# SOLVENT EXTRACTION

Worksheet - solvent extraction

1. When the oil and solvent mix after a brief period an equilibrium is set as shown below.

$$\text{Oil (l)} \rightleftharpoons \text{Oil (dissolved in hexane)} \quad \Delta H > 0$$

Which of the following factors will result in more oil extracted from the plant matter after equilibrium is established? Explain your reasoning.

i. Allowing the plant matter to remain in the solvent for a longer period of time before filtering.

*The system at equilibrium will not change, assuming there is no temperature change or volume change. So no increase in dissolved solute will occur once equilibrium is achieved.*

ii. Heating the solution.

*Since the dissolving of the oil is an endothermic process, heating the mixture will drive the system to the right increasing the amount of the solute dissolved in the solution.*

iii. Adding more solvent (hexane).

*"When additional solvent is added, the concentration of the solute decreases. In response to this change, the system shifts to the right to partially increase the solute concentration, thus re-establishing equilibrium where more solute is dissolved in the increased amount of solvent."*

iv. Introducing a blender to reduce the plant matter particle size.

*"Introducing a blender after the system has reached equilibrium will increase the surface area of plant matter and increase the site for solvent solute interaction. This will release more oile that may be trapped in plant matter thus increasing the amount of liquid oil present in the solvent. Increasing oil(l) will drive the system to the right.*

*Another valid explanation is that blending introduces kinetic energy and heat to the system and since the process is endothermic will drive the system to the right and achieve a new equilibrium position."*

2. Consider the three organic compounds derived from a plant, shown in fig 1. A plant extract mixture contains all three organic compounds.

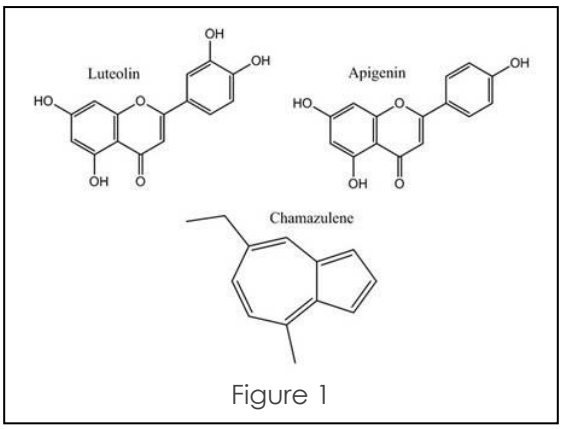

Figure 1

   a. Discuss how each of the four solvents listed below can be used to separate the compounds from the mixture. In your discussion refer to structure and bonding.

      *When considering the appropriate solvent to use to dissolve a compound it is important to match the polarity of the solvent and solute as closely as possible.*

      i. Hexane

      *Hexane is a non-polar compound and should be used as a solvent in the extraction of chamazulene only. Chamazulene is a hydrocarbon with aromatic rings and as such a non-polar substance. Hexane is also a non-polar substance that will interacts via weak dispersion forces with chamazulene.*

      ii. Hexan-1-ol

      *Hexan-1-ol is a mildly polar compound that can be used with limited success to dissolve luteolin and apigenin. It contains a long , 5 carbon long, hydrophobic section and a terminal hydroxyl group. Both luteolin and apigenin are moderately polar whilst containing aromatic hydrophobic structures, hence, an organic, highly polar, solvent such as ethanol is more likely to be effective in dissolving these two compounds. Although hexan-1-ol can interact, largely, via dispersion forces with all three compounds its limited polarity via the hydroxyl group enables it to also have limited interaction via hydrogen bonding with luteolin and apigenin only.*

      iii. Ethanol

      *Ethanol is a highly polar, organic, compound that can form hydrogen bonding via the hydroxyl group. It can be used with a high degree of success to dissolve luteolin and apigenin only. The non-polar nature of chamazulene does not allow for dipole-dipole interactions with polar ethanol molecules. Both luteolin and apigenin are moderately polar since they have a number of surface hydroxyl groups but also have large, hydrophobic, aromatic rings that enable it undergo interactions with ethanol such as dispersion forces and hydrogen bonding.*

      iv. Water

      *Water is a highly polar, inorganic compound that can form hydrogen bonding. Not being an organic compound it is limited in dissolving organic molecules with large hydrophobic section, as seen in all three compounds via aromatic rings. Water can not be used to purify any of these three molecules.*

3. The foliage of a plant has an abundance of both vitamins C and D. A laboratory is tasked with isolating each vitamin from the plant using solvent extraction and giving an accurate value of the concentration, in %m/m, of each vitamin.

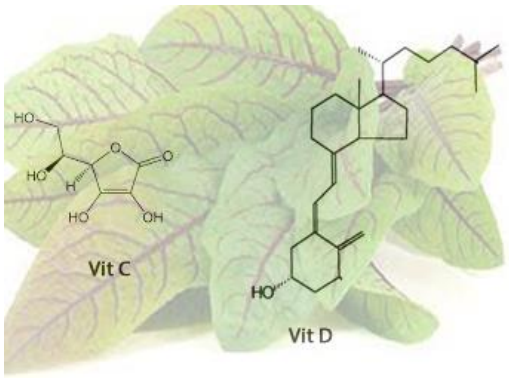

Figure 2

   a. Suggest an appropriate list of materials, including solvents, to use. Some are given below and must be used.
      - 2 X large evaporating dish
      - 2 X 10 mL measuring cylinder
      - 2 X plastic funnel
      - 1X box 0.18 mm thick filter paper (Whatman)
      - *100 mL of hexanol/hexanel or any other solvent that will extract vit D*
      - *100 mL conical flask*
      - *1 X electronic scale to three decimal places*

      *Any other plausible piece of equipment or material that is needed or used in the method below.*

   b. Suggest a step-by step procedure

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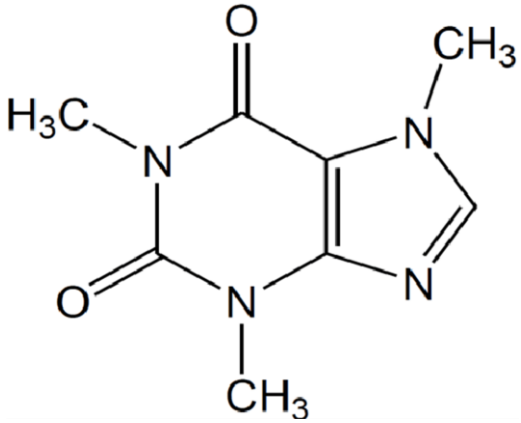
c. Are the results of the experiment deemed valid? Justify your answer.
*In this case the validity lies in the ability to measure the mass of vitamin C or D in the given mass of plant material. If the solvent used in the extraction process is highly selective for the target organic compound, the results can be more valid. However, if the solvent has a broad spectrum of solubility and dissolves various compounds, the extracted solution may contain impurities, affecting the accuracy of the concentration measurement. In this experiment we have no way of knowing if the solvents used are specific to dissolve the compounds in question only*

*d.* In what ways was the experiment designed to enhance the reliability and validity of its results?
*The experimental design in question b. had to be designed as to:*
*- identify all control variables and make sure their consistency throughout each trial.*
*- multiple trials had to be conducted.*
*- were there enough samples taken from different trees and different places on each tree to avoid sampling bias.*

4. Give three properties of a solvent that are desirable for solvent extraction and explain why.
   - *The solvent should exhibit selective solubility, meaning it has the ability to preferentially dissolve the target compound(s) from a mixture. This selectivity is crucial for efficiently extracting the desired components while minimizing the extraction of unwanted impurities.*
   - *Volatile so that after extraction, it's desirable for the solvent to be easily removed by evaporation from the extracted compounds, leaving behind a relatively pure sample of solute.*
   - *Chemically inert so it does not react with the solute molecules nor with the plant matter dissolved in the solvent.*
   - *Viscosity as less viscous solvents penetrate deeper in to the plant matrix to dissolve the solute.*
   *Any other properties such as cheap and easily accessible, not toxic, environmentally friendly and sustainable.*
5. Compare and contrast the separating techniques , solvent extraction and steam distillation by completing the table below.

| | Steam distillation | Solvent extraction |
|---|---|---|
| Type of molecule extracted | *Polar or semi polar volatile oils* | *Non-polar to semi non-polar organic molecules* |
| Temperature range | *At or around 100°C* | *Generally at room temperature* |
| Solvent required | *No solvent is required* | *Solvent required depending on the target molecule* |
| Specificity | *Limited specificity as arrange of compounds can be separated at the given temperature of 100°C* | *Specificity depends on the solubility of the target compound in the solvent.* |
| Usage | *To extract aromatic oils from plants.* | *Widely used in industry in pharmaceutical and food.* |

6. Caffeine is a stimulant found in coffee and its chemical structure is shown in fig.3. Decaffeinated coffee has the caffeine leached out of the coffee powder by solvents. Some of the compounds found in coffee and contribute to flavour are listed in table 1.

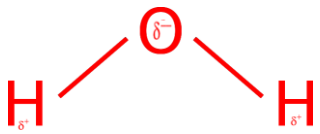


Caffeine
Figure 3

Consider two possible solvents ethyl acetate (fig 4) and water.

a. Which solvent is most polar? Justify your answer use diagrams.

*Water has a "V" shape, and its charge is asymmetrically distributed. Ethyl acetate also has*




*negative dipoles on the ester group, highlighted in green and slightly positive dipoles on either end, highlighted in red. Positive charges on either side makes the molecule less polar than asymmetrical nature of the water molecule.*

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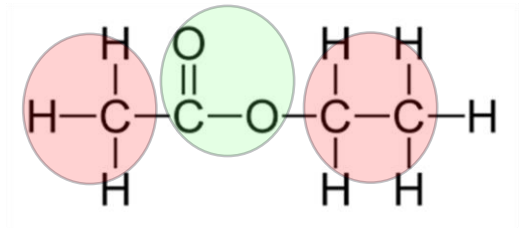

Ethyl acetate (BP 77 °C)
Figure 4

b. Select a solvent that will have minimum impact on taste but remove caffeine from the coffee granules. Explain your choice with reference to structure and intermolecular bonding.

*We are looking to have a solvent that has a similar polarity to caffeine but not to the other molecules contributing to flavour as shown in table 1.*

*From table 1 we can see that water as a solvent will extract all the compounds listed, however, ethyl acetate will extract predominantly caffeine and leave the rest of the compounds undissolved.*

c. Which purification method, solvent extraction or steam distillation, will be employed to extract and purify cafeine? Provide a rationale for the chosen method.

*Steam distillation is not an option as all compounds form vapours at much higher temperatures than 100°C at which steam distillation operates at.*

*Solvent extraction is the only method that can be used here with ethyl acetate as the solvent.*

*The coffee granules may be crushed and mixed with ethyl acetate. Caffeine will be preferentially dissolved and the mixture of beans and solvent filtered. The filtrate should contain a solution composed of caffeine dissolved in ethyl acetate. The ethyl acetate is then evaporated under mild heat around 80 °C to leave, predominantly, caffeine.*

| Compound found in coffee and contributing to flavour and taste. | Boiling point (°C) | Solubility in water | Solubility in ethyl acetate |
|---|---|---|---|
| Caffeine | 235 | high | high |
| Quinic acid | > 200 (But decomposes before this temp) | high | low |
| Formic acid | 101 | high | low |
| Acetic acid | 118 | high | low |
| Sucrose | 160 | high | low |

Table 1.

d. Ethyl acetate occurs naturally during the fermentation of fruit.

i. Provide balanced chemical equations, states included, for the two reactions that produce the reactants that form ethyl acetate at SLC.
Reaction 1 is an anaerobic fermentation reaction with glucose ($C_6H_{12}O_6$).

*$C_6H_{12}O_6(aq) \rightarrow 2CH_3CH_2OH(aq) + 2CO_2(g)$*

Reaction 2 is an oxidation reaction with the product of reaction 1 and atmospheric oxygen where water and an acid are produced.

*$CH_3CH_2OH(aq) + O_2(g) \rightarrow CH_3COOH(aq) + H_2O(l)$*

ii. Give the balanced chemical equation for the formation of ethyl acetate, states not included.

*$CH_3COOH + CH_3CH_2OH \rightarrow H_2O + CH_3CH_2OOCCH_3$*

iii. What class of reaction forms ethyl acetate. Circle the appropriate class of reaction.

Redox, Condensation, Addition, Complete combustion

iv. Justify your selection in iii.

*A reaction between two functional groups that produces a covalent bond and expels a small molecule, water.*
*It is also a redox reaction as the carbon in $CH_3COOH$ has an oxidation state of 0 and in $CH_3CH_2OOCCH_3$ has an oxidation state of -1. It is reduced. Whilst the oxidation state of carbon in $CH_3CH_2OH$ has an oxidation state of -2 and is oxidised to -1 in ethyl acetate.*