



Al-Mustaqbal University College

Chemical Engineering and Petroleum Industries

Unit Operations Lap

Experiment (8)

YORK-SCHEIBEL EXTRACTION COLUMN

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EXPERIMENT:

- ❖ To study the extraction of benzoic acid from toluene (dispersed phase) by water (continuous phase) using packed bed.

OBJECTIVE:

- ❖ To determine overall mass transfer co-efficient based on continuous phase i.e. water; k_{wa} .
- ❖ To determine overall mass transfer co-efficient based on dispersed phase i.e. toluene; k_{tb} .

INTRODUCTION:

The liquid extraction operation entails the transfer of one constituent of a solution to another liquid phase which is relatively immiscible (insoluble) in the first solution. If the constituent distributes differently between the two liquid phases, a certain degree of separation will occur. The degree of separation based on this solubility difference can be amplified by providing multiple stage countercurrent contacting and subsequent separation of the phases, similar to the distillation operation. In distillation, density differences between the gas-liquid phases are sufficient to permit adequate dispersion of one fluid in the other. However, in liquid extraction density differences are significantly smaller and mechanical agitation of the liquids is frequently employed at each stage to increase the mass transfer rates.

The primary objective of this experiment will be to determine the overall efficiency of the York-Scheibel column, employing butanol as the solvent to extract acetic acid from a water solution of the acid. The lighter n-butanol (dispersed phase) is to be passed up the column countercurrent through the more dense water/acetic acid solution (continuous phase) passing down through the column. For this operation the interface level between the phases is at the top of the column and can be maintained through the adjustment of the butanol out (extract) rotameter.

EQUIPMENT DESCRIPTION:

A York-Scheibel extraction column will be employed in this experiment to provide for the staged countercurrent contacting of the liquid phases. This is an agitated column extractor having internal paddle impellers connected to a central rotating shaft, with the motor drive being mounted above the tower. The sets of impellers for each stage are separated by partitions or calming sections to give a stack of mixer-settlers, one above the other. The calming sections consist of wire mesh packing to induce coalescence and separation of the liquid phases.

THEORY:

The experimental set up is designed for extraction of benzoic acid from toluene solution with water. Toluene solution here acts as dispersed phase and water acts as continuous phase. Toluene being lighter than water is Fed through bottom of extraction of water is flows upward in tower under buoyancy forces. Water which is here continuous phase flows downward under gravity and its flow is controlled by positioning its outlet head (Height).

Benzoic acid is transferred to water during extraction. The transferred quantity is determined by volumetric titration; to determine mass transfer co-efficient.

The solution whose components are to be separated is the feed to the process. The feed is composed of a diluent and solute. Liquid added to the feed for purposes of extraction is the solvent. If the solvent consists primarily of one substance (aside from small amounts of residual feed material that may be present in a recycled, recovered solvent), it is a single solvent. A solvent consisting of a solution of one or more substances chosen to provide special properties is a mixed solvent. The solvent-lean, residual feed solution, with one or more constituents removed by extraction, is the raffinate. The solvent-rich solution containing the extracted solute(s) is the extract.

The minimum requirement for the liquid extraction is the intimate contact of two immiscible liquids for the purpose of mass transfer of constituents from one liquid or phase to the other, followed by physical separation of the two immiscible liquids. Any device or combination of devices that accomplishes this once is a stage. Each stirrer and packing complex in the column is considered a stage. If the effluent liquids are in equilibrium, so that no further change in concentration would have occurred within them after longer contact time, the stage is a theoretical or ideal stage. The approach to equilibrium actually attained is the stage efficiency. A multi-stage cascade is a group of stages, arranged for countercurrent or other type of flow of liquids from one to the other for purposes of enhancing the extent of separation

PROCEDURE:

- ❖ Prepare a mixture of toluene and benzoic acid as feed mixture, with conc. of benzoic acid in the range of 1-2 kmole/m³.
- ❖ Fill the column with continuous phase (water).
- ❖ Allow the dispersed phase (toluene+benzoic acid) to enter from bottom of the column at some minimum rate.
- ❖ Adjust the two flow rates to some prefixed value (1 to 4×10^{-2} m³/s for dispersed phase and 0.5 to 2×10^{-2} m³/s)
- ❖ The inter phase at the top of water must be worked up and down as necessary by adjusting the height of water outlet.
- ❖ Take the first observation after steady state has reached. Steady state refers to the
 - (a). Steady flow rates of continuous phase and dispersed phase.
 - (b). Steady height of inter phase.
- ❖ At steady state record the flow rates of continuous phase i.e. water [Q_w in l/h] and ambient temperature.
- ❖ Collect the sample of extract and raffinate and measure the conc. of benzoic acid in each.
 - (a). Use standard solution of NaH (N/2 or N/5) in alcohol for titration (of dispersed phase i.e. Toluene + Benzoic acid after extraction)
 - (b). Use standard solution of NaOH (N/10 or N/20) in water for titration against water after extraction.
- ❖ The procedure is repeated for different flow rates.

OBSERVATION:

Column diameter D_c=

Calming section height Z =

Agitated section Diameter of Rashing ring=

Ambient temperature=

Initial conc. of Benzoic acid C_{t1} = gmole/L

Initial conc. of Benzoic acid in water C_{w2}= 0.0 gmole/ (as pure water is used)

CALCULATIONS:

- ❖ Determining concentration of Benzoic acid in water:

$$\begin{aligned} \text{Vol. titration at bottom (feed)} \quad V_1 &= 10 \text{ ml Toluene sol.} \\ N_1 &= ? \end{aligned}$$

$$\begin{aligned} \text{Toluene sol.} & & \text{Water sol.} \\ N_1 V_1 &= & N_2 V_2 \quad N_1 = \text{----- g eq / l} \end{aligned}$$

Similarly conc. of Benzoic acid at top is determined.

- ❖ Volumetric titration for water sol. after extraction are required which are done using 10 ml of water sol. with N/10 NaOH solution in water.
- ❖ Overall mass transfer co-efficient based on continuous phase. k_{wa}

$$2) \quad K_{wa} = \frac{N/t}{V(C_t)_{ln}} \quad \text{Water } (C_w)_{In} = \left(\frac{(C_{w1}^* - C_{w1}) - (C_{w2}^* - C_{w2})}{\ln \left[\frac{(C_{w1}^* - C_{w1})}{(C_{w2}^* - C_{w2})} \right]} \right)$$

$$\text{Similarly, } K_{ta} = \frac{N/t}{V(C_t)_{ln}}$$

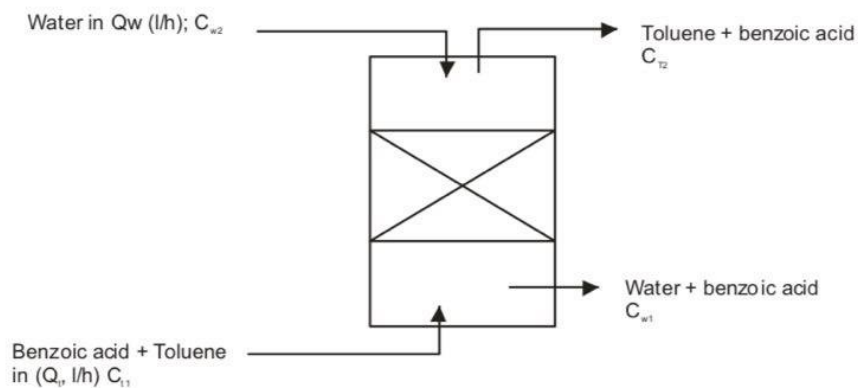
EQUILIBRIUM DATA:

Aqueous phase, C_{w1} gmole/L	0	.0016	.0032	.0064	.0080	.00961	.01121	.012816	.01442
Benzoic acid									
Organic phase, C_{w1} gmole/L	0	.0080	.0240	.0336	.0673	.08811	.012015	.14097	.17301
Benzoic acid									

The equilibrium data can be represented by:

$$C_w/C_t = H = 0.167267 - 0.837941 C_t + 2.27418 C_t^2$$

$$\begin{aligned} \text{and } C_w &= 0.00227866 + 0.0745606 C_t & m &= dc_w/dc_t \\ & & &= 0.00227418 C_t^2. \end{aligned}$$



LIQUID-LIQUID EXTRACTION IN YORK SCHEIBEL COLUMN

References By:

Rowan University – Department of Chemical Engineering

MATERIALS

- ❖ 10 L of 2 M Acetic Acid in Water
- ❖ 1-Butanol
- ❖ Density Meter
- ❖ 100 mL graduated cylinder
- ❖ Stop Watch
- ❖ Sample jars
- ❖ 20 L Nalgene container
- ❖ Beakers

MANIPULATED VARIABLES

Two runs will be performed with the water/acetic acid solution as the continuous phase and butanol as the dispersed phase. Two different feed ratios will be used and the results will be compared. The flow rates into the column will remain constant throughout both runs. The stirrer setting will also remain constant at a setting of 5. During the run, the interface level between the heavy and the light phase needs to be maintained at the top of the column between the top mesh and the butanol out (extract) nozzle. This interface is controlled by making small adjustments to the butanol out rotameter. Closing the rotameter causes the interface to move down the column, whereas, opening the rotameter causes the interface to rise up the column. During startup, it is necessary to close the butanol out valve completely in order to get the interface to form.

Rotameter Calibrations

The following rotameter calibration equations are provided to set the flow in of water with acetic acid and butanol into the column:

$$\{\text{Rotameter setting}\} = 0.6139 \cdot \{\text{flow (mL/min)}\} + 15.401 \text{ for 1 M water \& acetic acid}$$

$$\{\text{Rotameter setting}\} = 1.4552 \cdot \{\text{flow (mL/min)}\} - 15.438 \text{ for 2 M water \& acetic acid}$$

$$\{\text{Rotameter setting}\} = 0.7777 \cdot \{\text{flow (mL/min)}\} + 23.496 \text{ for butanol}$$

SAFETY:

- ❖ Safety glasses should be worn throughout the lab.
- ❖ Gloves should be worn when handling butanol and acetic acid.
- ❖ Butanol is a flammable liquid and should be handled with care.

PROCEDURE:

Preliminary: Prepare a 2 Molar Acetic Acid Solution in water. For 10 Liters of solution:

- ❖ Measure out 8.856 L tap water (DI water is not necessary) in a 20 L Nalgene container
- ❖ Add 1.1439 Liters of glacial acetic acid
- ❖ Mix/ Shake Well

START UP:

- ❖ Check to see that all the drainage valves are closed.
- ❖ Place the prepared acetic acid solution in the feed tank.
- ❖ Fill the butanol feed tank with butanol.
- ❖ Check to be sure the top water vent valve is open.
- ❖ Fill the column slowly with the water/acetic acid solution by turning on the left motor with the breaker on the right power panel. Set flow rate on left rotameter between 80 or 90 by adjusting the pump speed adjustment. **DO NOT ADJUST THE LOWER TWO ROTAMETERS.**
- ❖ When the liquid level in the column reaches the top right nozzle (water in nozzle), turn the water flow rate down to the desired set point. (See table 1) Turn on and set the butanol flow rate to the desired set point (See table 1) by adjusting the pump speed, and close the top water vent.
- ❖ Close the butanol out rotameter when the liquid level reaches the top left (butanol out) nozzle. (This will force water out the bottom of the column and allow the interface to form at the top)
- ❖ Allow the interface to form between the top mesh and the top left nozzle (butanol out). The interface appears as an immiscible layer between butanol and water with droplets of butanol accumulating at this interface.
- ❖ Once the interface is formed in the desired location, open the butanol out rotameter slowly until there are butanol and water flow rates out of the column. Adjusting this rotameter is used to

control the interface level. Opening the rotameter causes the interface to rise, while closing it causes the interface to drop. The optimum setting of this rotameter will allow for a semi-stable interface and give a minimal amount of drift in the interface level. Small adjustments should be made in order to keep the interface constant.

- ❖ Set the stirrer speed to a setting of 5 using the dial on the top right of the lab equipment panel. Make sure the motor is powered with the top center switch on the right power panel.
- ❖ Allow the column to run until steady state is achieved (about ½ hour). While waiting for steady state, adjustments may be made to the butanol out rotameter to keep the interface at the desired level (table 1).

Table 1: Rotameter settings

Run #	Rotameter Setting			Flow Rate (mL/min)		Rotor Speed Setting
	n-Butanol Inlet	H ₂ O w/acetic acid Inlet	n-Butanol Flow out	n-Butanol	H ₂ O w/acetic acid Inlet	
1	39	29	~ 39	19.78	30.54	5
2	32	29	~ 32	10.675	30.54	5

OPERATING PROCEDURE:

Once the column has reached steady state and the interface is well-maintained samples may be taken from the extract and raffinate streams.

SAMPLING PROCEDURE AND ANALYSIS:

- ❖ During operation, collect a sample of water (raffinate) in a sample jar (about 50 mL is necessary).
- ❖ Similarly collect a sample from the butanol extract stream. Label all sample jars appropriately.
- ❖ Measure the flow rate (g/mL) of the water out (raffinate) stream with a graduated cylinder and stopwatch. (Be sure the tubing allows the water to freely flow out and does not accumulate. (It is not necessary to measure the butanol out flow rate, this can be determined through an overall mass balance)

DENSITY METER OPERATION:

- ❖ Fill a 300 mL beaker with DI water and have an empty 300 mL beaker available.
- ❖ Place a plastic sample tube on the sample inlet
- ❖ Turn the density meter on

- ❖ Turn the hand wheel upward: the air in the bellows will be expelled.
- ❖ Immerse the sample tube in a beaker of distilled water.
- ❖ Turn the hand wheel downward: the water is drawn up through the measuring tube and partially fills the bellows (be sure the measuring tube is full of water free from bubbles).
- ❖ Check to see if the measured density corresponds to the given density on table 2 below. (If the value does not match within $.001 \text{ g/cm}^3$, the density meter should be calibrated with distilled water. See user manual for instructions.
- ❖ Measure and record the densities of raffinate and extract samples. Be sure to clean the sample tube between samples by filling and expelling the tube 2-3 times with water. Place the expelled samples in a waste container.

Table 2: Density table of distilled water free from bubbles

Temperature (°C)	0	1	2	3	4	5	6
Density (g/cc)	0.99984	0.99990	0.99994	0.99996	0.99997	0.99996	0.99994

Temperature (°C)	7	8	9	10	11	12	13
Density (g/cc)	0.99990	0.99985	0.99978	0.99970	0.99961	0.99950	0.99938

Temperature (°C)	14	15	16	17	18	19	20
Density (g/cc)	0.99925	0.99910	0.99894	0.99878	0.99860	0.99841	0.99821

Temperature (°C)	21	22	23	24	25	26	27
Density (g/cc)	0.99799	0.99777	0.99754	0.99730	0.99705	0.99679	0.99652

Temperature (°C)	28	29	30	31	32	33	34
Density (g/cc)	0.99624	0.99595	0.99565	0.99534	0.99503	0.99471	0.00438

Temperature (°C)	35	36	37	38	39	40
Density (g/cc)	0.99404	0.99369	0.99333	0.99297	0.99260	0.99222

SHUT DOWN PROCEDURE:

- ❖ Turn off all inlet flow rates on the right control panel.
- ❖ Shut off the stirrer on the right control panel.
- ❖ Open the top water in vent valve.
- ❖ Open the bottom center black valve to drain the column.

REPORT:

- ❖ Determine compositions and flow rates in mass units for writing a column material balance. The calibration equation is provided for acid content in water saturated with butanol. Density = $\{0.0082 * (\text{Molarity}) + 0.9858\}$ Draw the flow chart for the system using a standard column diagram as in Wankat [1].
- ❖ Obtain the necessary equilibrium data and/or equation from Wankat [1] for the n-butanol, acetic acid, and water system.
- ❖ Plot the equilibrium data and operating data on a McCabe-Thiele diagram to determine the number of theoretical equilibrium stages. (Refer to Wankat, Example 16-1 [1] separation of Acetic Acid from n-Butanol using solvent water.)

York Scheibel Extraction Column

