The Separation of a Mixture into Pure Substances

The experiment is designed to familiarize you with some standard chemical techniques and to encourage careful work in separating and weighing chemicals.

In this experiment you will separate a mixture of three substances, sodium chloride (NaCl), benzoic acid (C₆H₅COOH), and silicon dioxide (SiO₂), into pure substances based on their solubility in water. The amount of a substance that will dissolve in water depends on the nature of the substance and on the temperature. Water is a polar substance so other polar substances will dissolve in water - ("Likes dissolve Likes"). Sodium chloride is an ionic compound that is quite soluble in water. Silicon dioxide (sand) is a large macromolecule that is essentially insoluble in water. Benzoic acid is polar, but is much less polar than water so it dissolves in water only to a limited extent. The solubility of these three substances in water is given below. The solubility of each is expressed in terms of grams of the solid that dissolves in 100 g of water. It is evident that each of these has a different solubility/temperature relationship.

**Solubility (g/100g H₂O) as a Function of Temperature**

<table>
<thead>
<tr>
<th>Substance</th>
<th>0</th>
<th>20</th>
<th>40</th>
<th>60</th>
<th>80</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaCl</td>
<td>36.00</td>
<td>36.00</td>
<td>37.00</td>
<td>37.00</td>
<td>38.00</td>
<td>40.00</td>
</tr>
<tr>
<td>C₆H₅COOH</td>
<td>0.17</td>
<td>0.29</td>
<td>0.42</td>
<td>1.20</td>
<td>2.80</td>
<td>7.00</td>
</tr>
<tr>
<td>SiO₂</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

In this experiment you will use decantation and two types of filtration. Decantation is a process of separating a liquid (called supernatant) from a solid residue by gently pouring off the liquid from the solid. It is easier to decant when you pour the liquid down a stirring rod as shown in the procedure section. Filtration separates a solid and a liquid using a porous material such as filter paper which allows the liquid to pass but retains the solid. Gravity filtration uses the force of gravity to achieve separation. Vacuum filtration uses the suction created by an aspirator to speed up the separation.

**Materials List:** 250 mL beakers mixture wire screen ring stand
Burner stirring rod ice bath drying oven
Buchner funnel boiling chips spatula watch glass

**Procedure:**

1. Carefully weigh a clean dry 250-ml beaker (beaker 1) to the nearest .001 gram. Using a spatula, transfer about 5 to 6 grams of the mixture, which contains sodium chloride, benzoic acid, and silicon dioxide into beaker 1. Be sure to record which unknown you have. Weigh beaker 1 containing the mixture to the nearest 0.001 g. From the two weights, obtain the weight of the mixture. Record your results on the data
2. Add about 50 ml of distilled water to beaker. Place beaker 1 with its contents on a wire screen, which is resting on an iron ring connected to a ring stand. See the diagram below:

3. Heat the mixture to the boiling point (stop heating when boiling is observed), and stir the mixture with a stirring rod to make sure that all soluble material is dissolved. At the boiling point temperature, all benzoic acid and sodium chloride should be in solution. Thus, they have been separated from sand. (This is an extraction process.) Take about 15 ml of distilled water in a beaker and bring it to a boil. This boiling water will be used in step 5.

4. Decant the liquid while it is hot into another 250 ml beaker (beaker 2). Use Hot Hands to hold the hot beaker. Do not let the sand get into beaker 2 or wait too long before decanting. Use a glass rod to aid the decantation process. See figure below:

5. To dissolve as much benzoic acid and sodium chloride as possible, wash the sand in beaker 1 with about 10 ml or less of boiling water and decant the washing into beaker 2.
6. Place beaker 2 in an ice bath to let it cool. Observe carefully how the benzoic acid crystallizes out of the solution. Set beaker 2 aside to ensure maximum crystallization.

7. Place your beaker of sand in the drying oven so that it can dry completely before weighing. Make sure you mark the beaker with a grease pencil so you can identify it with your initials or some other mark. Leave the beaker in the oven until you have finished the rest of the lab to insure it is fully dried. When you take it out of the oven let it cool, and weigh. You should move on to the next step and come back to weigh the beaker at the end of the experiment.

8. Assemble the Buchner funnel with a filter flask. See the figure below.

9. Connect a piece of vacuum rubber tubing from the water aspirator to the filter flask. Vacuum rubber tubing is used so that it will not collapse when the pressure is reduced.

10. Place the proper size of filter paper in the Buchner funnel and wet it with distilled water using a wash bottle. This allows the filter paper to sit down on the grating. Make sure to weigh your filter paper before doing so.

11. Turn on the water aspirator. Make sure to turn it on just enough to have proper suction occurring.

12. Pour the contents of beaker 2 (benzoic acid crystals) into the Buchner funnel.

13. Wash the solid (benzoic acid) with about 5ml or less of distilled ice water to ensure that the benzoic acid is free of sodium chloride.
14. Continue to suction until the liquid no longer drips from the funnel.

15. Disconnect the rubber tubing from the filter flask before turning off the water aspirator. This prevents the water from backing up into the filter flask. The filtrate (the liquid collected) is often the desired material.

16. Using a spatula transfer the suction dried benzoic acid onto a pre-weighed marked watch glass. Place the filter paper and watch glass into the drying oven for a few minutes to dry. When dry, take it out and let it cool before weighing.

17. To a large beaker add three or four pieces of black boiling chips. Then weigh the beaker to the nearest 0.001g.

18. Transfer the filtrate (liquid) from the filter flask into the large beaker you just weighed in #17. Using a wash bottle rinse the filter flask with as little water as possible. Add the washings to the beaker. Place the beaker (which now contains sodium chloride and water) on a wire screen, which is resting on a iron ring. Start to heat the solution gently with a burner so that the liquid does not boil over. Later, reduce the flame to avoid overheating, which may cause splattering of solid sodium chloride or shatter your beaker. When the water is evaporated entirely, let the sodium chloride dry, and the beaker cool before weighing. Weigh the beaker and its contents to the nearest .001 g and calculate the weight of sodium chloride.

19. Calculate the percentage of each substance in the mixture by using the following formula:

\[ \% \text{component} = \frac{\text{grams of component (salt or benzoic acid or sand)}}{\text{Total grams of mixture}} \times 100 \]
REPORT SHEET: SEPARATION OF A MIXTURE

A. Record Identity of unknown

1. Weight of beaker 1
2. Weight of beaker 1 and mixture
3. Weight of mixture
4. Weight of beaker 1 and dry sand
5. Weight of dry sand
6. Weight of filter paper
7. Weight of watch glass
8. Weight of watch glass, filter paper, and benzoic acid.
9. Weight of benzoic acid
10. Weight of large beaker and boiling chips
11. Weight of beaker, boiling chips and sodium chloride
12. Weight of sodium chloride
13. Percentage of sand in mixture
14. Percentage of benzoic acid in mixture
15. Percentage of sodium chloride
16. Total percentage of sample recovered

QUESTIONS:
1. Based on the data provided in the introduction, graph the solubility of benzoic acid as it changes with temperature.

2. From your graph, determine the number of grams of benzoic acid that will dissolve in 100 grams of water at 25 degrees C.

3. If a solution is made up by mixing 6.0 g of benzoic acid in 1.00 L of water at 42 degrees C, would the solution be saturated? Would you expect to see solid on the bottom of the container?

4. From an 11.65 g sample containing sodium chloride, benzoic acid, and silicon dioxide, 3.64 g of NaCl, 1.56 g of C₆H₅COOH and 5.92 g of SiO₂, were recovered. Calculate the percentage of sodium chloride in the sample.