expected to be substantially higher in dispersed phase film and not in continuous phase film. Hence while calculating rates of mass transfer during liquid-liquid extraction of aromatics in packed column using solvent Dmso, Koc.a values are going to play a major role.

# 6.5.0 OPERATION OF PACKED COLUMN IN A STAGE WISE MANNER FOR LQUID\_LIQUID EXTRACTION OF AROMATICS

During liquid liquid extraction of aromatics if the value of solute extracted are on lower side, then by carrying out liquid liquid extraction in a stage wise manner happens to be the appropriate method for liquid liquid extraction of aromatics

Bansod and Puranik<sup>44</sup>, Dhabe and Puranik<sup>67</sup> have carried out liquid liquid extraction of aromatics in a stage wise manner using mixers settler as a contacting device. Hence it was thought desirable to carry out liquid liquid extraction of aromatics in a stage wise manner however using packed column as a contacting device where in information available is very limited.

## 6.5.1 Data Tables for Multistage operation in a packed column:

Liquid liquid extraction of aromatics using solvents Dimethyl formamide (Dmf) and Dimethyl sulfoxide (Dmso) has been carried out in a packed column in single stage and relevant data and its critical analysis about %AE and %PE has been already dealt in.

Instead of using three packed columns in series for multistage extraction, only single column has been used but thrice. Thus, raffinate phase, coming out of column no.1 becomes feed for column no.2. Likewise raffinate phase coming out of column no.2 becomes feed for column no. 3. Thus stage wise operation can be performed in a single column in this manner as if it is extraction consisting of three stages.

Observations recorded for packed column operation in multistage manner, under different flow rate conditions and corresponding values of %AE ,%PE for three stages for system B-H-80%Dmf-20%w at 30 0C are recorded in Tables 23 ,24, and 25 respectively.

Further, observations recorded for packed column operations in multistage manner, under different flow rate conditions and corresponding values of %AE ,%PE for three stages for system B-H-80%Dmso-20%w at 40 0C are recorded in Tables 26 ,27, and 28 respectively. For system B-H-80%Dmf-20%W, experimental runs were performed in winter season as a result temperature of the column was in the range of 30 0C .However, for system B-H-80%Dmso-20%W, experimental runs were performed in summer season as a result temperature of the column was in the range of 30 0C .However, for system B-H-80%Dmso-20%W, experimental runs were performed in summer season as a result temperature of the column was in the range of 40 0C

Table-23

Observations recorded for Packed column operations in multistage manner under different flow rate conditionfor L-L extraction of aromatics.

				systei	m: B-H-8	0%Dmf -	30%W at 3	30 OC	:			
Sr.No.	Vdo	Vdi	Vco	Vci	Vcavg	Vdavg	Vdo	Vdi	Vco	Vci	Vcavg	Vdavg
	gm/min	gmi/min	gm/min	gm/min	gm/min	gm/min	cm/min	cm/min	cm/min	cm/min	cm/min	cm/min
	1.545	2.547	11.574	10.567	Stage-I 11.071	2.046	0.098	0.165	0.629	0.560	0.595	0.132
1.2	1.244	1.545	8.661	8.360	Stage-II 8.510	1.394	0.079	0.098	0.471	0.443	0.457	0.088
1.3	1.140	1.244	6.833	6.730	Stage-III 6.781	1.192	0.072	0.079	0.371	0.357	0.364	0.076
2.1	3.833	4.469	11.414	10.174	Stage-I 10.794	4.151	0.243	0.290	0.620	0.539	0.580	0.266
2.2	3.253	3.833	10.636	10.056	Stage-II 10.346	3.543	0.206	0.243	0.578	0.533	0.556	0.225
2.3	2.939	3.253	8.850	8.536	Stage-III 8.693	3.096	0.186	0.206	0.481	0.452	0.467	0.196
3.1	1.901	2.157	8.653	8.290	Stage-I 8.471	2.029	0.121	0.140	0.470	0.439	0.455	0.130
3.2	1.607	1.901	8.376	8.083	Stage-II 8.229	1.754	0.102	0.121	0.455	0.428	0.442	0.111
, n N	° 1.487	1.607	6.955	6.835	Stage-III 6.895	1.547	0.094	0.102	0.378	0.362	0.370	0.098
4.1	4.292	5.394	17.490	16.391	Stage-I 16.940	4.843	0.272	0.349	0.951	0.869	0.910	0.311
4.2	3,633	4.292	15.343	14.684	Stage-II 15.014	3.963	0.230	0.272	0.834	0.778	0.806	0.251
4.3	3.189	3.633	12.875	12.430	Stage-III 12.652	3.411	0.202	0.230	0.700	0.659	0.679	0.216

Table-24

Processing data Table for selected observations for Packed column operations in stagewise manner on

%AE and %PEunder different sets of conditions

92.600 89.300 92.700 92.800 93.800 83.400 88.400 83.300 83.500 85.600 81.800 73.400 %PE cummulative 77.642 gm/min 89.093 39.000 95.249 84.830 51.000 63.347 62.736 46.000 71.907 77.741 87.261 %AE 39.000 gm/min Stage 62.736 64.000 46.000 48.000 50.000 51.000 55.000 50.000 40.000 45.000 65.000 %AE Bextracted cummulative gm/min 0.545 0.839 1.049 1.708 2.094 1.895 0.810 1.213 1.111 1.027 1.607 0.961 Bextracted system: B-H-80%Dmf-20%W at 30 0C gm/min 1.049 0.659 0.385 0.579 0.289 0.545 0.293 0.122 0.810 0.102 1.027 Stage 0.301 S/Fvol 3.485 2.926 2.857 2.148 2.148 2.148 3.494 3.485 2.857 4:534 4.534 4.534 S/Fwt 2.624 2.624 4.252 4.252 4.252 2.624 3.421 3.421 5.411 5.411 3.421 5.411 Vdavg 0.219 cm/min 0.254 0.089 0.076 0.266 0.199 0.130 0.113 0.099 0.311 0.132 0.227 Vcavg cm/min 0.910 0.679 0.595 0.580 0.556 0.455 0.442 0.370 0.806 0.457 0.364 0.467 Vdavg gm/min 3.096 2.029 1.754 I:547 4.843 3.963 2.046 1.394 1.192 3.543 3.411 4.151 Stage -II Stage -I Stage -III Stage -I Stage -III Stage-III. Stage -II Stage -II 15.0137 Stage-III 8.22945 Stage -I 16.940 Stage -II Vcavg gm/min Stage -I 10.794 0.3462 12.652 11.071 6.895 8.510 8.693 8.471 6.781 Sr.No. 2.2 2.3 4.3 3.2 3.3 4.2 1.2 1.3 2.1 3.1 4.1 

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Table-25 Values of %AE and %PE in multistage L-L extraction in Packed column

		•	System: <b>B</b>	-H-80%D	mf -20%	W at 30 <sup>0</sup> C	<b>r</b> \				
Sr.No.	Vcavg gm/min	Vdavg gm/min	Vcavg cm/min	Vdavg cm/min	S/Fwt	%AEI	%PEI	%AEII cummulative	%PEII.	%AEIII cummulative	%PEIII
<b>*</b> ;	11.071	2.046	0.594	0.131	5.411	62.736	73.400	87.261	83.500	95.249	85.600
7	10.794	4.114	0.580	0.270	2.624	46.000	81.800	71.907	92.800	84.830	93.800
ŝ	8.471	1.992	0.460	0.132	4.252	51.000	83.400	77.741	88.400	89.093	92.600
4	16.940	4.952	0.900	0.315	3.421	39.000	83.300	63.347	89.300	77.642	92.700

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Table-26Observations recorded for Packed column operations in multistage manner under differentflow rate condition

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				system	1: B-H-80	%Dmso -	20%W at	40 0C		•		- -
Sr.No.	Vdo	Vdi	Vco	Vci	Vcavg	Vdavg	Vdo	ipA	Vco	Vci	Vcavg	Vdavg
	gm/min	gm/min	gm/min	gm/min	gm/min	gm/min	cm/min	cm/min	cm/min	cm/min	cm/min	cm/min
-	15 720	17 547	11 657	10.596	Stage -I 11_127	16.634	1.054	1.139	0.550	0.500	0.531	1.097
<b>1.</b> 1					Stage -II							
12	14.713	15.720	11.698	10.690	11.194	15.217	0.986	1.054	0.552	0.504	0.528	1.020
1.3	13.768	14.713	10.949	10.005	Stage-III 10.477	14.241	0.923	0.986	0.517	0.472	0.494	0.955
2.1	3.356	4.391	13.035	12.398	Stage -I 12.716	3.873	0.225	0.285	0.615	0.585	0.600	0.255
2.2	2.842	3.356	11.405	10.891	Stage -II 11.148	3.099	0.191	0.225	0.538	0.514	0.526	0.208
2.3	2.519	2.842	9.548	9.224	Stage-III 9.386	2.680	0.169	0.191	0.451	0.435	0.443	0.180
3.1	2.759	3.512	13.353	12.907	Stage -I 13.130	3.136	0.185	0.228	0.630	0.609	0.620	0.207
3.2	2.290	2.759	12.804	12.334	Stage -II 12.569	2.524	0.154	0.185	0.604	0.582	0.593	0.169
	2.064	2.290	10.460	10.235	Stage -III 10.347	2.177	0.138	0.154	0.494	0.483	0.488	0.146
4.1	3.386	4.529	11.488	10.788	Stage -I 11.138	3.957	0.227	0.294	0.542	0.509	0.527	0.261
4.2	2.908	3.386	10.316	9.839	Stage -II 10.078	3.147	0.195	0.227	0.487	0.464	0.476	0.211
4	2.518	.2.908	8.841	8.451	Stage -III 8.646	2.713	0.169	0.195	0.417	0.399	0.408	0.182
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: Table-27

Processing data Table for selected observations for Packed column operations in stagewise manner for %AE and %PEunder different sets of conditions Svetam: R-H-20%Dmen\_20%W at 40 °C

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	%PE			98.871		97.600		98.100		95.231		96.150		96.550		93.149		92.140	•	93.640		92.138		91.140		92.630	×
	%AE		cummulative	11.640		23.127		33.889	cummulative	41.500		64.900		79.642		46.520		73.260		86.095		29.726		50.808		68.025	
	%AE		Stage	11.640		13.100		14.000	Stage	41.500		40.000		42.000		46.520		50.000		48.000		29.726	• •	30.000		35.000	
•	Bextracted	gm/min	cummulative	1.021		2.029	-	2.973	cummulative	0.911	•	1.425		1.748		0.817		1.287		1.512		0.673		1.151	-	1.541	
W at 40 C	Bextracted	gm/min	Stage I	1.021	Stage II	1.008	Stage III	0.944	Stage I	0.911	Stage II	0.514	Stage III	0.324	Stage I	0.817	Stage II	0.470	Stage III	0.225	Stage I	0.673	Stage II	0.477	Stage III	0.390	
0%07- 0SU	S/Fvol	-	T	0.484		0.484		0.484		2.353		2.353		2.353		3.004	·	3.004		3.004		2.022		2.022		2.022	  
R-H-80%D	S/Fwt			0.669		0.669		0.669		3.283		3.283		3.283		4.187		4.187	•	4.187	,	2.814		2.814		2.814	
System:	Vdavg	cm/min		1.097		1.020		0.955		0.255		0.208		0.180	·	0.207		0.169		0.146		0.261		0.211	· .	0.182	
	Vcavg	cm/min		0.531		0.528		0.494		0.600	÷	0.526		0.443	• •	0.620	-	0.593		0.488		0.527		0.476		0.408	•
	Vdavg	gm/min		16.634		15.217		14.241		3.873		3.099		2.680		3.136		2.524		2.177	v	3.957		3.147	·	2.713	
	Vcavg	gm/min	Stage -I	11.127		11.194		10.477	Stage -I	12.716		11.148		9.386		13.130		12.569		10.347		11.138		10.078	- 1	8.646	
	Sr.No.			1.1	34	1.2		<b></b>	-	2.1		2.2	•	2.3		3.1		3.2		3.3		4.1		4.2	-	4.3	

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Table-28Values of %AE and %PE in multistage L-L extraction in Packed column

		·	Svst	tem: B-I	<b>G%08-</b> E	mso -20%	6W at 4	0°C	•		•
Sr.No.	Vcavg gm/min	Vdavg gm/min	Vcavg cm/min	Vdavg cm/min	S/Fwt	%AEI	%PEI	%AEII	%PEII	%AEIII	%PEIII
	11.127	16.634	0.531	1.097	0.669	11.640	98.871	23.120	97.600	33.880	98.100
2	12.716	3.873	0.600	0.255	3.283	41.500	95.231	64.900	96.150	79.620	96.550
m	13.130	3.136	0.620	0.207	4.187	46.520	93.149	73.260	92.140	86.090	93.640
4	11.138	3.957	0.527	0.261	2.814	29.726	92.138	50.800	91.140	68.020	92.630

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# 6.5.2 Percentage Aromatics extracted and % Purity of extract for system B-H -80% Dmf-20%W :

During liquid liquid extraction of aromatics under mass transfer conditions for fixed value of Vdi and Vci after steady state is reached, the values of Vdo and Vco also achieve constant values. The value of Vco is expected to be more than Vci because of transfer of aromatics from feed to extract phase and correspondingly value of Vdo is expected to be less than Vdi because of transfer of aromatics from feed to extract phase. Hence, all these observations for three stage operation have been recorded along with average value of Vd and Vc.. Corresponding amount of aromatics extracted and %AE, %PE values for every stage are recorded against average value of Vc and Vd i.e.Vcavg. and Vd avg.

As could be seen from first set of observation-(1.1) from Table-24, during stage 1, for Vc avg. =0.594 cm/min and Vdavg= 0.131 cm/min, for fixed value of S/F=5.4 by wt., the value of % aromatics extracted is 62.7% and % purity extract being 73.4 %.

During stage 2 observation-(1.2), raffinate phase coming out from column Nol with Vdo=0.098 cm/min becomes feed Vdi for column 2. Additional benzene which get extracted in column 2 is 0.3 gm/min. Individual value of % aromatic extracted for stage 2 is 65%.

During stage 3, observation-(1.3) raffinate phase coming from column No2 with Vdo=0.079 cm/ min becomes feed Vdi for column 3. Additional benzene which gets extracted in column 3 is 0.102 gm/ min. Individual value of % aromatic extracted for stage 3 is 64%. Cumulative value of benzene extracted after stage 3 is 95.25% and purity of extract being 85.6%. Thus from above discussion it is crystal clear that by performing stage wise operation, value of % aromatic extracted can be increased from 62.7% to 95.25% and purity of extract is expected to be of the order of 86%.

Further, as could be seen from second set of observation-(2.1) from Table-24, during stage 1, for Vc avg. =0.58 cm/min and Vdavg= 0.27 cm/min, for fixed value of S/F=2.62 by wt. the value of % aromatics extracted is 46% and % purity extract being 81.8 %.

During stage 2, observation-(2.2) raffinate phase coming out from column No1 with Vdo=0.243 cm/min becomes feed Vdi for column 2. Additional benzene which gets extracted in column 2 is 0.579 gm/min. Individual value of % aromatic extracted for stage 2 is 48%. Cumulative value of benzene extracted after stage 2 is 71.9% and% purity of extract being 92.8%.

During stage 3 observation-(2.3), raffinate phase coming out from column No2 with Vdo=0.206 cm/ min becomes feed Vdi for column 3 . Additional benzene which gets extracted in column 3 is 0.289 gm/ min. Individual value of % aromatic extracted for stage 3 is 50% . Cumulative value of benzene extracted after stage 3 is 84.83% and % purity of extract being 93.8%. Thus from above discussion it is crystal clear that by performing stage wise operation , value of % aromatic extracted can be increased from 46% to 84.83% and % purity of extract is expected to be of the order of 92-93%

Thus from above two observations ,Vcavg.=0.594 cm/min and Vd avg. =0.31 cm/min. for S/F= 5.41 by wt., the value of % aromatic extracted is 95.255 with % purity of extract being 85.6%. As S/F ratio by wt. is decreased to 2.64(by wt.) for Vc avg. =0.58 cm/min and Vd avg. 0.27 cm/min, the value of % AE extracted decreases to 84.835 corresponding purity being 93.8% .This confirms that as S/F ratio decreases under otherwise identical conditions, the value of % AE decreases.

For other two values of S/F by wt. namely 3.42 (Vc=0.9 cm/min and Vd avg. 0.31 cm/min ) and 4.25 (Vc=0.46 cm/min and Vd avg.= 0.13 cm/min) observations-(3) and (4) from Table-24 the values of % aromatic extracted are 77.6% and 89 % respectively. The remaining two sets of observations can also be analysed in a similar manner, as adopted for earlier two sets of observations.

It is further observed that for S/F=2.6 by wt. (Vcavg.=0.58 cm/min and Vdavg.=0.2 cm/min) the value of %AE extracted for three stage operation is 84.83 % and for the value of S/F=3.4 by wt. (Vcavg.=0.9 cm/min and Vdavg.=0.315 cm/min), the value of %AE extracted happens to be 77.6 %. As such value should have been increased further beyond 84% as S/F ratio for second case is higher than that for previous case.

Critical analysis indicates that contact time O for S/F=3.4 by wt. is expected to be 50% lower than contact time O for the case where in S/F is 2.6 by wt. As a result the value of % AE for second case is lower than that for the first case.

Thus by performing stage wise operation, the value of % aromatic extracted of the order of 95% can be obtained in a packed column with purity of extract being of the order of 93 % for system B-H-80%Dmf-20%W under following sets of operating conditions :-

(i)Temperature of column for all three columns=30 0C

(ii)Vcavg.= 11.07 gm/min=0.591 cm/min

(iii)Vdavg.= 2.04 gm/min=0.131 cm/min

(iv) S/F=5.41 by wt.

6.5.3 Percentage Aromatics extracted and % Purity of extract for system B-H -80% Dmso-20%W :

During liquid liquid extraction of aromatics under mass transfer conditions for fixed value of Vdi and Vci after steady state is reached, the values of Vdo and Vco also achieve constant values. The value of Vco is expected to be more than Vci because of transfer of aromatics from feed to extract phase and correspondingly value of Vdo is expected to be less than Vdi because of transfer of aromatics from feed to extract phase. Hence, all these observations for three stage operations have been recorded along with average value of Vd and Vc.. Corresponding amount of aromatics extracted and and %AE , %PE value for every stage are recorded against average value of Vc and Vd i.e.Vcavg. and Vd avg.

As could be seen from first set of observation-(3.1) from Table-27, during stage 1, for Vc avg. =0.62 cm/min and Vdavg= 0.207 cm/min, for fixed value of S/F=4.187 wt., the value of % aromatics extracted is 46.52% and % purity extract being 93.149 %.

During stage 2 observation-(3.2),raffinate coming from column No1 with Vdo=0.185 cm/ min becomes feed Vdi for column 2. Additional benzene which get extracted in column 2 is 0.47 gm/ min. Individual value of % aromatic extracted for stage 2 is 50%. Cumulative value of aromatic extracted after stage 2 is 73.26% and purity of extract being 92.14%.

During stage 3 observation-(3.3),raffinate phase coming from column No2 with Vdo=0.154 cm/ min becomes feed Vdi for column 3. Additional benzene which get extracted in column 3 is 0.225 gm/ min. Individual value of % aromatic extracted for stage 3 is 48%. Cumulative value of benzene extracted after stage 3 is 86.10% and purity

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of extract being 93.6%. Thus from above discussion it is crystal clear that by performing stage wise operation, value of % aromatic extracted can be increased from 46.52% to 86.095% and purity of extract is expected to be of the order of 93.64%

Further, as could be seen from second set of observation-(4.1) from Table-27, during stage 1, for Vc avg. =0.527 cm/min and Vdavg= 0.261 cm/min, for fixed value of S/F=2.814 wt. the value of % aromatics extracted is 29.726% and % purity extract being 92.138 %.

During stage 2 observation-(4.2), raffinate phase coming out from column No1 with Vdo=0.227 cm/ min becomes feed Vdi for column 2. Additional benzene which get extracted in column 2 is 0.477 gm/ min. Individual value of % aromatic extracted for stage 2 is 30%. Cumulative value of aromatic extracted after stage 2 is 50.808% and% purity of extract being 91.14%.

During stage 3 observation-(4.3) ,raffinate coming out from column No2 with Vdo=0.169 cm/ min becomes feed Vdi for column 3 . Additional benzene which get extracted in column 3 is 0.390 gm/ min. Individual value of % aromatic extracted for stage 3 is 35%. Cumulative value of aromatic extracted after stage 3 is 68.025% and % purity of extract being 92.63%. Thus from above discussion it is crystal clear that by performing stage wise operation , value of % aromatic extracted can be increased from 29.726% to 68.025% and % purity of extract is expected to be of the order of 92.63%

Thus from above two observations ,Vcavg.=0.62 cm/min and Vd avg. =0.207 cm/min. for S/F= 4.187 wt., the value of % aromatic extracted is 86.09% with % purity of extract being 92.63% As S/F ratio is decreased to 3.202(by wt.) for Vc avg. =0.476 cm/min and Vd avg. 0.211 cm/min, the value of % AE extracted decreases to 68.02 corresponding purity being 92.63% .This confirms that as S/F ratio decreases under otherwise identical conditions, the value of % AE decreases.

For other two values of S/F by wt. namely 0.669 (Vc=0.531 cm/min and Vd avg. 1.097 cm/min ) and 3.597 (Vc=0.526 cm/min and Vd avg.= 0.208 cm/min) observation No the values of % aromatic extracted are 33.88% and 79.62 % respectively. The remaining two sets of observations can also be analyzed in a similar manner, as adopted for earlier two sets of observations.

Thus by performing stage wise operation, the value of % aromatic extracted of the order of 86% can be obtained in a packed column with purity of extract of the order of 93.5 % for system B-H-80%Dmso-20%W under following sets of operating conditions:-

(i)Temperature of column for all three columns=40 0C

(ii)Vcavg.= 13.13 gm/min=0.62 cm/min

(iii)Vdavg.= 3.136 gm/min=0.207 cm/min

(iv) S/F=4.187 by wt.

# 6.5.4 Over view on mass transfer aspects during liquid –liquid extraction of aromatics carried out in multistage manner :-

It has been already discussed during previous subsections that resistance to mass transfer during liquid –liquid extraction of aromatics lies in both the films- dispersed phase film and continuous phase film.

Mass transfer occurs of solute (Benzene) in feed to continuous phase (solvent). Thus solute from bulk phase i.e. dispersed phase gets transferred to dispersed phase film. From dispersed phase film solute gets transferred to the interphase of dispersed phase film and continuous phase film. From continuous phase film, solute gets transferred to bulk phase- solvent phase. In summary, solute (Benzene) gets transferred from bulk phase – feed- to continuous phase- solvent during liquid - liquid extraction in packed column. This mechanism of mass transfer is based on two film theory.

During stage wise operation i.e. during stage-II in a packed column operation of liquid- liquid extraction, raffinate phase from column in stage –I becomes feed for column -2 –stage-II. where in composition of benzene in the feed decreases and that of hexane in the feed increases. During further stage wise operation, i.e. during stage –III operation of liquid –liquid extraction of aromatics, raffinate phase coming out of stage-II becomes feed for column- 3- stage-III where in composition of benzene in the feed for stage-III further decreases in comparison to stage-II and that of hexane in the feed for stage-III further increases in comparison to stage II. It is observed that amount of benzene transferred during stage-II gets decreased than that transferred during stage –I Mass transfer mechanism either during stage-I or stage-II being the same, one has to analyze why benzene amount in stage –II has decreased . Further , rate of mass transfer of benzene from dispersed phase for stage –I is higher than that for stage –II

Thus, resistance to mass transfer in dispersed phase film in stage-II is more than that in stage-I. Continuous phase film in stage-I of column -1 or stage-II of column -2 or stage-III of column -3 is expected to remain same as fresh solvent has been used in all the three stages.

Hence, the value of overall dispersed phase side mass transfer coefficient (Kod.a) is expected to decrease in stage –II in comparison to stage-I. It will get decreased further in stage-III in comparison to stage –I and stage-II. However, the value of over all continuous phase side mass transfer coefficient (Koc.a) is expected to be the same. , as continuous phase is 100% pure solvent in all three stages. However, dispersed phase feed composition of benzene in feed in stage-I ,II and III gets decreased continuously and composition of hexane in stage-I ,II and III gets increased continuously. It appears that when composition of component hexane in feed increases and that component benzene in the feed decreases , resistance to mass transfer gets increased from stage-I to stage-III .As aresult rate of mass transfer of benzene decreases under other wise identical condition from stage-I to stage-III when one operates a set of column in stage wise manner.

Thus for any observation for system B-H-80%Dmf-20%W or B-H-80%Dmso-20%W, the value of Kod .a for stage –I is expected to be highest and for stage-III, it is expected to be lowest. The corresponding value of Koc.a for all three stages is expected to remain same.

Two typical illustrations one for system B-H-80%Dmf-20%W and second for B-H-80%Dmso-20%W have been considered. The relevant details are as under:-

# (I) Analysis for system B-H-80%Dmf-20%W at 30 °C:-

For stage-I the column was operated under following sets of conditions:-

Vcavg = 0.595 cm/min

Vdavg =0.132 cm/min

Vdi =0.165 cm/min

Vci=0.56 cm/min with feed composition 50 %Benzene and 50% Hexane.

S/F by wt. =5.41

Under these conditions benzene extracted was 0.810 gm/ min. and % AE value is 62.73% and corresponding % PE being 73.4 % From the above data raffinate phase flow rate (Vdo) is 0.098 cm/min and raffinate phase composition being the following  $X_{BR}=0.3$ ,  $X_{HR}=0.65$  and  $X_{SR}=0.05$ 

For stage-II as has been already pointed out raffinate phase (Vdo) from column -1 becomes feed (Vdi) for column-2

Hence for stage-II column was operated under following sets of conditions:-

Vdi = 0.1 cm/min

Vci=0.443 cm/min.

S/F by wt. =5.41

Under these conditions corresponding values of Vdo and Vco are 0.079 and 0.471 cm/min respectively. Under these conditions benzene extracted is 0.301 gm/min. and % AE value is 65% and corresponding % PE being 83.5 %. Under these conditions cumulative value of %AE at the end of second stage comes to 87.26 %

Thus as expected, during stage-II, the values of composition of feed are as under:-

X<sub>BF</sub>=30%,X<sub>HF</sub>=65% and X<sub>SF</sub>=5%

The corresponding values of feed composition in stage -I are the following:-

X<sub>BF</sub>=50%, X<sub>HF</sub>=50% and X<sub>SF</sub>=0%

Thus as expected, the value of benzene in feed during stage-II decreases in comparision to value of benzene in stage-I and hexane in feed increases in comparision to stage-I. The value of amount of benzene extracted in stage-I and stage –II are 0.81 gm/min and 0.30 gm/min

Thus the value of rate of extraction of aromatics in stage –II is lower than that of stage –I. Thus resistance to mass transfer during stage-II is more than that during stage-I.

For stage-III as has been already pointed out that raffinate phase (Vdo) from column -2 becomes feed (Vdi) for column-3

Hence for stage-III column was operated under following sets of conditions:-

Vdi =0.081 cm/min

Vci=0.357 cm/min.

S/F by wt. =5.41

Under these conditions corresponding values of Vdo and Vco are 0.072 and 0.371 cm/min respectively. Under these conditions benzene extracted is 0.102 gm/min. and % AE value for this third stage is 64% and corresponding % PE being 85.6 %. Under these conditions cumulative value of %AE at the end of stage-III comes to 95.25 %

Thus as expected the value of benzene in feed, during stage-III decreases in comparision to stage-II and hexane in feed, increases in stage-II. The value of amount of benzene extracted in stage-II and stage –III are 0.3 gm/min and 0.102 gm/min

Thus the value of rate of extraction of aromatics in stage –III is lower than that of stage –II. Thus resistance to mass transfer during stage-III is more than that during stage-II.

Since for stage –I or stage-II or stage-II solvent phase (Vci) happens to be 80% Dmf +20% W solvent for all three stages, resistance to mass transfer in continuous phase film is expected to remain same for all the three stages. However resistance to mass transfer in dispersed phase film increases continuously from stage-I to stage-III. Thus when liquid-liquid extraction of aromatics in a packed column is performed in multistage manner using three stages, mass transfer of solute from dispersed phase to continuous phase occurs in same manner as in single stage operation.

### (II) Analysis for system B-H-80%Dmso-20%W at 40 °C:-

For stage-I the column was operated under following sets of conditions:-

Vcavg.= 0.62cm/min

Vdavg =0.207 cm/min

The feed flow rate Vdi is 0.228 cm/min and solvent phase –(continuous phase)flow rate Vci is 0.609 cm/min with feed composition 50 %Benzene and 50% Hexane.

S/F by wt. =4.187

Under these conditions benzene extracted was 0.817 gm/ min. and % AE value is 46.52% and corresponding % PE being 93.149 % From the above data raffinate phase flow rate (Vdo) is 0.185 cm/min and raffinate phase composition being the following  $X_{BR}=34\%, X_{HR}=64\%$  and  $X_{SR}=2\%$ 

For stage-II as has been already pointed out raffinate phase (Vdo) from column -I becomes feed (Vdi) for column-II

Hence for stage-II column was operated under following sets of conditions:-

Vdi =0.185 cm/min

Vci=0.582 cm/min.

S/F by wt. =4.187

Under these conditions corresponding values of Vdo and Vco are 0.154 cm/min and 0.604 cm/min respectively. Under these conditions benzene extracted is 0.47 gm/ min. and % AE value is 50% and corresponding % PE being 92.14 % Under these conditions, cumulative value of %AE at the end of second stage comes to 73.26 %

Thus as expected, during stage-II, the values of composition of feed are as under:-

X<sub>BF</sub>=34%, X<sub>HF</sub>=64% and X<sub>SF</sub>=2%

The corresponding values of feed composition in stage -I are the following:-

 $X_{BF}$ =50%,  $X_{HF}$ =50% and  $X_{SF}$ =0%

Thus as expected the value of benzene in feed, during stage-II decreases in comparision to value of benzene in stage-I and hexane in feed increases in comparision to stage-I. The value of amount of benzene extracted in stage-I and stage -II are 0.817 gm/min and 0.47 gm/min

Thus, the value of rate of extraction of aromatics in stage –II is lower than that of stage –I. Thus resistance to mass transfer during stage-II is more than that during stage-I.

For stage-III as has been already pointed out raffinate phase (Vdo) from column -II becomes feed (Vdi) for column-III

Hence for stage-III column was operated under following sets of conditions:-

Vdi =0.154. cm/min

Vci=0.483 cm/min.

S/F by wt. =4.187

Under these conditions corresponding values of Vdo and Vco are 0.138 and 0.494 cm/min respectively. Under these conditions benzene extracted is 0.225 gm/min. and % AE value for this third stage is 48% and corresponding % PE being 93 %. Under these conditions cumulative value of %AE at the end of stage-III comes to 86.095 %

Thus as expected the value of benzene in feed, during stage-III decreases in comparision to stage-II and hexane in feed increases in stage-II. The value of amount of benzene extracted in stage-II and stage –III are 0.47 gm/min and 0.225 gm/min

Thus the value of rate of extraction of aromatics in stage –III is lower than that of stage-II. Thus resistance to mass transfer during stage-III is more than that during stage-II.

Since for stage –I or stage-II or stage-III solvent phase (Vci) happens to be 80% Dmso +20% W solvent for all three stages , resistance to mass transfer in continuous phase film is expected to remain same for all the three stages. However resistance to mass transfer in dispersed phase film increases continuously from stage-I to stage- III. Thus when liquid- liquid extraction of aromatics in a packed column is performed in multistage manner using three stages , mass transfer of solute from dispersed phase to continuous phase occurs in same manner as in single stage operation.

6.5.5 Performance of solvents-Dmf and Dmso during multistage operation:-

It has been already pointed out that solvents Dimethyl formamide (Dmf) and dimethyl sulfoxide(Dmso) being very costly, only limited observations and data have been taken during multistage operation of liquid –liquid extraction of aromatics. Results obtained for stage wise operations for solvent Dmf have been already reported in Table-25 and that for Dmso have been reported in Table-28.Based on these two Tables a comparison Table-29 has been prepared to evaluate performance of these solvents with respect to % aromatic extracted (%AE) and % purity of extract (%PE) during liquid-liquid extraction of aromatics in stagewise manner.

Table-29

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Sr.No.	Solvent	.Vcavg gm/min	Vdavg gm/min	Vcavg cm/min	Vdavg cm/min	S/Fwt	%AEI	%PEI	%AEII	%PEII	%AEIII	%PEIII
<b></b>	Dmf	8.479	1.992	0.46	0.132	4.252	51	83.4	77.741	88.4	89.09	92.6
6	Dmso	13.130	3.136	0.620	0.207	4.187	46.520	93.149	73.260	92.140	86.095	93.640
	Dmf	16.940	4.952	0.900	0.315	3.421	39.000	83.300	·63.347	89.300	77.642	92.700
4	Dmso	12.716	3.873	0.600	0.255	3.283	41.5	95.231	64.9	96.15	79.62	96.55

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For solvent Dmf under operating conditions of Vcavg.=0.46 cm/min, Vd=0.132 cm/min and S/F= 4.252 by wt., the value of %AE increases from 51% in stage –I to 77.7 % in stage-II and finally to 89.09 % in stage-III .Under otherwise similar conditions of Vc= 0.62 cm/min , Vd= 0.207 cm/min and S/F=4.18 by wt. for solvent Dmso the value of %AE increases from 46.52% in stage –I to 73.26% in stage-II and finally to 86.09 % in stage-III.

Thus, performance of both these solvents appear to be comparable for S/F ratio 4.2 by wt.. For both these solvents values of % AE are in the range of 86% to 89 %. Percentag purity of extract (%PE) for both the solvents under considerations appear to be also comparable as could be seen from the values 92.6 % for Dmf and 93.6 % for Dmso from observation -1 and 2.

For an additional observation No.-3 for solvent Dmf under operating conditions of Vcavg.=0.9 cm/min, Vdavg=0.315 cm/min and S/F= 3. 42 by wt., which is lower than previous set of observation for solvent Dmf where in S/F was 4.2 by wt, the values of %AE increases from 39% in stage –I to 63.3 % in stage-II and finally to 77.6 % in stage-III. The corresponding value of %AE in stage-I and stage-III for S/F=4.25 by wt. are 51 %, 77.7% and 89% respectively.

For solvent Dmso from observation No. -4 under otherwise similar conditions of Vc= 0.6 cm/min , Vd= 0.255 cm/min and S/F=3.2 by wt. which is lower than previous sets of observation for solvent Dmso where in S/F ratio was 4.18 by wt., the value of % AE increases from 41.5% in stage –I to 64.9 % in stage-II and finally to 79.62 % in stage-III. The corresponding value of %AE in stage-I , stage-II and stage-III for S/F=4.18 by wt. are 46.5 % , 73.26% and 86.09% respectively. Thus from these observation -3 and observation-4 from Table 29 , it can be concluded that performance of both the solvents appear to be comparable also for S/F ratio 3.3 by wt.. For both these solvents values of % AE are in the range of 77.5% to 79.5 %.

The value of %PE for both these solvents are also comparable. However, % purity of extract for solvent Dmso happen to be the highest being 96.5%. Hence, solvent Dmso is expected to give more purity than solvent Dmf.

In summary, it can be concluded that performance for both these solvents with respect to %AE value and %PE value appear to be comparable under other wise comparable operating conditions. Hence S/F ratio of the order of 5 to 6 by wt. having Vcavg. Value in the range of 0.6 to 0.8 cm/min and Vd value in the range of 0.15 to 0.2

cm/min. when one carries liquid – liquid extraction in stage wise manner up to 3 stages, the value of % AE extracted appear to be of the order of 95% and % PE is expected to be of the order of 96%.

Hence it appears that instead of using higher S/F ratio value which is expected to involve more quantity of solvent, using lower solvent ratio and carrying out operation in stage wise manner is expected to be a better option. However, using higher value of number of stages like V stages to VIII stages is likely to involve substantial increase in fixed cost and using higher S/F ratio is likely to increase operating cost . Hence balance of these two factors has to be made in industrial practice. Based on data obtained in this investigation S/F of the order of 5 to 6 appears to be the optimum value.

Similarly, to carry out the liquid-liquid extraction of aromatics in stage wise manner, the number of stages of the order of 3 to 4 appears to be the optimum value.

## 6.6.0 LIQUID – LIQUID EXTRECTION OF AROMATICS IN A BIGGER DIAMETER COLUMN:

Mass transfer data for Liquid- Liquid extraction of aromatics obtained on Laboratory Scale can be utilized conveniently on Industrial Scale only if it is free from End effects and as well as Wall effects.

Scale up of any Laboratory Scale data is feasible only if above requirements are fulfilled.

Hence after obtaining exhaustive Mass transfer data in a packed column of 5 cm. diameter, to enhance its utility on industrial scale, "Liquid-Liquid Extraction of Aromatics" was also performed in a packed column of 15 cms. diameter. However in view of large requirement of solvent quantity, only some selected experimental runs were taken because of the fact that the newer solvents are costly. Hence due to financial constraints, selected minimum experimental observations required for comparison purpose were taken in a 15 cm. diameter column. The relevant discussion constitutes comparison of Mass transfer data in 5 cm. diameter column v/s. 15 cm. diameter column.

#### 6.6.1 Derived tables for liquid-liquid extraction of aromatics :

Experimental observation recorded in Tables - XV to XVIII in Chapter-4: Experimental could be utilized conveniently to prepare derived tables -30 to 36 which can be utilized conveniently for discussion purpose.

The relevant details for liquid- liquid extraction of aromatics using mixed solvent- Dmf + W are as under:-

Table 30: Observations recorded for Packed column operation under different flow rate conditions for L-L extraction of aromatics in a column of 15 cm. diameter System: B-H-80%Dmf -20%W at 30  $^{\circ}$ C

Table 31:Values of %AE and %PE under different flow rate conditions for L-L extraction of aromatics in a column of 15 cm. diameter. System: B-H-80%Dmf -20%W at 30  $^{\circ}$ C

Table 32: Comparison of %AE and %PE under different Sets of conditions. System: B-H-80%Dmf -20%W at 30  $^{\circ}$ C

Table 33 : Observations recorded for Packed column operation under different flow rate conditions for L-L extraction of aromatics in a column of 15 cm. diameter. System: B-H-80%Dmso -20%W at 40  $^{\circ}$ C

Table 34: Values of %AE and %PE under different flow rate conditions for Liquid-Liquid extraction of aromatics in a column of 15 cm. diameter. System: B-H-80%Dmso -20%W at 40  $^{0}$ C

Table 35 : Comparison of %Ae and %Pe under different sets of conditions. System: B-H-80%Dmso -20%W at 40  $^{\circ}C$ .

Table 36 contains comparison of values of  $A_E$  and  $P_E$  obtained in this investigation for two mixed solvents- (Dmf + W) & (Dmso + W)

6.6.2 Discussion for liquid- liquid extraction of aromatics using mixed solvent- Dmf + W.

After critical analysis of Table 31, the following are important conclusions:-

Table-30

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Observations recorded for Packed column operations under different flow rate conditions

for L-L extraction of aromatics in a column of 15 cm. diameter.

system: **B-H-80%Dmf -20%W** at 30 <sup>0</sup>C

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Sr.No.	Vdo cm <sup>3</sup> /min	Vdi cm <sup>3</sup> /min	Vco cm <sup>3</sup> /min	Vci cm <sup>3</sup> /min	Vcavg cm <sup>3</sup> /min	Vdavg cm <sup>3</sup> /min	Vdo cm/min	Vdi cm/min	Vco cm/min	Vci cm/min	Vcavg cm/min	Vdavg cm/min
74	16.21	27.15	103.8	92.41	98.11	21.68	0.108	0.181	0.692	0.616	0.655	0.145
р	32.710	39.150	83.720	72.750	78.230	35.930	0.218	0.261	0.558	0.485	0.522	0.239
ŝ	19.950	23.120	77.550	72.300	74.920	21.53	0.133	0.154	0.517	0.482	0.501	0.143
4	36.720	47.120	128.400	117,3	122.850	41.920	0.245	0.314	0.856	0.782	0.819	0.279

Table-31

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Values of  $\%A_E$  and  $\%P_E$  under different flow rate conditions for L-L extraction of aromatics in a column of 15 cm. diameter.

				System:	B-H-80%	%Dmf -2	0% W at	30 0C				
Sr.No.	Vcavg	Vdavg	Vdo	Vdi	Vco	Vci	Vcavg	Vdavg	S/F	$%A_{\rm E}$	$%P_{E}$	
	cm <sup>3</sup> /min	cim <sup>3</sup> /min	cm/min	cm/min	cm/min	cm/min	cm/min	cm/min	wt. inlet	•		
	98.11	21.68 -	0.108	0.181	0.692	0.616	0.655	0.145	5.36	67.11	78.14	
7	78.230	35.930	0.218	0.261	0.558	0.485	0.522	0.239	2.590	50.500	88.350	•
ñ	74.920	21.53	0.133	0.154	0.517	0.482	0.501	0.143	3.85	48.81	89.51	
4	122.850	41.920	0.245	0.314	0.856	0.782	0.819	0.279	3.480	41.120	89.450	•

(i) Under operating conditions of a fixed value of Vc = 0.522 cm./ min., Vd = 0.239 cm/ min. and S/F = 2.59 by wt, the values of % AE and % PE obtained in a column of 15 cm. diameter are 50.5% and 88.3% respectively. For this case (Vc + Vd) value is 0.76 cm./ min.

Further under operating conditions of a fixed values of Vc = 0.655 cm/min., Vd = 0.145 cm/min. and S/F = 5.36 by weight, the values of % AE and % PE obtained in a column of 15 cm. diameter are 67.11% and 78.14% respectively. For this case (Vc + Vd) value is 0.8 cm/min.

Thus under otherwise a constant value of (Vc + Vd) of the order of 0.78 cm/min., as S/F ratio increases from 2.59 to 5.36, the values of  $%A_E$  gets increased from 50.5 % to 67.11%.

(ii) In general as the values of S/F ratio by weight increase from 3.48, 3.85 to 5.36, the corresponding values of percentage aromatic extracted (%AE) increase from 41.12%, 48.81% to 67.11% respectively.

(iii) Under otherwise constant value of Vd = 0.145 cm/min, as Vc is increased from 0.501 cm/min. to 0.655 cm./min., the value of % aromatic extracted (%AE) increases from 48.81% to 67.11%.

(iv) For first case under otherwise constant value of Vc = 0.522 cm/ min. Vd=0.239 and S/F ratio=2.59 by wt., the value of % aromatic extracted is 50.5%. However for the Second case having Vc=0.501cm/min and Vd=0.143 cm/min wherein S/F = 3.85, the value of %AE is 48.81%. Under these conditions since S/F ratio is higher, values of %AE should have been higher than which is obtained at S/F = 2.59 i.e. higher than %A<sub>E</sub> = 50.5%. However on the contrary it is 48.81% i.e. lower than % A<sub>E</sub>=50.5%.

This behaviour can be explained as below : Since value of dispersed phase flow rate for higher S/F ratio (3.85) is 0.143 cm/ min. and the value of dispersed phase flow rate for lower S/F ratio (2.59) is 0.239 cm/ min., the values of dispersed phase hold up are expected to be higher for the first case wherein value of Vd is higher.

Since dispersed phase flow rate proportionally increases value of effective interfacial area and also increases rate of mass transfer, the value of % aromatics

extracted are also expected to be higher. Hence even though S/F ratio is on lower side for first case, the value of % AE is on higher side.

After critical analysis of Table- 32, where in data has been compared for 5cm and 15 cm diameter columns, the following are the important conclusions :-

(i) For a fixed value of Vc = 0.655 cm./ min., Vd = 0.145 cm/ min. and S/F = 5.36 by wt, the value of % AE obtained in a column of 15 cm. dia is 67.11%. Further for a fixed values of Vc = 0.595 cm/ min. & Vd = 0.132 cm/ min. and S/F = 5.41 by weight, the value of % AE obtained in a 5 cm. dia is 62.7%. The values of %AE are comparable for both the cases.

(ii) For a fixed value of Vc = 0.522 cm./ min., Vd = 0.239 cm/ min. and S/F = 2.59 by wt, the value of % AE obtained in a column of 15 cm. dia is 50.5%. Further for a fixed values of Vc = 0.58 cm/ min. Vd = 0.27 cm/ min., and S/F = 2.62 by weight , the values of % AE % obtained in a 5 cm. dia is 46%. The values of %AE are comparable for both the cases.

.(iii) For a fixed value of Vc = 0.501 cm./ min., Vd = 0.143 cm/ min. and S/F = 3.85, the value of % AE obtained in a column of 15 cm. dia is 48.81%.Further for a fixed values of Vc = 0.455 cm/ min., Vd = 0.132 cm/ min. and S/F = 4.25% by weight, the values of % AE obtained in a 5 cm. dia is 51%. The values of %AE are comparable for both the cases.

(iv) For a fixed value of Vc = 0.819 cm./ min., Vd = 0.279 cm/ min. and S/F = 3.48, the value of % AE obtained in a column of 15 cm. dia is 41.12%. Further for a fixed values of Vc = 0.91 cm/ min., Vd = 0.315 cm/ min. and S/F = 3.42 by weight the values of % AE obtained in a 5 cm. dia is 39%. The values of %AE are comparable for both the cases.

As could be seen from above four observations [No.(i) to (iv)] wherein data of % AE values have been compared for the two cases i.e. Case No. I: column dia. 15 and Case No. II: dia 5 cms. For all the four observations undr otherwise comparable conditions, the values of %AE are comparable for Case I and Case II.

## Table-32

# Comparison of %AE and %PE under different Sets of conditions system: B-H-80%Dmf-20%W at 30 <sup>0</sup>C

Sr.No.	Column dia.	Vcavg	Vdavg	S/F wt.	%A <sub>E</sub>	%P <sub>E</sub>
	ст	cm/min	cm/min	inlet		• ,
1	5 cm	0.594	0.131	5.411	62.73	73.4
	15 cm	0.655	0.145	5.36	67.11	78.14
2	5 cm	0.580	0.270	2.62	46.00	81.81
	15 cm	0.522	0.239	2.59	50.50	88.35
3	5 cm	0.46	0.132	4.25	51.00	83.44
	15 cm	0.501	0.143	3.85	48.81	89.51
4	5 cm	0.900	0.315	3.42	39.00	83.33
	15 cm	0.819	0.279	3.48	41.12	89.45

Thus for these selected runs wherein operating conditions in both the columns are comparable, it is interesting to observe that the values of % AE obtained are comparable.

Hence, it can be concluded that all the mass transfer data obtained in this investigation for mixed solvent- Dmf + W in a 5 cm. dia column in free of End effects and Wall effects.

Thus, since data obtained in 5 cm. dia column compares very well with data obtained in 15 cm. dia column, Exhaustive Experimental Data obtained in a 5 cm. diameter column can be utilized conveniently in Industrial Practice.

6.6.3 Discussion for liquid-liquid extraction of aromatics using mixed solvents Dmso + W.

After critical analysis of Table 34, the following are important conclusions:-

(i) Under operating conditions of a fixed values of Vc = 0.474 cm./min. Vd = 0.235 cm/min. and S/F = 2.59 by wt. the values of % AE and % PE obtained in a column of 15 cm. dia are 34.19% and 94.88 % respectively. For this case (Vc + Vd) value is 0.71 cm./min.

Further under operating conditions of a fixed values of Vc = 0.558 cm/min., Vd = 0.186 cm/min. and S/F = 3:88 by weight the values of % AE & % PE obtained in a column of 15 cm. dia are 49.28% and 94.64% respectively. For this case (Vc + Vd) value is 0.744 cm./min.

Thus under otherwise a constant value of (Vc + Vd) of the order of 0.73 cm/ min. as S/F ratio increases from 2.59 to 3.88., the values of  $%A_E$  gets increase from 43.19% to 49.28%.

(ii) In general as the values of S/F ratio by weight increases from 3.63, 2.6, 3.04 to 3.88, the corresponding values of percentage aromatic extracted (%AE) increase from 14.15%, 34.19%, 45.0% to 49.3% respectively. Table-33

Observations recorded for Packed column operation under different flow rate conditions for L-L extraction of aromatics in a column of 15 cm. diameter.

System: B-H-80%Dmso -20%W at 40 <sup>o</sup>C

87.62 180.5 1.159 1.254 0.605 (	82.5 87.62 180.5 1.159 1.254 0.605 (	90.75 82.5 87.62 180.5 1.159 1.254 0.605 (
99.350 42.330 0.247 0.313 0.676 0.643	96.520 99.350 42.330 0.247 0.313 0.676 0.643	101.470 96.520 99.350 42.330 0.247 0.313 0.676 0.643
99.350 42.330 0.247 0.313 (	96.520 99.350 42.330 0.247 0.313 (	101.470 96.520 99.350 42.330 0.247 0.313 (
99.350 42.330 0.247 83 740 27 02 0.166	96.520 99.350 42.330 0.247 82.240 83.740 27.02 0.166	101.470 96.520 99.350 42.330 0.247 85.050 82.240 27.02 0.166
87.62 180.5 99.350 42.330 83.740 27.92	82.55 87.62 180.5 96.520 99.350 42.330 82.210 83.740 27.92	90.75         82.5         87.62         180.5           101.470         96.520         99.350         42.330           85.050         82.210         83.740         27.92
87.62 99.350 83.740	82.55 87.62 96.520 99.350 82.210 83.740	90.75         82.5         87.62           101.470         96.520         99.350           85.050         82.210         83.740
	82.5 96.520 82.210	90.75 82.5 101.470 96.520 85.050 82.210
188.11 90.75 47.030 101.470 30.780 85.050	188.11 47.030 30.780	
173.91         188.11         90.75           37.130         47.030         101.470           24.970         30.780         85.050	173.91 188.11 37.130 47.030 24.970 30.780	173.91 37.130 24.970

Table-34 Values of %AE and %PE under different flow rate conditions

		H		racuon ol	aromatic	s in a col	r 10 umn	o cm. di	ameter.		
		•		System:	B-H-80%	<b>6Dmso -2</b>	0%W at	: 40 <sup>0</sup> C			
Sr.No.	Vcavg	Vdavg	Vdo	Vdi	Vco	Vci	Vcavg	Vdavg	S/F	%AE	%PE
	cm <sup>3</sup> /min	cm <sup>3</sup> /min	cm/min	cm/min	cm/min	cm/min	cm/min	cm/min	wt. inlet		
`en.d	87.62	180.5	1.159	1.254	0.605	0.55	0.584	1.201	0.632	14.15	066.76
2	99.350	42.330	0.247	0.313	0.676	0.643	0.662	0.282	3.042	45.080	96.880
<del></del>	83.740	27.92	0.166	0.205	0.567	0.548	0.558	0.186	3.887	49.28	94.640
4	71.110	35.250	0.204	0.264	0.487	0.458	0.474	0.235	2.597	34.190	94.880

(iii) Under otherwise constant value of Vd in the range of 0.24 cm/min. to 0.28 cm/min. as Vc is increased from 0.474 cm/min. to 0.66 cm./min., the value of % aromatic extracted increases from 34.2% to 45.0%.

(iv) Under otherwise constant value of Vc in the range of 0.56cm/ min. to 0.58 cm/min, as Vd decreases from 1.2 cm/min to 0.186 cm/min ,the value of %AE increases from 14.15% to 49.3%

As Vd decreases ,the value of S/F ratio increases. As a result, the value of S/F ratio in the present case increase from 0.63 to 3.88 and consequently, the value %AE increases from 14.15 to 49.3%.

After critical analysis of Table- 35 where in data has been compared for 5cm and 15 cm diameter columns, the following are the important conclusions :-

(i) For a fixed value of Vc = 0.584 cm./ min. Vd = 1.2 cm/ min. and S/F = 0.632, by wt. ,the values of % AE obtained in a column of 15 cm. dia is 14.15%. Further for a fixed value of Vc = 0.531 cm/ min., Vd = 1.09 cm/ min. and S/F = 0.669 by weight, the value of % AE obtained in a 5 cm. dia is 11.64% The values of %AE are comparable for both the cases.

(ii) For a fixed value of Vc = 0.662 cm./ min. Vd = 0.282 cm/ min. and S/F = 3.04, the value of % AE obtained in a column of 15 cm. dia is 45.08%. Further for a fixed values of Vc = 0.6 cm/ min., Vd = 0.255 cm/ min. and S/F = 3.28 by weight, the values of % AE obtained in a 5 cm. dia is 41.5%. The values of %AE are comparable for both the cases.

.(iii) For a fixed value of Vc = 0.558 cm./ min. Vd = 0.186 cm/ min. and S/F = 3.88, the values of % AE obtained in a column of 15 cm. dia is 49.28%. Further for a fixed values of Vc = 0.62 cm/ min. Vd = 0.207 cm/ min. and S/F = 4.18% by weight, the value of % AE obtained in a 5 cm. dia is 46.52%.. The values of %AE are comparable for both the cases

(iv) For a fixed value of Vc = 0.474 cm./ min., Vd = 0.235 cm/ min. and S/F = 2.59 the value of % AE obtained in a column of 15 cm. dia is 34.19% Further for a fixed values of Vc = 0.527 cm/ min., Vd = 0.261 cm/ min. and S/F = 2.81 by weight,

Table-35 Comparison of %AE and %PE under different sets of conditions

System: B-H-80%Dmso -20%W at 40 <sup>o</sup>C Sr.No

$\mathbf{\%P_E}$	
%AE	•
S/F	wt.
Vdavg	
Vcavg	
Column dia	
0	

	CE	cm/min	cim/min	inlet			ſ
• ••							
-4	15 cm	0.584	1.201	0.669 0.632	11.64 14.15	97.990	
7	5 cm 15 cm	0.600 0.662	0.255 0.282	3.283 3.042	41.500 45.080	95.231 96.880	•
m	5 cm 15 cm	0.620 0.558	0.207 0.186	4.187 3.887	46.52 49.28	93.149 94.640	
4	5 cm 15 cm	0.527 0.474	0.261 <sub>.</sub> 0.235	2.814 2.597	29.726 34.190	92.138 94.880	

the value of % AE obtained in a 5 cm. dia is 29.72%. The values of %AE are comparable for both the cases As could be seen from above four observations [No.(i) to (iv)] wherein data of % AE values has been compared for the two cases i.e. Case No. I column dia. 15 and Case No. II dia 5 cms. For these four selected experimental observations wherein operating conditions in both the column are comparable, it is interesting to observe that the values of % AE obtained are also comparable. Hence, it can be concluded that all the Mass transfer data obtained in this investigation for mixed solvent-(Dmso + W) in a 5 cm. dia column is free of End effects and wall effects. Thus, since data obtained in 5 cm. dia column compares very well with data obtained in 15 cm. dia column can be utilized conveniently in Industrial Practice.

## 6.7.0 OVER VIEW OF PERFORMANCE OF MIXED SOLVENTS AND RECOMMENDED OPERATING CONDITIONS:-

Results obtained with respect to % aromatic extracted (%AE) and purity of extract (%PE) has been compared in Table -36 for both the solvents under otherwise comparable operating conditions for the two categories as under:

Category-(I) Masstransfer data on liquid-liquid extraction of aromatics obtained in a 5 cms diameter column.

Category – (II) Masstransfer data on liquid-liquid extraction of aromatics obtained in a 15 cms diameter column.

For mixed solvent- (Dmf +W) under conditions of Vc =0.9 cms/ min, Vd=0.315 cms/ min, and S/F ratio of 3.42 by weight, when liquid liquid extraction of aromatics is performed in a stage wise manner (multistage operation) the values of (%AE) obtained in stage (I) and last stage – stage (IV) in a 5 cm diameter column are 39% and 77.64% respectively. Further for the same mixed solvent- (Dmf +W) under otherwise comparable conditions of Vc=0.819 cm/ min, Vd=0.279 cm/ min and S/F ratio of 3.48 by weight, when liquid liquid extraction of aromatics is performed in a stage wise manner (multistage operation), the values of %AE obtained in stage-(I) and the last stage i.e. stage-(IV) in a 15 cm diameter column are 41.12% and 79.76% (Projected value) respectively. The above comparison in the case of mixed solvent- (Dmf+W) indicates that results obtained in a 5 cm diameter column are free from the "End

No.         Solvent Column dia.         Vcavg         S/F         %A <sub>E1</sub> %A <sub>E1</sub> %A <sub>E1</sub> %A <sub>E11</sub> <th< th=""><th></th><th></th><th>Co. during</th><th>mparison Multista</th><th>l of %AE al ge Operati</th><th>on for Li</th><th>quid-Liq</th><th>uid Extr</th><th>action of</th><th>f Aroma</th><th>tics.</th><th>•</th><th></th></th<>			Co. during	mparison Multista	l of %AE al ge Operati	on for Li	quid-Liq	uid Extr	action of	f Aroma	tics.	•	
1         Dmf         5 cm         0.900         0.315         3.42         39         83.30         63.34         89.3         77.64         92.7           15 cm         0.819         0.279         3.480         41.12         89.450         65.46*         90.45*         79.76*         93.85*           2         Dmso         5 cm         0.622         0.207         4.18         46.52         93.15         73.26         92.14         86.09         93.64           15 cm         0.658         0.186         3.88         49.28         94.64         75.99*         93.65*         95.13*	.No.	Solvent	Column dia.	Vcavg	Vdavg	S/F wt.	%A <sub>EI</sub>	%P <sub>EI</sub>	%A <sub>EII</sub>	%P <sub>EII</sub>	%A <sub>EIII</sub>	%P <sub>EIII</sub>	
15 cm         0.819         0.279         3.480         41.12         89.450         65.46*         90.45*         79.76*         93.85*           2         Dmso         5 cm         0.622         0.207         4.18         46.52         93.15         73.26         92.14         86.09         93.64           15 cm         0.558         0.186         3.88         49.28         94.64         75.99*         93.63*         95.13*		Dmf	5 cm	0.900	0.315	3.42	66	<b>83.30</b>	63.34	89.3	77.64	92.7	
2 Dmso 5 cm 0.62 0.207 4.18 46.52 93.15 73.26 92.14 86.09 93.64 15 cm 0.558 0.186 3.88 49.28 94.64 76.99 93.63* 88.82* 95.13*		, <b>.</b>	15 cm	0.819	0.279	3.480	41.12	89.450	65.46*	90.45*	79.76*	93.85*	
2 Dmso 5 cm 0.62 0.207 4.18 46.52 93.15 73.26 92.14 86.09 93.64 15 cm 0.558 0.186 3.88 49.28 94.64 75.99 <sup>*</sup> 93.63 <sup>*</sup> 88.82 <sup>*</sup> 95.13 <sup>*</sup>	•	• •	•										
15 cm 0.558 0.186 3.88 49.28 94.64 75.99* 93.63* 88.82* 95.13*	7	Dmso	5 cm	0.62	0.207	4.18	46.52	93.15	73.26	92.14	86.09	93.64	
			15 cm	0.558	0.186	3.88	49.28	94.64	75.99*	93.63*	88.82*	95.13*	, )
							· • <u>.</u> • . • .	·			•		

•

uperation. and 10 cm dia. packed column, during single stage •

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Effects" and "Wall Effects" because of the fact that values of % AE for both the categories are comparable under otherwise comparable operating conditions.For mixed solvent- (Dmso +W) under conditions of Vc =0.62 cms/ min, Vd=0.207 cms/ min, and S/F ratio of 4.18 by weight, when liquid liquid extraction of aromatics is performed in a stage wise manner (multistage operation) the values of (%AE) obtained in stage (I) and last stage - stage (IV) in a 5 cm diameter column are 46.52% and 86.09% respectively.Further for the same mixed solvent- (Dmso +W) under otherwise comparable conditions of Vc=0.558 cm/ min, Vd=0.186 cm/ min and S/F ratio of 3.88 by weight, when liquid liquid extraction of aromatics is performed in a stage wise manner (multistage operation), the values of %AE obtained in stage-(I) and the last stage i.e. stage-(IV) in a 15 cm diameter column are 49.28% and 88.82% (Projected value) respectively. The above comparison also in the case of mixed solvent-(Dmso+W) indicates that results obtained in a 5 cm diameter column are free from the "End Effects" and "Wall Effects" because of the fact that values of % AE for both the categories are comparable under otherwise comparable operating conditions. Thus for the both newer solvents - (Dmf+W) & (Dmso+W) when S/F ratio is in the range of 3.5 to 4.0 by weight the values of (%AE) are expected to be in the range of 65% to 75%. By increasing S/F ratio range to 5to6 by wt., the values of % aromatics extracted (%AE) are expected to be in the range of 90%. Thus newer solvents- Dmf and Dmso appeared to be attractive for liquid liquid extraction of aromatics in comparison to conventional solvents DEG and TEG wherein liquid liquid extraction column has to be operated at 120 °C because of very low values of distribution coefficients at 30 °C for solvents-DEG/TEG.

The information available in the literature <sup>2,6</sup> regarding the values of Distribution coefficient (m) for solvents DEG and TEG and the values of Distribution coefficient(m) for solvents- Dmf and Dmso obtained in this investigation are listed below:-

Sr.	System	Temperature	"m" Value
(i)	B+Hept+92.29%+DEG+7.71%W	120 °C	0.125
(ii)	B+Hept+92.79%+TEG+7.21%W	120 ℃	0.175
(iii)	B+Hex+80%+Dmf+20%W	30 ℃	0.25
(iv)	B+Hex+90%+Dmso+10%W	30 ℃	0.35
The c	orresponding values of selectivity a	nd relevant detail	s for the above

mentioned four systems are critically analyzed as under:-

For a fixed temperature of 120 °C and for a fixed value of benzene in raffinate phase of the order of 25% by weight, the corresponding value of Benzene in extract phase for mixed solvent- (92.29% DEG+7.77%W) is 78% and under otherwise identical conditions, the corresponding value of Benzene in extract phase for mixed solvent -(92.79%TEG +7.21%W) is 70%. For a fixed temperature of only 30 °C and for a fixed value of benzene in raffinate phase of the order of 25% by weight, the corresponding values of benzene in extract phase for solvent-( 80% Dmf +20%W) and (90%Dmso + 10% W) are 95% and 92% respectively. From the above data it is crystal clear that the value of benzene in extract phase for newer solvent is of the order of 95% and the corresponding values of benzene in extract phase for conventional solvents-DEG and TEG is of the order of 70%-75% only. Thus separation load for conventional solvents is expected to be higher than that for newer solvents. Further, the values of Distributions coefficients (m) for newer solvents are approximately double than the values of distribution coefficients (m) for conventional solvents. Thus rates of liquidliquid extraction of aromatics are expected to be substantially higher for newer solvents. Further to have reasonable values of Distribution coefficient (m), the temperature of extraction for conventional solvents is of the order of 120 °C . By chance this temperature of extraction is reduced to 30 °C, the value of distribution coefficient(m) is expected to be very low for conventional solvents- DEG and TEG wherein to carry out liquid liquid extraction is probably not feasible. However for solvents-Dmf/Dmso liquid liquid extraction column could be operated conveniently at temperature of the order of 30 °C to 40 °C.

Thus using newer solvents-(Dmf) and (Dmso) however as mixed solvents in combination with antisolvent water to increase their selectivity, appears to be a very attractive proposition for liquid liquid extraction of aromatics.

# 6.8.0 SUMMARY OF LIQUID – LIQUID EXTRACTION OF AROMATICS IN PACKED COLUMN:-

Liquid – liquid extraction of aromatics has been performed in packed column using feed stock consisting of aromatic-Benzene and aliphatic- Hexane.and Mixed solvents-(80%Dmf+20%W) and (80%Dmso+20%W) 685 Temperature of 30 0C and 40 0C for Dmf and Dmso has been utilized for extraction of aromatic –Benzene from mixture of Benzene+Hexane. Relevant ranges of dispersed phase flow rate % continuous phase flow rate (Vc) etc. and details regarding various mass transfer aspects investigated have been listed below.

#### 6.8.1 Details for mixed solvent - 80%Dmf+20%W:-

For liquid – liquid extraction of aromatics in packed column, the solvent consisting of 80% Dmf and 20% water was used as a continuous phase and feed consisting of 50% aromatics + 50% aliphatics was used as a dispersed phase. All the experiments were performed at temperature 30  $^{\circ}$ C in a 5.0 cms. diameter column packed with glass Raschig rings/ceramic Raschig rings under different sets of conditions. However, some selected experiments were also performed in a 15 cm diameter packed column in order to confirm that the results obtained in 5 cm diameter column are free of "End Effects" and "Wall Effects".

The operating variables studied were the following :

(i)Continuous phase flow rate (Vc) varied from 0.1 cm/min to 0.7 cm/min.

(ii) Dispersed phase flow rate (Vd) varied from 0.2 cm/min to 2.0 cm/min.

(iii)Solvent to feed ratio (S/F) by wt. varied from 1.0 to 10.

The values of % hold up of dispersed phase, % aromatic extracted and % purity of extract were determined under different sets of conditions. The values of mass transfer rates, number of transfer units (NTU), height of transfer unit (HTU) and mass transfer coefficients (dispersed phase side as well as continous phase side) were also estimated.

#### 6.8.2 Details for mixed solvent - 80%Dmso+20%W:-

Experiments were also performed at 40  $^{0}$ C using the second solvent consisting of 80% Dmso and 20% water as a continuous phase under otherwise other conditions like feedstock as dispersed phase etc. being the same as mentioned for first solvent – 80% Dmf and 20% water. The range of operating variables were as under:-

(i) Continuous phase flow rate (Vc) varied from 0.25 cm/min to 1.0 cm/min.
(ii)Dispersed phase flow rate (Vd) varied from 0.2 cm/min to 2.0 cm/min.
(iii)Solvent to feed ratio (S/F) by wt. varied from 0.5 to 5.0.

All the mass transfer aspects investigated for first mixed solvent-(80%Dmf-20%W), have been also investigated for second mixed solvent-(80% Dmso+20%W)

6.8.3 Most important generalized conclusions applicable to both mixed solvents-(Dmf+W) & (Dmso+W):-

(I) Hold-up of the dispersed phase:-

(i)As dispersed phase flow rate (Vd) increases, the value of % hold up (%x)increases. The continuous phase flow rate (Vc) exercises little effect on the values of % hold up.

(ii) The values of dispersed phase hold up can be satisfactorily correlated by the correlation proposed by Pratt and co-workers<sup>100</sup> The agreement between the values of characteristic droplet velocity (Vo) obtained experimentally and predicted by Laddha's correlation<sup>101</sup> appears to be satisfactory. Hence the well known Laddha's correlation can also be used in a petroleum refinery engineering field for liquid – liquid extraction of aromatics.

(II) Values of %AE and %PE:-

(i) With an increase in the dispersed phase flow rate Vd for a fixed value of continuous phase flow rate (Vc), the value of % aromatic extracted decreases., however marginally.

(ii)Further with an increase in the ratio of S/F, for a fixed value of dispersed phase flow rate, the values of % aromatic extracted %(AE) increases considerably. The values of %  $P_E$  remain practically the same i.e. of the order of 95-98%.

(ii) Some selected experimental observations were also taken by carrying out liquid-liquid extraction of aromatics in a packed column of 15 cm dia. The values of %AE and %PE obtained for the two cases under consideration are comparable. Hence, it has been concluded that the exhaustive mass-transfer data obtained in a packed column of 5 cm. diameter is free from "End effects" and "Wall effects" Thus mass transfer data obtained in this investigation in a column of 5 cm diameter, could be utilized conveniently for the scale up purpose.

(iv) Favorable operating conditions for single stage liquid-liquid extraction of aromatics have been mentioned for both the mixed solvents under consideration- i.e. (Dmf+W) and (Dmso+W).

#### (III) Mass Transfer data on NTU and HTU :-

(i)The values of NTUod, NTUoc, HTUod and HTUoc have been obtained under different sets of operating conditions for liquid –liquid extraction of aromatics using mixed solvent – Dmf+W. Based on the average values of HTU)d and HTU)c obtained in this investigation, it can be concluded that during liquid-liquid extraction of aromatics when benzene gets transferred from dispersed phase to continuous phase consisting of (80%Dmf+20%W), the resistance to mass transfer lies in both the films.

(ii)The values of NTUod, NTUoc, HTUod and HTUoc have been obtained under different sets of operating conditions for liquid –liquid extraction of aromatics using mixed solvent – Dmso+W. Based on the average values of HTU)d and HTU)c obtained in this investigation, it can be concluded that during liquid-liquid extraction when benzene gets transferred from dispersed phase to continuous phase consisting of 80%Dmso+20%W, the major resistance to mass transfer lies in the dispersed phase film and not in the continuous phase film.

(iii) Mass transfer data on NTU and HTU values is expected to be very useful while designing a packed column to be utilized on industrial scale for liquid-liquid extraction of aromatics using newer solvents –Dmf and Dmso.

#### (IV)Mass transfer coefficients - Kod.a and Koc.a:-

(i) The values of Kod..a and Koc..a have been obtained under different sets of operating conditions for liquid-liquid extraction of aromatics using solvents-(Dmf+W) and (Dmso+W). The values of Kod.a appear to be a strong function of dispersed phase flow rate (Vd) and continuous phase flow rate(Vc) exercises little effect on the value of Kod.a for both the cases under consideration..

(ii) The range of values of Koc.a appear to be higher than the range of values of Kod.a in the case of liquid-liquid extraction of aromatics using mixed solvent-(Dmf+W). Thus, resistance to mass transfer for transfer of aromatics – benzene from

dispersed phase to continuous phase lies in the both phases- i.e. dispersed phase film as well as continuous phase film.

(iii) The range of values of Koc.a appear to be substantially higher than the range of values of Kod..a in the case of liquid-liquid extraction of aromatics using mixed solvent- (Dmso +W). Hence, resistance to mass transfer for transfer of aromatics benzene from dispersed phase to continuous phase basically lies in the dispersed phase film only.

Mass transfer data on(Kod..a) &(Koc..a) values is expected to be very contributory and useful while designing a packed column on industrial scale for liquid-liquid extraction of aromatics.

#### (V) Multi stage operation in a packed column:-

Since the range of values of %AE obtained during single stage extraction are relatively on lower side for both the mixed solvents under consideration, it is preferable to carry out liquid-liquid extraction of aromatics in a multi stage wise manner. "The mass transfer data obtained in this regard" can also be considered as one of the most important contribution of this research work.

Favorable operating conditions for both the mixed solvents under consideration have been mentioned. It has been concluded that by utilizing S/F ratio of the order of 5 to 6 by wt. having (Vc)avg in the range of 0.6 cm/min to 0.8 cm/min and (Vd)avg in the range of 0.15 cm/min to 0.2 cm/min , when one performs liquid-liquid extraction in a stage wise manner consisting of three stages , the value of % aromatics extracted (%AE) appear to be of the order of 95% and %PE is expected to be of the order of 96%.

#### (VI) Comparison of %AE & %PE Values with Limiting Values:-

It is interesting to observe that the "Lmiting Values of %AE and %PE" calculated based on the" Quaternary Liquid-liquid Phase Equilibrium Data " obtained in this investigation can be achieved in actual practice; however only under a fixed set of operating conditions of (Vc, Vd, S/F &  $\theta$ )for both the mixed solvents –(80% Dmf+20% W) & (80%Dmso+20%W). Obtaining "Limiting Values of %AE and %PE" for multi component feed mixture is very tedious and time consuming. However, such Limiting Values for multicomponent feed mixture and extraction being carried out in a multi stage manner has also been attempted in this investigation. Summary of all the relevant calculations have been presented in next Chapter –VII.

It is expected that these Limiting Values of % AE & %PE " can also be achieved in actual practice under a fixed set of operating conditions of ( Vc, Vd, S/F, &  $\theta$ ) for both the mixed solvents ( Dmf+W ) & ( Dmso+W ).

### · (VII) Designing of Packed column for Liquid-Liquid Extraction

In summary, it can be concluded that "Mass transfer Data" for extraction of aromatics obtained in this investigation could be utilized conveniently in the designing of liquid – liquid extraction column for liquid – liquid extraction of aromatics.