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Separation of a Mixture of Benzoic acid (C_6H_5COOH) and Benzophenone ($C_6H_5COC_6H_5$) by Extraction

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***Abstract*—** Extraction is a technique used to isolate and purify organic compounds when they are in a mixture with other compounds. The reactions between acids and bases were initially studied, with the solubilization and recovery of both the acidic and the basic properties of the compounds. Benzoic acid, naphthole and acetic acid are the acids that reacted with NaOH, Na2CO3 and NaHCO3 and the corresponding salt was formed. Naphtol did not react with NaHCO3 because it is a weaker acid. Depending on the ability to react with or not an acid with carbonates, the compound can be identified as carboxylic acid or phenol. Salt solutions were neutralized with HCl and the original insoluble acids were re-formed. This was followed by the dissolution of amine with HCl to form its salt, and the original molecule was recovered with NaOH. In addition, a mixture of benzoic acid and benzophenone was separated by extraction. Dichloromethane was used as the solvent and the solution was dissolved with NaOH. As a result, benzoic acid was moved to the aqueous layer as salt and benzophenone was moved to the organic layer. After these two layers were separated, the salt of the benzoic acid was neutralized with HCl and the initial acid was formed.

***Keywords*—** Extraction, Separation, Benzoic Acid, Benzophenone, Phenol

I. INTRODUCTION

Extraction is one of the oldest "chemical" activities of man. The preparation of a decoction (coffee, tea, etc.), but also other similar processes, such as the extraction of a fragrance, a dye or an active drug from a vegetable raw material, are basically extraction processes, wherein the desired ingredient with the use of hot water is usually transferred from the vegetable raw material to the aqueous phase.

Extraction is a technique used in organic chemistry to separate desired substances from other undesirable. Extraction involves the selective removal of one or more components of a solid, liquid or gaseous mixture in a separate phase. The extracted substance will be separated between the two immiscible phases in contact and the proportion of its distribution between phases will depend on the relative solubility of the solute in each phase [1].

Solvent extraction is the purification technique commonly used in laboratories using a separatory funnel [2]. This process is used not only to isolate natural products but also to isolate and purify most chemicals reactions. The technique presupposes the distribution of a soluble substance A, between two immiscible liquids. The commonly miscible liquids are water and some organic solvents, such as diethyl ether [$(C_2H_5)_2O$] or dichloromethane (CH_2Cl_2) [1]. In some solvent extractions, the desired product migrates from the initial liquid phase. in the second liquid phase. In other solvent extractions, the impurities migrate from the initial liquid phase to the second liquid phase, thus leaving the product in the initial liquid phase. The phase containing the desired product is called the product layer, while the phase containing the impurities, the excess of reagents and other undesirable compounds is called the waste layer [2].

In acid/base extraction, the molecule to be extracted is converted so that a new solubility is made on the molecule. Acid-base extraction is one of the difficult principles in organic chemistry to understand. The simplest approach to understanding the topic is to create a diagrammatic flow (mentally or on paper) that depicts what species have been created and where the molecule is located [3]. Therefore, the purpose of this study is to provide information for acid-base dissolution and for the isolation and purification of organic acids and bases, when they are in mixtures, with the technique of extraction.

II. METHODOLOGY

Initially, the separator funnel is placed on a holder and it is confirmed that its faucet is closed. A funnel with an elongated stem is placed on the neck of the separating funnel. Then the liquid to be extracted and the extraction solvent are added. The total volume in the separator funnel shall not exceed three-quarters of the volume of the funnel. The cap is placed on the neck of the separator funnel. With particular care, the separator funnel is gently shaken in order to release the excess pressure. The funnel is repositioned on the finger support to completely separate the layers.

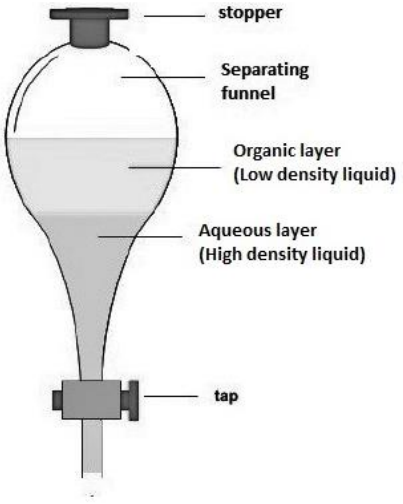

Figure 1: a separating funnel

i) A small amount of C_6H_5COOH (crystalline substance) is put into a test tube and a little distilled H_2O is added. The acid does not dissolve. Check the pH of the suspension. Drops of NaOH solution are added to dissolve the acidity. The amount of NaOH solution required depends on its concentration and the amount of C_6H_5COOH.

Repeat the procedure with $NaHCO_3$ solution and Na_2CO_3 solution instead of NaOH solution, and any dissolution and release of CO_2 is observed. The dissolution experiment was repeated using α- or β-naphthol, instead of C_6H_5COOH, with NaOH solution, Na_2CO_3 solution and $NaHCO_3$ solution.

ii) To the solutions of benzoic acid and naphthol, formed with NaOH solution, HCl solution is added dropwise until solid cloudiness and precipitation is observed.
Exercise is repeated using acetic acid (CH_3COOH) instead of benzoic acid, and any similarities and differences are observed.

iii) The first exercise to solubilize and recover basic compounds (amines) instead of acidic compounds is repeated. Check the pH of a small amount of basic compound H_2O, then dissolve with HCl solution and then recover by adding NaOH solution. All relevant comments are noted.

iv) Separation of a mixture of benzoic acid C_6H_5COOH (mp 122 ° C) and benzophenone $C_6H_5COC_6H_5$ (mp 49 ° C) by extraction (NaOH / H_2O-CH_2Cl_2).

III. RESULTS AND DISCUSSION

Benzoic acid, like all aromatic compounds, having a ring-linked carbon, exhibits two classes of reactions:

- Ring reactions (aromatic). That is, it easily yields hydrogen substitution reactions of the benzene ring.
- Side-group reactions. That is, in the case of benzoic acid, it gives carboxylic acid reactions.

Due to its acidic nature, the sodium salt of benzoic acid (C_6H_5COONa) is formed when the NaOH solution is added to benzoic acid. The reaction that takes place is:

$$C_6H_5COOH + NaOH \rightarrow C_6H_5COONa + H_2O$$

On the other hand, upon addition of $NaHCO_3$ and Na_2CO_3 solution, salt formation, as well as CO_2 gas evolution, is observed. The reactions that take place are:

$$C_6H_5COOH + NaHCO_3 \rightarrow C_6H_5COONa + CO_2 + H_2O$$

$$C_6H_5COOH + Na_2CO_3 \rightarrow C_6H_5COONa + HCO_3^-$$

Naphthol is a phenol, it has acidic properties. Naphthol is reacted with NaOH solution and Na_2CO_3 solution to produce sodium salt, that is, it is converted to a basic solution. With other words, the reactions that take place are:

$$C_{10}H_8O \text{ (naphthol)} + NaOH \rightarrow C_{10}H_7ONa + H_2O$$

$$C_{10}H_8O + Na_2CO_3 \rightarrow C_{10}H_7ONa + NaHCO_3$$

On the other hand, phenols cannot react with carbonates because they are weaker acids. For this reason, when adding NaHCO3 solution, naphthol remains in its original organic phase.

$$C_{10}H_8O + NaHCO_3 \rightarrow \text{does not occur}$$

By exploiting this particular difference between carboxylic acids and phenols, that is, the ability to react or not with $NaHCO_3$ solution, we can identify whether a substance is a carboxylic acid or a phenol.

The same procedure was repeated, this time with acetic acid. Sodium salt of acetic acid (CH_3COONa) is formed upon addition of NaOH, Na_2CO_3 and NaHCO3 solution to acetic acid. The reactions that occur are:

$$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$$

$$CH_3COOH + NaHCO_3 \rightarrow CH_3COONa + CO_2 + H_2O$$

$$CH_3COOH + Na_2CO_3 \rightarrow CH_3COONa + HCO_3^-$$

Sodium benzoate is a soluble salt. Adding HCl solution to sodium benzoate reconstitutes benzoic acid, which precipitates as a precipitate, and the NaCl salt. The same results are observed when adding an HCl solution to the naphthol salt and sodium ethane. The difference however is in the amount of HCl added to each solution until a precipitate is formed, which depends on the acidic strength of each substance. Most were added to benzoic acid and the least to naphthol.

$$C_6H_5COONa + HCl \rightarrow C_6H_5COOH + NaCl$$

$$C_{10}H_7ONa + HCl \rightarrow C_{10}H_8O + NaCl$$

$$CH_3COONa + HCl \rightarrow CH_3COOH + NaCl$$

Benzoic acid is a stronger acid than ethanoic acid. This is due to the displacement of the single pair of electrons onto the oxygen atom in the benzene. This increases the polarity of the hydroxyl bond (-OH) and makes the H^+ ion cleavage easier. The relocation of electrons into the benzene ring also stabilizes the carboxyl ion. Consequently, the equilibrium position is more to the right, indicating that benzoic acid is a more acidic acid [4].

On the other hand, naphthol, like all phenols, is an aromatic ring compound and has the hydroxyl directly attached to a carbon of the aromatic ring. The excellent stability of the aromatic ring causes a large polarization of the O-H bond of the hydroxyl, thus removing it very easily. Ethanol is therefore more potent than naphthol [5].

To make an amine water-soluble, it has to be converted to a salt, while base needs to be added to form the initial amine again. Upon the addition of the HCl solution to the amine, the corresponding ammonium salt is formed, which is extremely soluble in water. The $NH4^+$ ions, in contrast to the other Group V cations, are hydrolyzed to a great extent and the solutions of the ammonium salts with strong acids are weakly acidic [5]. After the addition of the NaOH solution to the soluble RNH_3Cl salt, a precipitate is formed and the pH in the solution is increased. The increase in pH can be detected either by the coloring it causes on damped pH paper (deep staining of the pH paper by increasing pH).

The reactions that take place are:

$$RNH_2 + HCl \rightarrow RNH_3Cl$$

$$RNH_3Cl + NaOH \rightarrow RNH_2 + NaCl + H_2O$$

Lastly, benzoic acid and benzophenone are separated by extraction. Since benzophenone can further delocalize this positive charge into its phenyl groups, the conjugate acid of benzophenone is the most stabilized of the three acids, leading to benzophenone being the strongest base and does not react with NaOH solution. Benzoic acid, like most organic carboxylic acids (with more than 5 carbons) is not very soluble in water but is soluble in various organic solvents, such as dichloromethane.

However, it can be easily deprotonated with a base to give a charged ionic species that is readily soluble in water. By the addition of NaOH solution the benzoic acid is converted to a sodium salt of benzoic acid, which is soluble in water and therefore moves to the aqueous layer. The organic layer containing the benzophenone is separated from the aqueous layer and is isolated.

To obtain the starting compound, the salt must be protonated with a strong inorganic acid. Once benzoic acid is recovered by the addition of acid, it will precipitate in water to provide a pure compound [3].

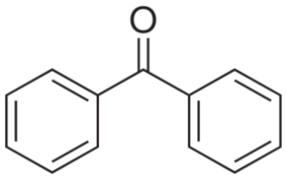

Figure 2: Benzophenone

IV. CONCLUSION

From this study, it is concluded that a compound to be separated and isolated from a mixture, it must be interconverted from non-ionic organic-soluble forms to water-soluble ionic forms. That is, in order to separate benzoic acid and benzophenone, benzoic acid must be converted to salt, and once the two phases are separated, it can be recovered by neutralization.

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