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# Liquid-Liquid Extraction of O-Cresol from O-Cresol and Water Mixture by Using Ethylene Dichloride as an Extractant

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***Abstract:*** **Liquid-liquid extraction is a unit operation and it is a process of transferring a solute from one liquid phase to another immiscible or partially miscible liquid in contact with the first. The liquid- liquid extraction of O-Cresol and Water mixture (38.1% O- Cresol, 61.9% water) is carried out by using Ethylene Dichloride as a solvent. Liquid-liquid extraction is a method to achieve high recovery of solute with good selectivity in recovery of O-cresol from O-cresol and water mixture. The liquid- liquid extraction efficiency is calculated in terms of KD and degree of extraction. In liquid-liquid extraction, chemical equilibrium experiments are carried to calculate the number of stages required to recover high purity and yield of O-Cresol by using Ethylene Dichloride. It was found that the rate of extraction increase with increases in the solvent to feed ratio. By using this process 99% pure O-Cresol from the bottom of distillation column can be obtained.This O-cresol can be used as a raw material for producing DMBPC & this will also helpful in minimizing water pollution & Also energy conservation.**

***Keywords:*** **Liquid-liquid counter current Extraction, Distribution Coefficient (KD), Degree of extraction (%E), O-Cresol, Ethylene Dichloride, Water.**

## 1. INTRODUCTION

**Liquid–Liquid Extraction:**

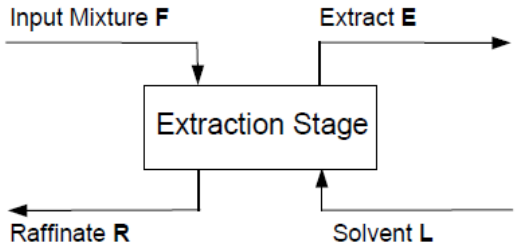


**Fig. 1** Schematic representation of an extraction-stage

The liquid-liquid extraction is defined as separation of the components of a liquid mixture by treatment with a solvent in which one or more of the desired components is preferentially soluble. e.g. in the processing of coal tar liquids and in the production of fuels in the nuclear industry, and which has been applied extensively to the separation of hydrocarbons in the petroleum industry. In this operation, it is essential that the liquid-mixture feed and solvent are at least partially if not completely immiscible & in essence, three stages are involved:

(1) Bringing the feed mixture and the solvent into intimate contact,

(2) Separation of the resulting two phases, and

(3) Removal and recovery of the solvent from each phase after extraction.

It is possible to combine stages (1) and (2) into a single piece of equipment such as a column which is then operated continuously. Such an operation is known as differential contacting. Liquid–liquid extraction is also carried out in stage wise equipment and the prime example being a mixer–settler unit in which the main features are the mixing of the two liquid phases by agitation, followed by settling in a separate vessel by gravity. This mixing of two liquids by agitation is of considerable importance. Extraction is in many ways complementary to distillation and is preferable in the following cases:

1) Where distillation would require excessive amounts of heat, e.g. when the relative volatility is near unity.

2) When the formation of azeotropes limits the degree of separation obtainable distillation.

3) When heating must be avoided.

4) When the components to be separated are quite different in nature.

Depends upon following factors process can be selected,

(1) Feed Composition

(2) Economy

(3) Safety

In the extraction column O- cresol is separated from water by using EDC as a solvent. As the O -Cresol is more soluble in EDC; it is recovered as extract phase.

## 2. EXPERIMENTAL MATERIAL

### 2.1 Physical properties

**O-Cresol**: molar mass 108.14g/mol and density 1.04 g/ml.

**Ethylene Dichloride:** molar mass 98.96 g/mol mol and density 1.26 g/ml.

**Water:** molar mass 18 g/mol and density 1 g/ml.

### 2.2 EXPERIMENTAL PROCEDURE:

#### *2.2.1 For Raffinate phase:*

1). Take 7conical flasks and no. them as 1, 2…7.

2). Take 30 ml water in each flask add 0, 6, 12, 18,24,30,36 ml of EDC respectively in flask no. 1, 2, 3….7.

3).Measure the volume of o-cresol consumed in burette till the turbid solution is formed in the conical flask.

4).Note down the volume of o-cresol required in each flask.

5).Find out the composition of three component mixture present in conical flask.

6).Plot the composition on a triangular plot as raffinate curve.

#### *2.2.2 For extract phase:*

1).Take 7 conical flasks and no. them as 1, 2…7.

2).Take 30 ml o-cresol in each flask add 0, 6, 12, 18,24,30,36 ml of EDC respectively in flask no.1, 2, 3….7.

3). Measure the volume of water consumed in burette till the turbid solution is formed in the conical flask.

4). Note down the volume of water required in each flask.

5. Find out the composition of three component mixture present in conical flask.

6).Plot the composition on a triangular plot as an extract curve

### 2.3 EXPERMENTAL DATA:

For Plotting the “BINODAL CURVE” the Raffinate & Extract Phase Data should be needed. The following data is generated from “VOLUMETRIC TITRATION”.

Table No. 6.1 Raffinate & Extract phase

A= Water, B= EDC, C=O-Cresol.

**Extract Phase Data (wt. % basis)**

**Raffinate Phase Data (wt. % basis)**

**Table No. 3.1**

| Sr. No. | A In ML | C B.R in ML | B In ML | Weight in gm | | | Mass fraction | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | A | C | B | %A | %C | %B |
| **1** | 30 | 0.97 | 0 | 30 | 1.02 | 0 | 3.20 | 96.88 | 00.00 |
| **2** | 30 | 1.718 | 6 | 30 | 1.80 | 7.518 | 9.59 | 74.84 | 15.57 |
| **3** | 30 | 11.13 | 12 | 30 | 11.69 | 15.03 | 20.62 | 56.06 | 23.32 |
| **4** | 30 | 20.76 | 18 | 30 | 21.8 | 22.55 | 29.32 | 43.52 | 27.16 |
| **5** | 30 | 28.97 | 24 | 30 | 30.42 | 30.07 | 33.67 | 36.21 | 30.12 |
| **6** | 30 | 36.64 | 30 | 30 | 38.48 | 37.59 | 36.28 | 31.24 | 32.49 |

**Table No. 3.2**

| Sr. No. | A In ML | C B.R in ML | B In ML | Weight in gm | | | Mass fraction | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | A | C | B | %A | %C | %B |
| **1** | 1.35 | 30 | 0 | 1.35 | 31.5 | 0 | 4.12 | 95.88 | 0 |
| **2** | 2.78 | 30 | 6 | 2.78 | 31.5 | 7.51 | 6.66 | 79.93 | 12.89 |
| **3** | 4.87 | 30 | 12 | 4.87 | 31.5 | 15.03 | 9.48 | 67.78 | 22.74 |
| **4** | 6.32 | 30 | 18 | 6.32 | 31.5 | 22.55 | 10.56 | 59.50 | 29.94 |
| **5** | 10.00 | 30 | 24 | 10.00 | 31.5 | 30.07 | 13.98 | 51.48 | 34.54 |
| **6** | 21.81 | 30 | 30 | 21.81 | 31.5 | 37.59 | 24 | 41.33 | 34.67 |

## 3. EXTRACTION COLUMN DESIGN

### 3.1 Factor needed for selection of extractor:

Following Factors are needed,

1) The No. of Stages Required

2) The Throughputs

3) The Settling Characteristics of Phases

4) The available Floor Area & Head Room

### 3.2 Design Calculation:

***3.2.3 Mass balance for the counter current extraction:***

$$\begin{aligned}
&\text{1. stage} && m_F + m_{E2} = m_{R1} + m_{E1} \\
& && m_{R1} - m_{E2} = m_F - m_{E1} = Q \\
&\text{1. to 2. stage} && m_{R2} - m_{E3} = m_F - m_{E1} = Q \\
& && \vdots \\
&\text{1. to n-th stage} && m_{Rn} - m_L = m_F - m_{E1} = Q
\end{aligned}$$

According to these above equations we can define for every stage a constant mass-difference-stream Q and which is equal to the difference between the feed and the extract of the first stage. Every balance-equation (5) to (7) is, according to the mixture law, a line in the triangular diagram. For every stage I, the line cuts the point for the leaving raffinate R i & the

incoming extract E i +1. The lines for all n stages meet also in one fixed point Q & which is therefore called pole or working-point.

The position of Q can be determined, if concentrations are known or demanded:

1). The feed F in the first stage

2). The leaving extract in the first stage

3). The leaving raffinate in n-th stage, determined by the problem

4). The solvent incoming to the last stage (usually pure solvent)

### 3.3 Stage Calculation for Extraction Column by using binodal curve

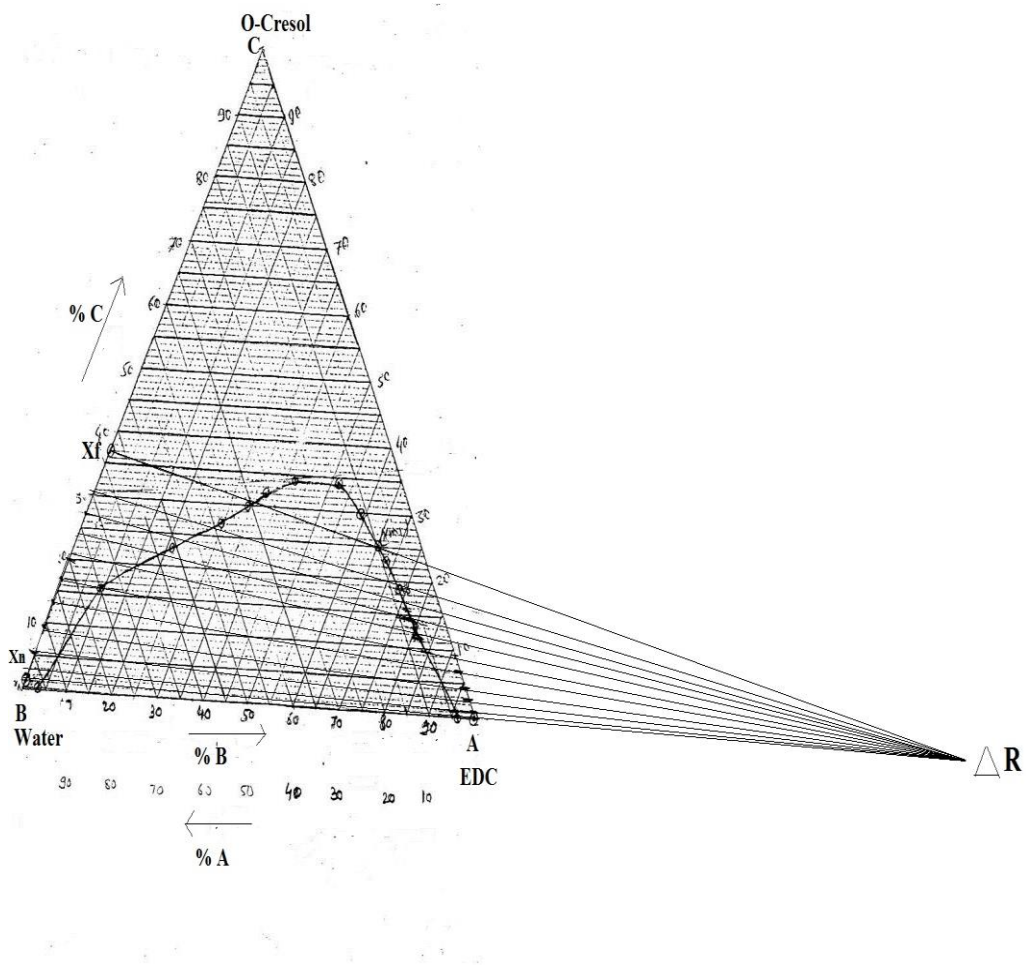


**Fig. No. 3.2 Stage calculation**

### 3.4 Process design of Extraction Column result:

- No. of stages required for 99% recovery of O- Cresol (theoretical) = 10
- Solvent to Feed ratio (based on composition) = 2
- Recovery of O- Cresol by experiment = 96%

## 4. EXPERIMENTAL RESULT AND DISCUSSION

The chemical equilibrium isotherms for liquid-liquid extraction of o-cresol from mixture of o-cresol and water with ethylene dichloride as a solvent. Distribution coefficient (KD) of O-cresol and Ethylene Dichloride was good and with recovery of o-cresol (%of 85.5,90 – 96 at different solvent to feed ratio), range from 1.0,1.5 to 2.0 of o-cresol. At solvent to feed ratio 2.0, highest recovery of o-cresol 96% is achieved at room temperature. It was found that if Solvent to feed ratio increases, the recovery of o-cresol is increases.

**Table No. 4.1**

| Flask No. | ml collected | conc. of O-Cresol in ml (measured in HPLC) |
|---|---|---|
| **V1** | 31.5 | 29.47 |
| **V2** | 10.0 | 2.78 |
| **V3** | 7.5 | 1.10 |
| **V4** | 7.4 | 0.4654 |
| **V5** | 7.2 | 0.038 |
| **V6** | 7.0 | 0.0294 |
| **V7** | 7.7 | 0.0161 |
| **V8** | 7.2 | 0.01152 |
| **V9** | 7.1 | 0.00512 |
| **V10** | 7.4 | 0.00296 |
| **Total** | **99** | ∑ **32.33** |

% Recovery (experimental) = **(**36.92 / 38.1)*100

= 96 **%**

## 5. CONCLUSION

The advanced Extraction Process can be used in many of the industries. To reduce pollution, load on ETP this type of advanced extraction process is used. The extraction process is compatible with various unit operations. For increasing the profit, many of industries use this method.

The liquid-liquid extraction of o-cresol and water with Sodium hydroxide was studied. Distribution coefficient (KD) of O-cresol and Ethylene Dichloride was good and with recovery of o-cresol (%of 85.5,90 – 96 at different solvent to feed ratio), range from 1.0, 1.5 to 2.0 of o-cresol. At solvent to feed ratio 2.0, highest recovery of o-cresol 96% is achieved at room temperature. It was found that if Solvent to feed ratio increases, the recovery of o-cresol is increases.

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