Recristalização

Purificação de ácido benzóico

Introdução

- ***Purificação***
  - *separação física de contaminantes de uma amostra para a obtenção do composto puro desejado.*
  - *remoção de sub-produtos e impurezas de uma amostra.*
  - *líquidos: destilação*
  - *sólidos: recristalização*

Introduction

- ***Recristalização***
  - *sólidos orgânicos*
  - *dissolução do sólido a temperaturas elevadas em solvente apropriado e recristalização por abaixamento da temperatura*
  - *impurezas: (1) mais solúveis do que o componente principal (2) menos solúveis do que o componente principal*

Processo

- *Dissolução do sólido impuro a quente*
- *Cristalização por resfriamento da solução*
- *Pureza do sólido formado deve-se à seleção das partículas para a formação do retículo cristalino*
- *Depende da diferenmça de solubilidade das espécies envolvidas*

Solvente para Recristalização

- ***Na situação ideal...***
  - *o composto-alvo é completamente solúvel no solvente em temperatura próxima à de ebulição e totalmente insolúvel à temperatura ambiente ou a 0°C.*
  - *e o contrário para a impureza*

Recrystallization Solvent

- **No mundo real...**

  - *isso nunca acontece e esse procedimento é tanto mais eficiente quanto mais praticado*

  - *a pureza do sólido pode ser avaliada por:*
    - *cromatografia*
    - *ponto de fusão*

Solventes comuns

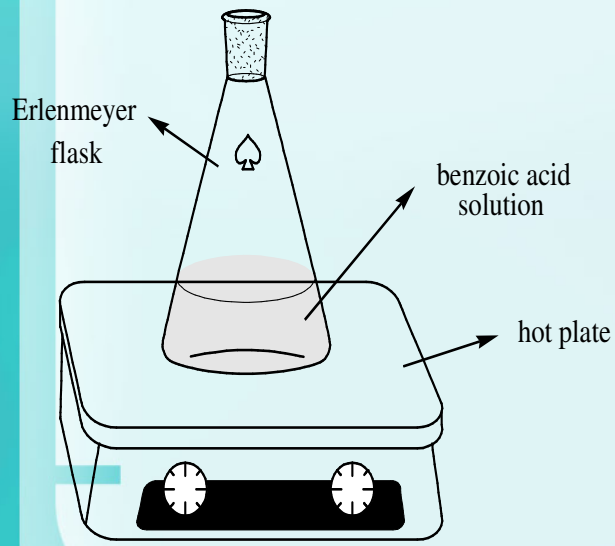
solvent	formula	polarity	boiling point (^0C)
water	H_2O	very polar	100
ethanol	CH_3CH_2OH	polar	78
methanol	CH_3OH	polar	65
dichloromethane	CH_2Cl_2	slightly polar	40
diethyl ether	$(CH_3CH_2)_2O$	slightly polar	35

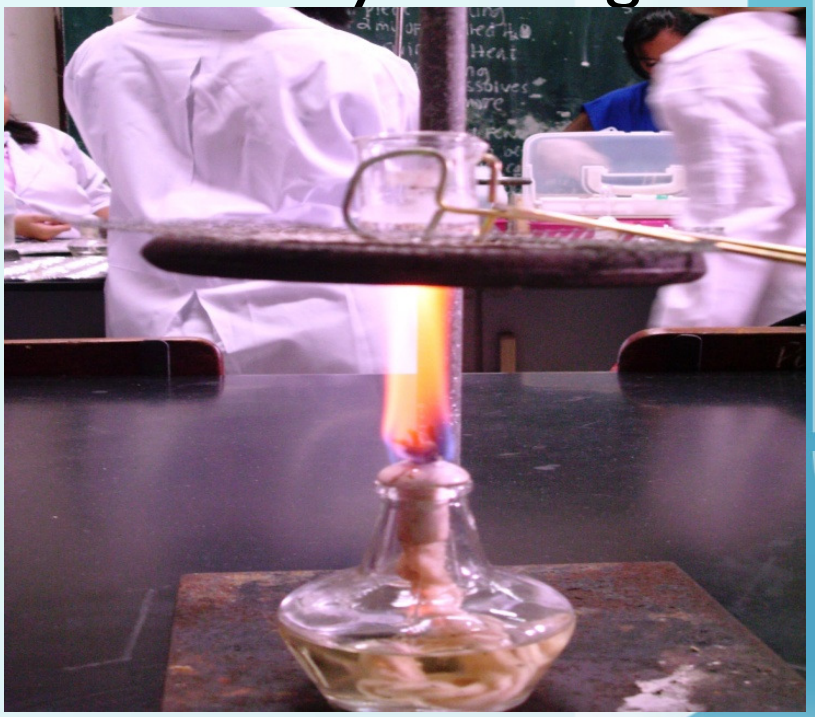
Recristalização vs. Precipitação

	Recristalização	Precipitação
Velocidade	lenta	rápida
Formação de cristais	seletiva	aleatória
Forma dos cristais	cristais regulares puros	sólido amorfo
Quantidade de impurezas	negligenciável	significativa

Experimental

Step 1: Mix boiling chip, 100 mg impure benzoic acid, ***& 2 mL distilled water?????.*** *Dissolve and heat while constantly swirling.*





Discussion

- ***Water is an ideal solvent for benzoic acid.***
  - *at 10°C, 2.1 g of benzoic acid dissolves in 1000 mL of water.*
  - *but at 95°C, 68g benzoic acid is soluble per 1000 mL of water.*
  - *this implies that at different temperatures, benzoic acid has an huge solubility difference in water.*

Discussion

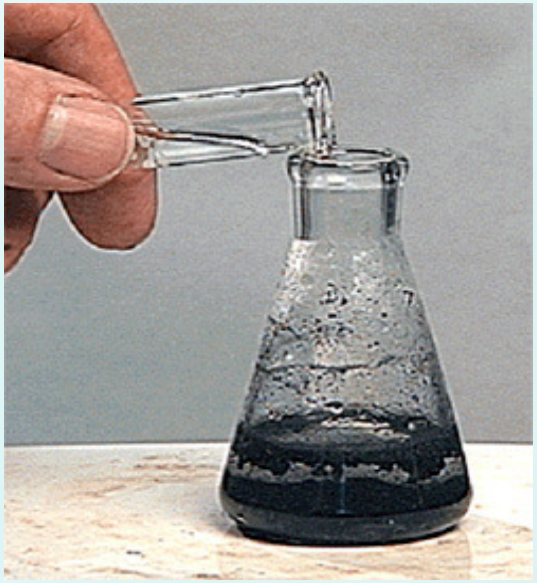
- ***Constant swirling at a high temperature.***
  - *swirling speeds up the dissolution of benzoic acid in water*
  - *agitation increases the entropy of the system, thus increasing the interaction between benzoic acid and water molecules.*
  - *the complete dissolution of benzoic acid results to a* ***clear solution****.*

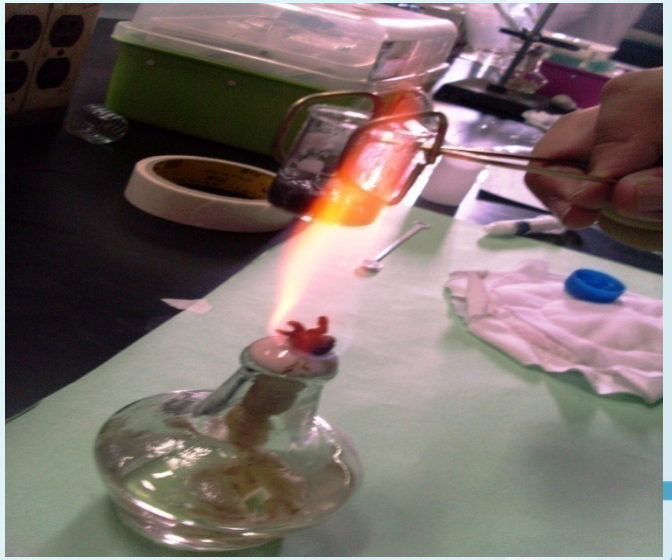
Discussion

- ***Adding the boiling chip while at room temperature.***
  - *adding the boiling chip at room temperature prevents **boiling over**.*
  - *this means that the solution will not spill out, since the boiling chip induces boiling of the mixture.*

Experimental

Step 2: Cool the solution. Add activated charcoal. Add a few drops of water. Heat again until observable change is seen.



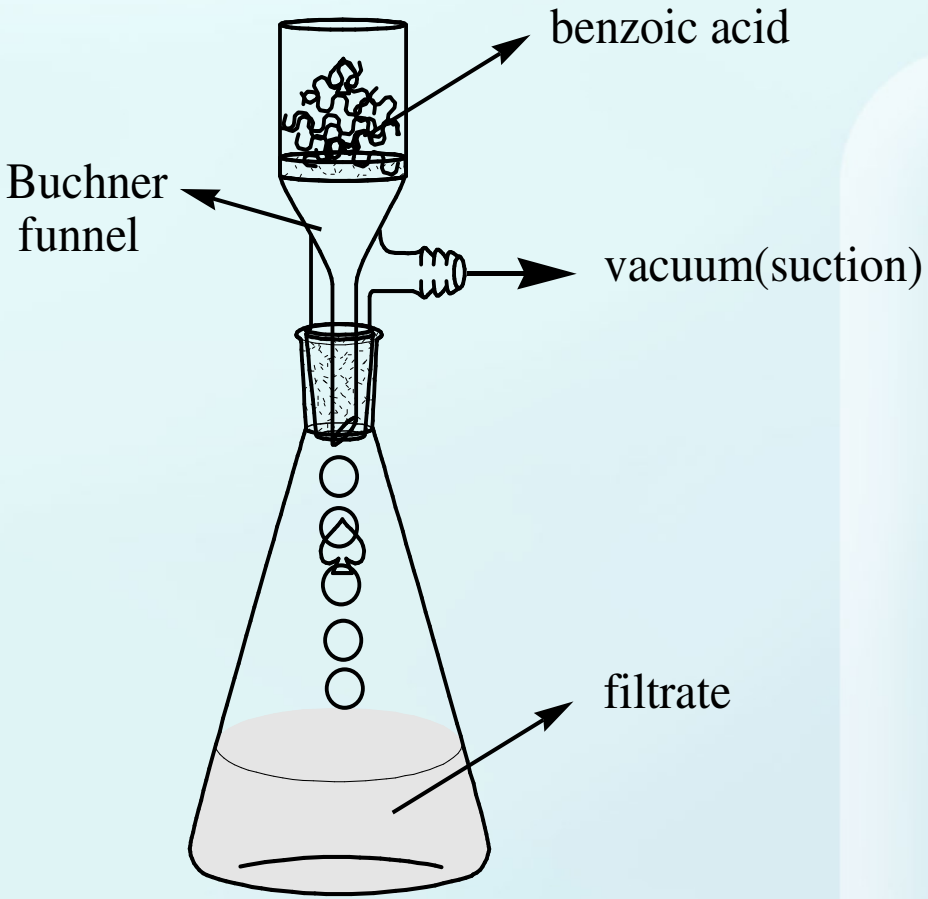


Discussion

- ***Decolorizing the solution with activated charcoal.***
  - *activated charcoal are carbon atoms that are finely separated.*
  - *these can adsorb impurities (stick to the surface of the substance) from the solution but are quite large to pass through the filter paper.*
  - *this results to minimization of impurities, and increased purity.*
  - ***WARNING:** too much activated carbon could cause the loss of the pure substance.*

Experimental

Step 3:



Discussion

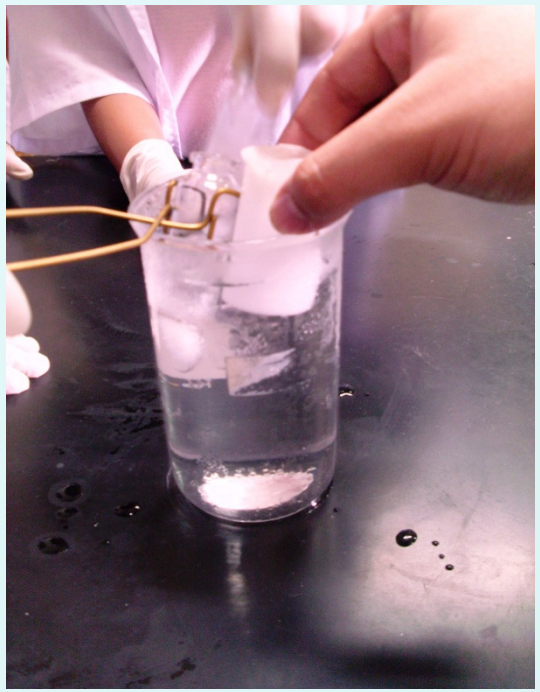
- ***First filtration of the solution***
  - *activated charcoal used, as well as other impurities, would be separated from the solution and left in the cotton plug.*
  - *this lessens the impurities in the crystallization process, and increases the purity of the yielded substance.*

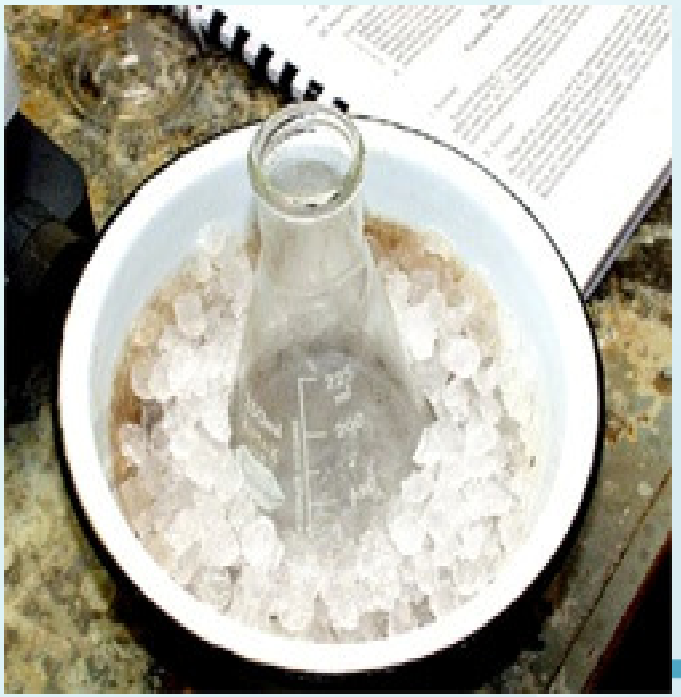
Discussion

- ***Filtering the solution rapidly.***
  - *as filtration is taking place so is the crystallization process.*
  - *the decrease in temperature causes a decrease in the solubility of the benzoic acid crystals.*
  - *some of the pure crystals would be separated from the filtrate and would be left as residue.*
  - *a lesser yield would result if the solution was not poured rapidly.*

Experimental

Step 4: Let the mixture cool in the ice bath.



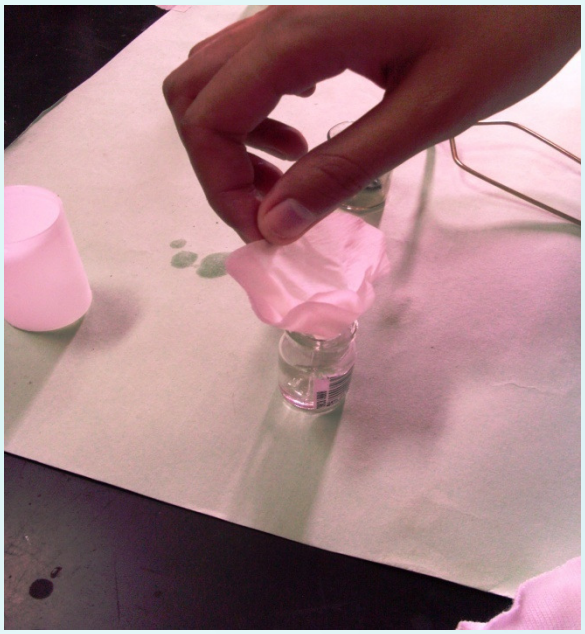


Discussion

- ***Slow cooling in ice bath.***
  - *slow cooling makes the crystals arrange finely, thus ensuring correct molecular arrangements/geometry.*
  - *this helps the crystals form in an undistorted manner and exclude the impurities in crystal formation.*

Experimental

Step 5: Collect the crystals on a filter paper. Rinse vial with ice –cold water to collect the remaining crystals in it. Use a seed crystal if necessary.



Discussion

- ***Using a seed crystal.***
  - *in cases, that crystallization while cooling does not take place, a seed crystal is employed.*
  - *the seed crystal has the same structure as the pure crystal to be recovered.*
  - *the seed crystal serves as a "source code" where the desired solid in the solution begins crystallization.*

Discussion

- ***Using a seed crystal.***
  - *since the lattice is a perfect fit, the other dissolved crystals would crystallize out as well.*
  - *impurities would remain dissolved in solution since its structure differs from the seed crystal and cannot fit in the lattice.*

Experimental

Step 6: Squeeze excess water from the filter paper. Dry it completely & weigh the filter paper.

Discussion

- ***The filter paper and crystals must be completely dried.***
  - *the added mass of water while weighing produces an inaccuracy in the desired data (% recovery) due to the solvent molecules.*

Results

- **% recovery of benzoic acid crystals**

Weight of impure sample = ***100 mg***

Weight of filter paper = ***400 mg***

Weight of filter paper and benzoic acid = ***450 mg***

Weight of pure crystals = ***50mg***

% recovery: 50mg/100mg x 100% = ***50%***

Guide Questions

Question#1:

- *List the properties that an ideal solvent should have to perform the purification of organic compound by recrystallization technique.*

Guide Questions

- **Question#2:**

What advantages does water have as a crystallization solvent?

Guide Questions

- **Question#3:**

Two students crystallized 10g samples of benzoic acid from water, the first dissolving benzoic acid at 80°C and filtering at 10°C , the second dissolving at 95°C and filtering at 18°C .

Calculate the quantity of water each student was required to use and the maximum recovery of benzoic acid possible in each case.

Guide Questions

Question # 4:

A Solid (X) is soluble in water to the extent of 1 g per 100 g of water at room temperature and 10 g per 100 g of water at the boiling point.

Guide Questions

a) *How would you purify X from a mixture of 10 g of X with 0.1 g impurity Y, which is completely insoluble in water and 1 g impurity Z having the same solubility characteristics in water as X?*

Guide Question

b) *How much pure X could be obtained after one recrystallization from water?*

Guide Question

c) *How much pure X could be obtained after one recrystallization from a mixture of 10g of X with 9 g of Z?*

d) *Based on the results obtained, what is suggested about the use of crystallization as a purification technique?*