Lab 5 - Extraction Lab

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1 Introduction

Extraction is a technique used to separate components from a mixture of compounds. The goal of this extraction was to separate a mixture of unknown quantities of cinnamic acid and naphthalene. The extraction process uses a volatile organic solvent, such as ethyl acetate, and aqueous acids/bases to exploit the acidic/basic functional groups present on the compounds of the mixture. The solvents are separated into two immiscible layers. The compound extracted from the mixture remains in solution with the layer it is most soluble. The two layers are separated through the use of a separatory funnel and the process is repeated until an acceptable fraction of the extracted compound is separated. Once the extracted compound is ready for recovery, a rotary vaporizer is used to remove the excess solvent. The extraction for this experiment is summarized in *figure* 1.

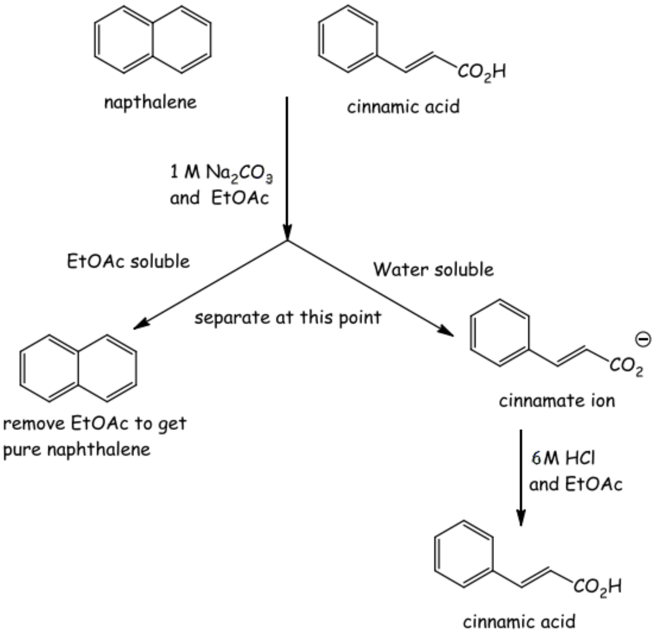

Figure 1: Extraction Summary

2 Materials

3.00g cinnamic acid, naphthalene mixture

6.00g anhydrous sodium sulfate (Na_2SO_4)

55mL ethyl acetate (EtOAc)

$1MNa_2CO_3$ solution (50mL)

6 M HCL (20ml)

Ether (20ml)

Ethanol (20ml)

(50, 150, 250)mL Erlenmeyer flasks

2 Round bottom flasks (50mL)

Ring stand & Ring

Funnel

Separatory funnel

Magnetic stir stick

Rubber stopper

pH paper

3 Procedure

A 3.00g of *mixture C*, having unknown proportions of cinnamic acid and naphthalene, was weighed and added to a 150mL Erlenmeyer flask along with a magnetic stir bar.

20mL ethyl acetate (EtOAc) was added to the flask and stirred to dissolve the compounds.

Once the compounds had dissolved, 50mL $1MNa_2CO_3$ was added to the solution and stirred for 30 minutes. Then poured into a separatory funnel. The erlenmeyer flask was then rinsed with 5mL ethyl acetate which was also added to the separatory funnel.

The pH of the aqueous layer (bottom layer) was checked. The funnel was caped and shaken, The Naphthalene had precipitated from the solution, Ethanol was added until solution formed two separate layers.

Once two layers formed, the aqueous layer was drained from the seperatory funnel into a 250mL Erlenmeyer flask labeled "cinnamate soln." The remaining EtOAc layer was then poured from the top of the separatory funnel into a 50mL erlenmeyer flask. 3.00g anhydrous sodium sulfate (Na_2SO_4) was then added to both $250mL, and 50mL$ flasks to dry. The $50mL$flask was then corked and labeled "EtOAc/naphthalene."

20mL of ethyl acetate and 20mL 6M HCL was added to the 250 mL flask labeled "cinnamate soln." and was stirred using the magnetic stir stick for 20 minutes. The solution was then transferred to the separatory funnel and shaken allowing the layers to separate.

The pH of the aqueous solution was checked. Ether was added to the solution in the separatory funnel to help dissolve the precipitate. H_2O was then added to dissolve any remaining Na_2SO_4. The funnel was shaken and left to settle until two individual layers formed in the separatory funnel.

Once the two layers formed, the aqueous layer was drained into a 250mL Erlenmeyer flask and saved. The remaining EtOAc was poured from the separatory funnel into a 50mL Erlenmeyer flask and was labeled "EtOAc/cinnamic acid.". 3.00g Na_2SO_4 was then added to the flask and it was left to dry for 30 minutes.

2 apparatuses were setup for gravity filtration. The glass funnel was plugged using cotton. Each EtOAc solution was individually filtered using different funnels to remove any Na_2SO_4 and were collected in their own pre-weighed 50mL round bottom flask. Each funnel was then

rinsed with $5mL$ EtOAc that was collected in its corresponding flask. The ethyl acetate was then removed from each flask individually by rotary evaporation. To rotovap flask was clipped onto the vacuum tube and arm of rotovap was tilted until the bottom of the flask was dipped into the $55°C$ water bath. Then Flask was rotated at $150rpm$ until all solvent had evaporated. The flasks were left uncorked in a locker until the next lab period.

Both flasks were weighed individually to determine the amount of cinnamic acid and naphthalene recovered. Acetone was used to clean the round bottom flasks, the saved aqueous layers were poured down the drain, and the erlenmeyer flasks were rinsed with water.

4 Measurements and Calculations

Experimental Data

Mass cinnamate/naphthalene mixture:

$Mixture$ "C"$= 3.0000g$

Mass naphthalene flask:

Initial (empty) weight $= 51.3704g$
Final weight (with naphthalene) $= 52.3917g$

Mass cinnamic acid flask:

Initial (empty) weight $= 52.4037g$
Final weight (with cinnamic acid) $= 52.4387g$

5 Results

Naphthalene recovered $= M_f - M_i = 1.0213g$
Cinnamic acid recovered$= M_f - M_i = 0.0350g$
Percent recovery$= [total\ mass\ recovered/mass\ mixture\ C]*100 = 35.21\%$

6 Conclusion

The objective of this lab was to use the extraction method to separate an unknown mixture of cinnamic acid and naphthalene. This was accomplished by dissolving the mixture in ethyl acetate and adding Na_2CO_3. It was necessary to add ethanol to force the separation of the solvents. Two immiscible layers formed and were separated using a separatory funnel and the EtOAc/naphthalene layer was stored in a $50mL$ flask. Further separation, of cinnamic acid, was accomplished by adding $6MHCL$ and EtOAc to the remaining aqueous layer to form the acid from the cinnamate anion. It was necessary to add ether to the solution to force the separation. The two layers that formed were separated. Finally cinnamic acid was recovered by using a rotary evaporator to remove the EtOAc from the extracted cinnamic acid.

The final yields of the separated mixture were as follows; naphthalene recovered$= 1.0213g$ cinnamic acid recovered$= 0.0350g$ which totaled to a of $1.0563g$ from original $3.0000g$ mixture "C" at the beginning of the experiment and was a recovery of 35.21%.

Conclusion Responses

- A test can be conducted to identifying which is the aqueous or organic layer during an extraction experiment by releasing a few milliliters of the lower layer from the separatory funnel into a test tube and adding an equal volume of water. If the layer is organic it will fall to the bottom of the test tube forming two layers.

- Before removing the lower layer from the separatory funnel, the stopper must be removed. Removing the stopper prevents the build up of a vacuum above the liquid upon draining, which will stop its flow and suck air in through the stem. Removing the stopper allows air to flow into the separatory funnel and take the place of the liquid as it flows out.

- The extraction of an organic compound from a water layer by an organic solvent could be improved by adding an inorganic salt such as NaCl. The addition of an inorganic salt creates a highly ionic solution that an organic solvent, such as diethyl ether, is less soluble in. The increased polarity of the solution forces the separation of the two liquids.