Introduction:

The separation of the components of a liquid mixture by treatment with a solvent which one or more of the desired components is preferentially soluble is known as liquid-liquid extraction, i.e. separation of hydrocarbons in petroleum industry. In the operation it is essential that the liquid mixture feed and the solvent are at least partially or completely immiscible.

Three stages are involved: 1- bringing the feed mixture and the solvent in to intimate contact. 2- Separation of the resulting two phases. 3-Removal and recovery of the solvent from each phase.

It is possible to combine stages 1 & 2 in to a single piece of equipment such as a column.

Theory:

Write the theoretical back ground of liq.-liq. Extraction and derive the equations used in the calculations.

Aim of the Experiment:

To study the extraction process of (Kerosene - acetic acid- Water) system:

1- Calculate The Concentrations of the inlet and outlet raffinate (CR₁& CR₂) and extract(CE₁ & CE₂).

- 2- Calculate: HOE, NOE, K_{OE} *a
- 3- Check the process had reached the steady state
- 4- Calculate the process efficiency

Flow Diagram and Description of the Extraction Unit:



Fig.1 The liquid-liquid extraction unit.



Fig.2 Flow Diagram of Liquid-Liquid Extraction Unit.

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Spray column

Pump 1: Used to pump the inlet pure water to the side of the column top.

Pump 2: Used to pump the kerosene from vessel 1 to the bottom of the spray column

Vessel 1: The inlet kerosene + acetic acid (CR₁)

Vessel 2: The outlet kerosene with a little conc. of acetic acid CR₂

Vessel 3: The inlet pure water CE₂

Vessel 4: The outlet water + the extracted acetic acid CE₁

Rotameter 1: Used to measure the flow rate of the inlet kerosene + HAC. It is calibrated using water at 20°C and 1 atm., so its readings should be corrected.

Rotameter 2: Used to measure the flow rate of the inlet water. It is calibrated as water at 20°C and 1 atm., so its readings did not need to be corrected.

Rotameter 3: Used to measure the flow rate of the outlet water+ the extracted HAC.

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Procedure:

1- The pure water was pumped from vessel 3 using pump 2 to rotameter 2, which was used to measure the volumetric flow rate of the inlet water. The water entered from the upper side of the spray column as a shower. The outlet water+ acetic acid left from the bottom side of the column and entered to rotameter 3 to measure the volumetric flow rate then collected in vessel 4. Both the flow rates of inlet and outlet water were controlled to be the same and the system should be left to reach stability then the stream of (kerosene + acetic acid) was pumped.

2- The kerosene + acetic acid stream was pumped from vessel 1 to rotameter 1, which was used to measure the flow rate of kerosene + acetic acid. Note that the rotameter was calibrated using H_2O at $20^{\circ}C$ and 1 atm., so its readings should to be corrected. The stream entered from the column bottom as droplets. The outlet stream of kerosene + the remained HAC were removed from the column top and collected in vessel 2.

3-The system was left to reach the steady state. 4- Four samples, with a volume of 10 ml, have been taken from the four streams and titrated with 0.1 N NaOH solution with a few drops of phenolphthalein. The volume of NaOH was recorded for each and the concentrations were calculated (CR₁, CR_2 , CE₁ &CE₂).

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Date of the experiment:

Date of Report Delivery:

DATA:

Column Inside Diameter= 0.152m

Column Height (Z) = 1.62m

Results:

1	Volumetric flow rate of inlet kerosene	60
	+ HAC ,L _R (liter /h)	
2	Volumetric flow rate of inlet pure	30
	water ,L _E (liter /h)	
3	Volumetric flow rate of outlet water+	30
	HAC , (liter /h)	
4	Normality of inlet kerosene + HAC	
5	Normality of outlet kerosene +HAC	
6	Normality of inlet pure water	0
7	Normality of outlet water+ HAC	

Group Names: 1-

3- 4-

2-

Lab. Conditions:

Air Temperature = , Air Humidity=

Asst. Prof. Maha M. Alhussaini

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*Fig.3 Counter current flow of the spray column

* Spray column can be considered as a packed column without packing.

1- Prove that the normality equals to the concentration for (kerosene-HAC-water) system. Fix the values of CE_1 , CE_2 , $CR_1 \& CR_2$.

2- Plot the operating line.

3- The equilibrium curve can be taken as a straight line, then assuming equilibrium at the interface. Plot the equilibrium line, on the same graph with the operating line, using the following equation:

4-Calculate $\Delta \text{CE}_{\text{Im}}$:

$$\Delta CE_{lm} = \frac{(CE^* - CE)2 - (CE^* - CE)1}{ln\frac{(CE^* - CE)2}{(CE^* - CE)1}}$$

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:

5- Calculate NOE

$$\text{NOE} = \frac{CE_{1-} CE_2}{\Delta CE_{lm}}$$

7- Calculate K_{OE}*a:

$$HOE = \frac{L_E}{K_{OE} \times a}$$

Note: L_E unit is m^3/m^2 . sec

8- Check if the system had reached the steady state:

 $L_{E} (CE_{1}-CE_{2}) = L_{R} (CR_{1}-CR_{2})$

9- Calculate the process efficiency:

$$\% \operatorname{Eff} = \frac{CR_{2-} CR_1}{CR_2 * - CR_1}$$

Note:

The rotameter readings of the inlet (kerosene+ HAC) should be corrected because the rotameter was calibrated using water. Use the following equation:

$$\frac{Q_1}{Q_2} = \sqrt{\frac{\rho_2(\rho_f - \rho_1)}{\rho_1(\rho_f - \rho_2)}}$$
 Derive the equation in the theory

Where:

 ρ_f : Float density (7960 Kg/m³)

 ρ_1 : Density of water at 20°C & 1 atm. (1000 Kg/m³)

 ρ_2 Density of kerosene + HAC at 20°C & 1 atm. (880 Kg/m³), measured by using pycnometer or hydrometer.

Q₁: Volumetric flow rate recorded by the rotameter (liter/hr.)

Q₂: The corrected volumetric flow rate (liter/hr.)

Symbols

 CR_1 , CR_2 , CE_1 , CE_2 : The concentrations of the solute in the raffinate and extract phases (Kmole/m³)

HOE : Height of overall transfer unit based on concentration in extract phase(m).

NOE: Number of overall transfer unit based on concentration in extract phase(m).

a: The interfacial area per unit volume

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KoE :The overall transfer coefficient for extract

D_i: Column inside diameter (m)

Z: Column Height (m)

CE^{*}: The concentration in phase E in equilibrium

with C_R in phase R

 $\Delta \text{CE}_{\mbox{ Im}}$:The logarithmic mean driving force

 $L_{E,L_{R}}$: The volumetric flow rates of extract and raffinate phases per unit area (m³/m². sec).

References:

- 1- Foust, "Unit Operations"
- 2- Coulson & Richardson," Chemical Engineering", Vol.2
- 3- Treble, "Mass Transfer Operations".

Asst. Prof. Maha M. Alhussaini