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#16 Separation Science Lab

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Introduction

Description

In this inquiry-based experiment, the students are put in the role of a method development chemist. The scenario they are given is that a train wreck has occurred resulting in a chemical spill. They are told that the spill mixture consists of three substances: a proprietary chemical called CARBOSE (simply sodium chloride), naphthalene, and sand from the dry Florida riverbed where the accident took place. The students work in teams of 2-3 and the team's task is to design a method to separate the three components of the mixture in the most efficient, cost-effective manner possible. In addition, their method must allow them to recover the CARBOSE in the solid form. The lab is divided into three parts and should be spread out over two lab periods to allow students time to collect data and reflect on questions. During parts 1 and 2 of the lab, the students will gather qualitative and quantitative data regarding the solubility of the three substances in three different solvents: methanol, cyclohexane (or mineral spirits), and water. This data is used to design a separation scheme. This separation scheme is then tested on a model sample of the mixture during part 3 of the experiment. A final report is prepared by the students in which they outline the separation method they developed, discuss the effectiveness of their method, provide suggestions for modifications which could improve the separation, and answer trouble-shooting questions.

Student Audience

This lab is suitable for the first semester of an introductory level or general chemistry course or even a high school chemistry course. The amount of instructor guidance and pre-lab discussion can be varied to match the prior knowledge and laboratory skill of the students.

Goals

In this experiment the student will

- become familiar with the simple laboratory techniques of filtration and evaporation used by chemists to separate substances in a mixture.
- improve their interpersonal skills as they work cooperatively with other students to solve a problem.
- gain an understanding of how differences in solubility can be used to separate the components of a mixture.
- work on their written and verbal communication skills.

Recommended Placement in the Curriculum

First semester of an introductory or general chemistry course.

Student Handout

Separation Science Lab

Problem Scenario:

You are employed by a hazardous waste disposal company that has been hired to clean up a chemical spill resulting from a train derailment. The derailment occurred in Florida over a dry, sandy riverbed. During the cleanup of the accident, roughly a quarter of a ton of naphthalene, a toxic chemical, was combined with a ton of CARBOSE, a non-toxic substance being developed by a defense contractor. A significant quantity of sand was also collected in the removal of the hazardous mixture from the derailment site. Since CARBOSE is proprietary (patent pending), the defense contractor would like it back, separated from the other components of the spill mixture. The remaining components of the spill should be disposed of in the most cost-effective and safe manner. Sanitary landfills will not accept waste containing hazardous chemicals. Hazardous waste landfills, however, will accept waste containing both non-hazardous and hazardous chemicals. Costs for waste disposal at sites near you are as follows:

Sanitary landfill - $35/ton of material
Hazardous waste landfill - $100/55 gallon drum

You are the chemist assigned to design a cost effective procedure for handling the CARBOSE separation and recovery and hazardous waste disposal. Here are the three criteria your scheme should meet:

1) The CARBOSE must be returned to the defense contractor in the SOLID form.

2) The process should be the most cost effective one possible.

3) Quantities of materials used in the separation process should be kept to a minimum.

A colleague of yours suggested that you consider using the relative solubilities of the three components in some common, inexpensive solvents as a means of separating the waste materials.

Materials and Chemicals Needed

1. Samples of test solvents: water, cyclohexane, and methanol
2. Pure samples of the three substances in the spill mixture: sand, naphthalene, and CARBOSE
3. 9-10 small test tubes (10 mL) and test tube rack
4. wax pencil or labels for identifying test solutions
5. Stirring rods
6. Small spatulas
7. Three 10-mL graduated cylinders (one for each solvent)
8. Two 100-mL beakers
9. Other equipment will depend on the procedure you develop

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Safety, Handling, and Disposal

Methanol and cyclohexane are flammable, so keep away from flames. In addition, these two chemicals, along with naphthalene, are respiratory hazards and skin irritants and should be handled appropriately. Wear goggles. Read MSDSs for all chemicals.

Dispose of all waste materials in the labeled containers located in the lab hoods. DO NOT DUMP ANY CHEMICALS DOWN THE DRAIN. The waste in the containers will be disposed of according to local ordinances.

Procedure

Since this is an inquiry-based lab, the directions are not as specific as those you may be used to in other labs.

Part 1-Qualitative Investigation of Solubility

Following your colleague's advice, you have decided to determine the solubility of sand, naphthalene, and CARBOSE in each of three common solvents: water, methanol, and cyclohexane. A standard microscale procedure recommends using less than 0.15 gram of solid solute and less than 3 mL of solvent for the solubility test. The procedure contains a cautionary statement that some solids may take longer than others to dissolve and may require some agitation (stirring or shaking) for complete dissolution. In addition, some materials may exhibit only partial solubility.

Conduct your analysis of the solubility of the three solutes in each of the three solvents and record your observations in a data table.

Based on your observations, answer the following questions.

1. a. Suppose you want to separate the CARBOSE from a CARBOSE/sand mixture. What solvent would you recommend to dissolve only one of the two solids?

b. Will that same solvent be useful in separating a naphthalene and CARBOSE mixture? Why or why not?

2. a. Based on your observations, what solvent would allow you to separate a sand/naphthalene mixture?

b. Would that same solvent be useful in separating a naphthalene and CARBOSE mixture? Why or why not?

3. Once you have dissolved a component of a mixture, you will have an undissolved solid and a liquid composed of solvent and dissolved solute. What experimental technique can be used to separate the solid from the liquid phase?

4. After the solid phase has been removed from the liquid solution, how will you separate the solvent from the dissolved solute?

5. Consider your answers to the above questions and then briefly outline the steps you propose using to separate and dispose of the components of the mixture from the train wreck. Remember, this is initially a solid mixture of sand, naphthalene, and CARBOSE. Compare your separation scheme with the schemes of other students in the class. Modify your plan, if necessary, before proceeding to Part 2 of the experiment.

Part 2-Quantitating Solubility

At this point you have a basic plan for separating the substances from each other, but how much solvent is needed to dissolve each solute? Remember, your company does not get paid for cleaning up its own waste! You should determine the smallest quantity of solvent needed to dissolve a known quantity of solute. One approach to finding this relationship is to take a measured quantity of solid solute (about 0.5 gram) and add solvent in 1 mL increments until all of the solute dissolves. This should allow you to get a fairly good estimate of the solubility of the solute in that particular solvent system (# grams of solute/1 mL of solvent).

6. Which solute/solvent mixtures do you need to evaluate? It does not make sense to determine the solubility of a solute which is INSOLUBLE (or relatively insoluble) in a certain solvent.

Conduct your experiment and record your results in a data table.

You should now have enough information to design a procedure which will allow you to take a sample of the spill mixture and separate it into the three separate components. A model sample of the spill mixture will be available for you to evaluate the effectiveness of your method. This sample will have a total mass of about 5.0 grams and contain 20% sand, 70% CARBOSE, and 10% naphthalene by mass.

7. Based on the information above and your results from Parts 1 and 2, outline a specific separation scheme for the model sample mixture. This should involve merely using your procedure from question # 5, but modifying it to include specific quantities of solvents you will use. Be specific with your instructions since a fellow employee will be performing the actual separation of the wreck mixture-your job is method development.

Part 3-Evaluation of Separation Scheme

Test the procedure you have developed by performing the separation of a 5.0 gram sample of the model mixture. Collect the recovered CARBOSE and use this value to calculate the % by mass of CARBOSE in the sample mixture.

8. How does your % CARBOSE compare to the known value for the model mixture of 70%?

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Final Report

Prepare a report to your supervisor (the instructor) which include the following information:

- Data tables summarizing the observations and results from Parts 1 and 2 of the experiment.
- An outline of the separation scheme used to isolate CARBOSE from the model spill sample.
- Results from the trial run using the model sample.
- A brief summary of the overall effectiveness of your procedure.
- Suggestions for modifications to the procedure which could improve the recovery of CARBOSE.
- Answers to Trouble-Shooting Questions.

Trouble-Shooting Questions

I. Your procedure has been accepted and is currently being used to clean up the spill. You get an urgent message from the lab technician who is performing the analysis who states that he grabbed the wrong solvent bottles (tsk, tsk, not reading labels) and accidentally added the solvents in the wrong order. He wants to know how this error will affect the separation. What would you tell him?

II. Your supervisor says that while your scheme is effective as is, she would like you to modify it and choose a cheaper, less hazardous solvent to replace cyclohexane. What solvent could be used in place of cyclohexane and not significantly affect the separation? Use the CRC handbook of Chemistry and Physics and Material Safety Data Sheets to help you answer this question.

Instructor Notes

Separation Science Lab

Time Required

Approximately two 3-hour lab periods. This includes pre-lab instructions, but the students may need to work on their separation scheme design outside of lab time. Parts 1 and 2 can be done during the first lab period; Part 3 in the second lab period.

Group Size

Students should work in teams of two or three. The number of students working in the lab will depend on your lab facilities (20-24 students).

Materials Needed

per student station:

<u>Chemicals:</u>

- solvents-cyclohexane, water, methanol; 40-50 mL in wash bottles
- solutes-2 grams each of sand (fine), naphthalene, and sodium chloride (CARBOSE) in separate containers
- model sample mixture-a 5-6 gram sample of the model mixture (20% by mass sand, 10% naphthalene, and 70% NaCl)

<u>Equipment:</u>

- 9-10 small test tubes (10 mL)
- marking pencils or labels
- stirring rods
- spatulas
- three 10-mL graduated cylinders
- two 100-mL beakers
- filtration equipment (gravity or vacuum)
- evaporating dishes
- tongs
- hot plate or Bunsen burner
- balances which can measure to the nearest 0.1 or 0.01 gram

Safety, Handling, and Disposal

Set up waste containers in the lab hood: methanol-CARBOSE-naphthalene-sand
cyclohexane-CARBOSE-naphthalene-sand
water - naphthalene

The aqueous salt (CARBOSE) and sand solutions can go down the drain. Dispose of waste according to local ordinances.

Students are cautioned in the lab handout about the flammability and health hazards associated with methanol, cyclohexane and naphthalene, but it is a good idea to reinforce these cautions in class during pre-lab discussion.

Points to Cover in the Pre-Lab Discussion

Part 1-The lab handout suggests solubility as a property suitable for separating the components in the spill mixture. You may need to cover certain terminology (solute, solvent, solubility) and separation techniques (evaporation, filtration) if these topics have not already been discussed in other labs or in lecture. Students are given basic instructions as to how to test relative solubility using small quantities of chemicals. The lab also instructs the students that time and amount of agitation are important variables to consider. You should stress this. Depending on the skill level of the students, you may wish to demonstrate the appropriate technique for testing solubility. Make sure to reinforce the hazards associated with the chemicals and proper disposal of waste solutions.

For students unfamiliar with flow diagrams, you may wish to illustrate this method of mapping out a procedure with a hypothetical mixture.

Part 2-In this part of the experiment the students are quantifying the solubility of the solutes in the various solvents. You may wish to show a sample set of data and calculation of solubility (g solute/mL). You can also illustrate how this solubility value can be used to estimate the minimum amount of solvent which would be needed to dissolve a certain amount of solute.

After completion of Parts 1 and 2 of the experiment the students will be asked to develop a procedure for separating the three components in a model sample of the spill mixture. They will be given the % by mass of each component in the model sample so that they can use their solubility values to estimate the amount of each solvent to use. If there is not sufficient time during lab to complete this step, student groups should arrange to meet outside of class to finish their procedure. The procedure should be written and ready for implementation by the next lab period.

Part 3-During pre-lab you should explain what type of a final report you want from each student (or student group). If students are unfamiliar with filtration and evaporation techniques, this is the time to demonstrate them. Time should be given for student groups to confer with other groups and instructor to work out any major errors in their procedures PRIOR to running the experiment.

Procedural Tips and Suggestions

- Mineral spirits may be substituted for cyclohexane with similar results.
- Students may need time outside of lab to organize and write their final written lab reports.
- Collecting student answers to questions 1-8 is optional. The purpose of these questions is to guide the students in their design of an effective separation scheme.
- NaCl (CARBOSE) is partially soluble in methanol, but it is possible that students may miss this if they do not agitate the solution thoroughly and wait several minutes.

Sample Results

Table 1: Results from Part 1 of the Experiment-Solubility Tests*			
Solute/Solvent	Water	Cyclohexane	Methanol
NaCl (CARBOSE)	immediately dissolves	insoluble	partially soluble, slow process
Naphthalene	insoluble	completely dissolved, slow process	completely dissolved, very slow process
Sand	insoluble	insoluble	insoluble

Results from Part 2 of the Experiment—Quantifying Solubility				
	Mass of Solute (g)	Volume of Cyclohexane (mL)	Volume of Water (mL)	Volume of Methanol (mL)
Naphthalene	0.5g	4– 6 mL with 3– 5 minutes of agitation **Solubility = 0.083 – 0.13 g/mL**	Not applicable since naphthalene is insoluble in water.	16– 17 mL with 5– 10 minutes of agitation **Solubility = 0.03 g/mL**
NaCl (CARBOSE)	0.5g	Not applicable	2 mL, immediate **Solubility = 0.25 g/mL**	Not tested. Partial solubility may not be observed. Water is much better solvent.

Results from Part 3

Students typically recover 3.0-3.3 grams of NaCl. This corresponds to 60-66% CARBOSE by mass in the model sample. The students can compare this to the true value of 70% CARBOSE (3.5 g). This correlates to an 85-95% recovery of CARBOSE.

Plausible Answers to Student Questions

1.a. *Suppose you want to separate the CARBOSE from a CARBOSE/sand mixture. What solvent would you recommend to dissolve only one of the two solids?*
Water

b. *Will that same solvent be useful in separating a naphthalene and CARBOSE mixture? Why or why not?*
Yes. CARBOSE is soluble in water while naphthalene is not. The CARBOSE will dissolve in the liquid solvent and the naphthalene will remain in the solid phase.

2.a. *Based on your observations, what solvent would allow you to separate a sand/naphthalene mixture?*
Either cyclohexane or methanol would work although methanol will require a larger volume and longer agitation times.

b. Would that same solvent be useful in separating a naphthalene and CARBOSE mixture? Why or why not?
If the answer to 2a is cyclohexane; yes, because CARBOSE is insoluble in cyclohexane.
If the answer to 2a is methanol; the answer at this point will depend on the solubility results. If they observed that CARBOSE was insoluble in methanol, their answer will be yes.
If they observed the partial solubility of the CARBOSE in methanol, the answer is no.

3. *Once you have dissolved a component of a mixture, you will have an undissolved solid and a liquid composed of solvent and dissolved solute. What experimental technique can be used to separate the solid from the liquid phase?*
   Filtration

4. *After the solid phase has been removed from the liquid solution, how will you separate the solvent from the dissolved solute?*
   Evaporation

5. *Consider your answers to the above questions and then briefly outline the steps you propose using to separate and dispose of the components of the mixture from the train wreck. Remember, this is initially a solid mixture of sand, naphthalene, and CARBOSE. Compare your separation scheme with the schemes of other students in the class. Modify your plan, if necessary, before proceeding to Part 2 of the experiment.*
   1. Sand/naphthalene/CARBOSE mixture + water ——> solid (sand/naphthalene) + aqueous CARBOSE solution. Filter to separate the two phases.
   2. Aqueous CARBOSE solution + heat ———> solid CARBOSE.
   3. Solid naphthalene/sand + cyclohexane ———> solid sand + naphthalene/cyclohexane solution. Filter to separate.
   4. Send sand to sanitary landfill. Try to recover the cyclohexane and naphthalene or send naphthalene/cyclohexane solution to hazardous waste landfill. Return CARBOSE to contractor.

   <u>Alternate Plan</u>
   1. Sand/naphthalene/CARBOSE mixture + cyclohexane ——> solid sand/CARBOSE + naphthalene/cyclohexane solution. Filter to separate.
   2. Solid sand/CARBOSE + water ————> solid sand + aqueous CARBOSE solution. Filter to separate.
   3. Aqueous CARBOSE solution + heat ———> solid CARBOSE.
   4. Send sand to sanitary landfill. Send naphthalene/cyclohexane solution to hazardous waste landfill. Return CARBOSE to contractor.

Students who did NOT observe any partial solubility of CARBOSE in methanol may propose using methanol instead of cyclohexane. This procedure will work but may result in decreased recovery of CARBOSE.

6. *Which solute/solvent mixtures do you need to evaluate? It does not make sense to determine the solubility of a solute which is INSOLUBLE (or relatively insoluble) in a certain solvent.*
   The answer to this question should match their proposed plan. Some students may decide to evaluate the solubility of naphthalene and CARBOSE in methanol even though their scheme doesn't require that solvent. Although this is not necessary, it won't hurt. In fact, the students who do investigate the solubility of naphthalene in methanol may revise their scheme once they see that the solubility is much lower than that of naphthalene in cyclohexane.

7. *Based on the information above and your results from Parts 1 and 2, outline a specific separation scheme for the model sample mixture. This should involve merely using your procedure from question # 5, but modifying it to include specific quantities of solvents you will use. Be specific with your instructions since a fellow employee will be performing the actual separation of the wreck mixture-your job is method development.*
   Answers here will be similar to those in # 5 except that solvent amounts should be calculated based on the % by mass data given for the model spill mixture.

8. *How does your % CARBOSE compare to the known value for the model mixture of 70%?*
   Answers will depend on their results.

Trouble-Shooting Questions

I. *Your procedure has been accepted and is currently being used to clean up the spill. You get an urgent message from the lab technician who is performing the analysis who states that he grabbed the wrong solvent bottles (tsk, tsk, not reading labels) and accidentally added the solvents in the wrong order. He wants to know how this error will affect the separation. What would you tell him?*
   It should not matter in which order the solvents are added since the naphthalene is only soluble in the cyclohexane and the CARBOSE is only soluble in water.

II. *Your supervisor says that while your scheme is effective as is, she would like you to modify it and choose a cheaper, less hazardous solvent to replace cyclohexane. What solvent could be used in place of cyclohexane and not significantly effect the separation? Use the CRC handbook of Chemistry and Physics and Material Safety Data Sheets to help you answer this question.*
   Students should be encouraged to look up naphthalene and see what other solvents it is soluble in that are similar in polarity to cyclohexane (petroleum ether, mineral spirits, xylene, and other nonpolar solvents).

Evaluation of Experiment-Written Lab Report

Student groups will turn in a report which summarizes the solubility data, describes the procedure they developed, and gives the results of the model mixture separation. (See student handout for more specific criteria.) The report should include comments on how effective they thought their procedure was, and suggestions on how it could be improved. The report should also include the answers to the Trouble-Shooting questions. If a formal lab report is required, you will need to allow the students time outside of class to complete them. Be creative! Remember the original scenario-they are a method development team and you are their supervisor.

Extensions and Variations

For most students, this type of lab will be a bit frustrating since it is more open-ended than what they are used to. The questions placed throughout the lab are designed to lead the students' thinking, but they may need some extra guidance from the instructor. If this lab is being used in an introductory chemistry course or in a high school chemistry class, the instructor may want to modify the student handout to include more specific directions and even tables for students to record their observations and data. (See sample answers for possible table formats.) Too much information, however, and the "inquiry" aspect of the lab is lost. It is also not necessary to require a formal lab report.

In addition to recovering the CARBOSE from the sample and using the % recovered as a way to determine the effectiveness of their separation scheme, the students could also evaporate the cyclohexane and recover the naphthalene. Determining the melting point of the naphthalene would indicate its relative purity and provide further evidence to support the effectiveness (or ineffectiveness) of the separation procedure.

Another variation is to have student teams trade separation schemes and let other student teams be the technicians assigned to test the procedures using the model sample mixture. Written feedback from the "technician" team could point out errors in the procedure or highlight places where the directions are confusing.

References

There are no specific references for this particular lab. Almost every lab manual has an experiment involving the separation of a mixture and this lab is merely an extension of that idea.