CHEM 0011

Experiment 4 – Introduction to Separation Techniques I

Objectives

1. To learn the gravity filtration technique
2. To learn the suction filtration technique
3. To learn about solvent extraction

Apparatus:

1. Separatory funnel (Instructor demonstration)
2. Vacuum filtration apparatus (Instructor demonstration)
3. Long-stem funnel
4. Whatman fast flow filter paper
5. Funnel rack
6. 400 mL beaker
7. Rubber policeman
8. Water bottle

Mixture:

1. A small beaker containing a sand and water mixture

Solvent:

1. Paint thinner

Chemicals:

1. 0.30 g of sodium sulphate
2. 6 N hydrochloric acid
3. 5% barium chloride solution
4. Iodine crystals
Introduction

There are many methods that one can use to separate one substance from another. The method that one would choose will be dependent on the physical properties of the substances involved.

In this lab, we will focus on two separation techniques:

1. Solvent Extraction
2. Filtration – by Gravity and Vacuum (suction)

Solvent Extraction Technique

Solvent extraction is a very useful process for the separation and isolation of compounds from mixtures. For example, if you want to separate the sand from a mixture of sand and sugar, you would add water to the mixture to dissolve the sugar into the water. In this case, water is used as the extracting solvent. The sand, which remains as a solid, would be separated by filtration.

To carry out a liquid-liquid solvent extraction, that is, the extraction of one or more substances dissolved in a solution using an appropriate extracting solvent, the extracting solvent must be:

- **Immiscible** (*incapable of being mixed to form a homogeneous solution*) with the solvent containing the substance(s). *Note: this term is used to describe two or more liquids.*
- Able to dissolve the substance to be extracted

The apparatus that is needed to carry out a solvent extraction is a separatory funnel. The diagram to the left shows a separatory funnel. Depending on the relative densities of the solution containing the substance and the extracting solvent, the extracting solvent may form a layer above or below the solution containing the substance.

During the extraction process, the separatory funnel is held inverted with the stopper pressed tightly against the palm of the hand. This prevents the content of the separatory funnel from leaking through the stopper. With the valve stem pointing upward, the separatory funnel is shaken several times. After each shaking, the valve is open to release any pressure that may be built up during the shaking.

During this process, the substance that is more soluble in the extracting solvent than in the original solvent passes from the original solvent to the extraction
solvent. The extraction solvent is then drained from the original solvent and fresh extraction solvent is introduced to the original solution. This is repeated until all the substance is extracted from the original solution into the extracting solvent.

**Gravity Filtration Technique**

In gravity filtration, the filtrate passes through the filter medium under the force of gravity and the capillary action between the liquid and the funnel stem.

There are several varieties of filter paper. Good filtration depends on the retention of the filter paper and the speed of the filter paper. Usually, fast papers will retain coarse particles. Slow papers will retain fine particles. The optimum choice is a paper, which is as fast as possible, yet retains all visible particles, and thus giving a clear filtrate.

Low-ash or ashless quantitative-grade papers can be ignited without leaving an ash. The residue left by an 11-cm circle of a low-ash paper may be as low as 0.06 mg. An ashless-grade paper typically leaves 0.05 mg or less from an 11-cm circle. This small mass is considered negligible in most analytical procedures.

**Decantation** is a process used to separate the liquid from the mixture to be filtered. To decant a liquid from a solid, in one hand hold the beaker that has the mixture in it. Hold a glass-stirring rod in the other hand. Touch the lip of the beaker to the glass rod and pour the mixture to be filtered using the glass rod as a guide to pour slowly to ensure that the solid is not carried along. This also prevents the liquid from running back along the outside of the beaker.

**Washing** of the solid to remove soluble impurities follows the decantation of the supernatant liquid. Use a small amount of wash liquid and mix it thoroughly with the solid. Allow the solid to settle and decant the wash liquid through the filter. Repeat this procedure several times. Several washings with small volumes of liquid are more effective in removing soluble contaminants.

**Transfer the washed solid** in the filter funnel is the final step. The bulk of the solid is transferred to the funnel by squirting a stream of wash liquid from a wash bottle. The last traces of the solid are removed from the walls of the beaker by scrubbing with a rubber policeman. Rinse the beaker and rubber policeman and transfer the rinse liquid to the funnel. Repeat this at least two times.
Step to prepare a gravity filtration

1. It is important to use the correct size filter paper. Properly sized filter paper should stop just below the rim of the glass funnel. As a guide, use filter paper whose diameter is about 1 cm less than twice the diameter of the funnel, for example a six-centimeter diameter funnel uses a filter paper of eleven-centimeter diameter. The filter paper should sit a few millimeters from the rim of the funnel.

   ![Filter paper diagrams](image)

   - Correct size of filter paper is (c)

2. Fold the filter paper as shown below.

   ![Folding diagrams](image)

   - Folding a filter paper

   (a) Fold the filter paper in half.
   (b) Fold the filter again to within about 10° of a 90° fold. The second fold is not exactly at a right angle. Tear off the corner of top fold.
   (c) Open the filter paper so that the torn corner is on the outside of the cone. The tear enables the paper to stick better to the funnel.

3. Place the folded filter paper snugly into the funnel by moistening the filter paper with the solvent of the mixture to be filtered. This should resemble the figure shown in step 1(c).
4. Press the filter paper against the top wall of the funnel to form a seal. Support the funnel with a funnel rack.

5. Set up the gravity filtration apparatus as shown in the diagram on the left. Ensure that the funnel rack is positioned so that the funnel stem is inside the beaker. Position the beaker so that the funnel stem is touching the side of the beaker to avoid splashing.

6. Allow the mixture to settle and then **decant** the liquid from the solid. **Wash** the solid which remains in the beaker several times. Finally, **transfer the washed solid** to the funnel.

**Vacuum filtration (or suction filtration)**

This type of filtration is used with water or high-boiling organic solvents and is much faster than gravity filtration. For the set-up, a filter flask must be clamped in position before attaching the rubber tubing, rubber ring (adapter) and Büchner funnel. This prevents the top-heavy apparatus from toppling over and spilling material. A medium- or slow-speed filter paper is used that is wetted with the solvent before the vacuum is applied with the water aspirator. Use a large beaker under the water aspirator to minimize splashing. Check that there is a good seal between the apparatus when vacuum is applied before filtering the sample.

In some experiments, the Büchner funnel is replaced with a Gooch crucible or fritted glass funnel depending on the particle size needed to be collected or if the filtered solid needs to be heated to dryness.
During the filtration, the mixture should be poured at a rate that the bottom of the funnel is covered with some solution. The collected crystals can be washed with some chilled solvent. Do not discard the mother liquor (in the filter flask) as more compound could be recovered.

**Caution:**
Water can be sucked back into the filtration apparatus if the water pressure decreases. Be sure to break the vacuum by disconnecting the tubing at the aspirator.

Suction filtration is used to isolate solids formed from chemical reactions. A reaction that forms solids from two aqueous solutions is a precipitation reaction. A general example of this reaction is: 

\[
AB_{(aq)} + CD_{(aq)} \rightarrow AD_{(s)} + CB_{(aq)}
\]

The subscript (aq) represent a compound that is soluble in aqueous (water is the solvent) solution. The subscript (s) represents a compound that is insoluble in a solvent.

One example is shown below as a word equation and the balanced chemical equation.

silver nitrate + sodium chloride forms silver chloride + sodium nitrate  {word equation}

\[
AgNO_3(aq) + NaCl(aq) \rightarrow AgCl(s) + NaNO_3(aq)
\]  {chemical equation}

This is also known as a double displacement reaction because two chemical species from two different compounds exchange places to form two new compounds.
Part A – Demonstration of Extracting Iodine Dissolved Water

Procedure:

1. Dissolve one crystal of iodine in about 100 mL of water in an Erlenmeyer flask.
2. Stopper the Erlenmeyer flask and shake the content.

| Does the iodine appear to dissolve easily in water? ________________ |
| What colour is the solution? ____________________________________ |

3. Transfer the iodine/water solution to a separatory funnel. Do not pour in the undissolved iodine.
4. Remove the stopper and using a small beaker, pour about 3 cm. of paint thinner slowly into the separatory funnel.

| Describe where is the paint thinner relative to the iodine/water solution. Which solvent (water or paint thinner) is on top? |
|___________________________________________________________________________|
|___________________________________________________________________________|
|___________________________________________________________________________|
5. Put the stopper on the test tube again and shake the contents.

Describe what happens when the content is shaken. _______________________
________________________________________________________________
________________________________________________________________
This top layer contains __________________________ which has _________________ dissolved in it.

6. Put the “waste beaker” under the separatory funnel, remove the stopper at the top and slowly let the bottom (water) layer out. Close the stopcock just as the last of the bottom layer goes through. You should now have only the top layer (pink) in the separatory funnel.
Part B – Demonstration of Vacuum (Suction) Filtration

Procedure:

1. The apparatus for vacuum filtration will be set-up.
2. A precipitate of barium sulfate (BaSO₄) will be made by mixing an acidic solution of sodium sulfate (Na₂SO₄) with barium chloride (BaCl₂). This is called a precipitation reaction or a double displacement reaction.

   Describe the solution of sodium sulfate ______________________________
   ____________________________________________________________________

   Describe the solution of barium chloride ______________________________
   ____________________________________________________________________

   Record your observations when the two solutions are mixed ______________
   ____________________________________________________________________
   ____________________________________________________________________

   How do you know a chemical reaction occurred? _______________________
   ____________________________________________________________________

   The chemical reaction in this demonstration is:
   Na₂SO₄(aq) + BaCl₂(aq) → BaSO₄(s) + NaCl(aq)

   Write the word equation for this reaction:

3. Filter the mixture of barium sulfate with the suction apparatus. The mixture is filtered using decanting. Rinsing with a wash bottle helps to quantitatively transfer the remaining solid in the beaker.
Part C – Gravity Filtration

Procedure

1. Set up the gravity filtration apparatus.
2. Prepare the filter paper and wet it down with distilled water.
3. Obtain a beaker of sand and water mixture.
4. Using proper gravity filtration technique, separate the sand from water.

Questions:

1. Are water and paint thinner miscible or immiscible?

2. Will solvent extraction work if the two solvents are miscible? Why or why not?

3. Do you think solvent extraction could effectively remove salt from salt water? Why or why not?