EXPERIMENT: SOLID-LIQUID EXTRACTION

OBJECTIVE

Investigation of the solid-liquid extraction parameters on the extraction yield.

THEORY

Leaching or solid-liquid extraction, crystallization, and adsorption are separation processes for solid-liquid separation. The solid-liquid extraction (leaching or extraction) process is the removing the soluble solid from the inert solid by dissolving it with an appropriate solvent. The dissolved substance is then purified by the methods such as crystallization or evaporation.

The best example of solid-liquid extraction, which is widely used in the production of biological, inorganic, and organic substances in chemical engineering, is tea or coffee making from daily life. Sugar production from sugar beets and oil from oilseeds are examples of solid-liquid extraction on the industrial scale [1].

Although solid-liquid extraction appears to be a similar process to liquid-liquid extraction, solid-liquid extraction is a more difficult process. Because diffusion rate in a solid is much slower than diffusion rate in a liquid, and therefore it is more difficult to reach equilibrium [1].

Factors affecting solid-liquid extraction can be grouped into three main groups.

- *Solid solvent interaction*: Preparation of the solid can be carried out by crushing, grinding, dividing into pieces, or reshaping. If the substance to be dissolved is on the solid surface, it is easy to extract with solvent. If the substance desired to dissolve is in the solid, the extraction efficiency increases by increasing the solid-solvent contact surface by reducing the particle size by subjecting the solid to a pre-treatment.
- *Selection of the solvent*: General properties of solvents that can be used in extraction are given in Appendix 1. The solvent chosen for the extraction process must be capable of dissolving the desired substance. The solvent capacity is determined depending on the saturation point of the solute in the solvent. However, regardless of the solvent capacity, the amount of solvent used should be chosen according to the amount of inert solid. The solvent must be easily separated from the extracted solid and the extracted solvent (solute + solvent). It is preferable to use low boiling solvents so that they can be easily separated from the product obtained after extraction.
- *Temperature*: Working at higher temperatures in the extraction process accelerates the diffusion of the solute to the solvent. Although the extraction efficiency increases with

heat depending on the structure of the solid to be extracted, degradation may occur in the structure of some components at high temperatures and undesirable compounds may also dissolve. [2, 5].

EXPERIMENTAL SET-UP and METHOD

The flow chart of the set up to be used in solid-liquid extraction experiment is given in Figure 1. The appropriate amount of known weighed sample is placed in the first compartment. The appropriate solvent (pure water for our experiment) is filled into the feed tank. The solvent is poured drop wise onto the substance to be extracted from the solution feed pipes on the compartments. In the solid-liquid extractor, extraction takes place in the compartments divided into three compartments by the continuous rotation of the solid. The solution consisting of the substance and solvent extracted from the solid is taken from the 1st compartment and given to the solvent feed of the 2nd compartment. The solution taken from the second compartment is also given to the 3rd compartment. The solvent was transferred between compartments with the help of peristaltic pumps. The system is designed as a rotary cell in three steps. The temperature and flow rates of each compartment are adjustable. Concentration values of each compartment can be read from the conductivity values measured in the system with the help of the computer program. At the end of the extraction process, the mixture of solvent and extracted substance (extract) comes to the discharge pipe and is taken from there. Inert solid matter is taken from the bottom of the last compartment.

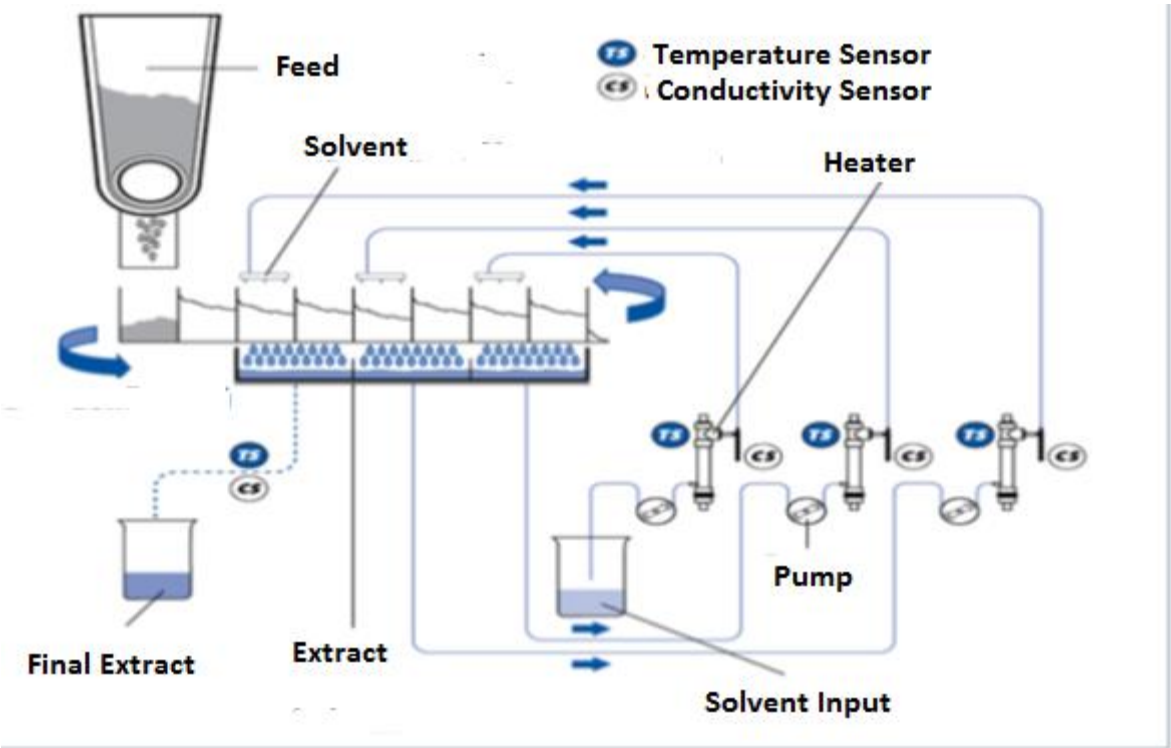

Figure 1. Experimental set-up and flow diagram

Experiment A: Effect of Solid Content on Extraction

- In this part of the experiment, the effect of solid amount on extraction is examined by using 5, 10, and 15 grams of substance respectively.
- The feeding tank is filled with pure water.
- The experimental setup shown in Figure 1 is prepared. Connections are checked.
- The flow rate of the water is set to 5mL/s and its temperature to 25 ^{o}C.
- The system is turned on with the on/off button. The heaters are turned on, the rotation speed of the cells is adjusted.
- Concentration data are obtained for every 10 seconds with the computer program to be used.

Experiment B: Effect of Flow Rate on Extraction

- In this part of the experiment, the effect of flow rate is examined by keeping the amount of material to be extracted and the temperature constant.
- The flow rate of the water is set to 5 mL/s first, then 6 mL/s, and finally 7 mL/s.
- The extraction process is repeated using 5 grams of substance with a solvent (pure water) at a temperature of 25 ^{o}C at each flow rate.
- Concentration data are obtained for every 10 seconds with the computer program to be used.

Experiment C: Effect of Temperature on Extraction

- In this part of the experiment, the effect of different temperatures on extraction at 5 g material feed and 5mL/s flow rate will be examined.
- All three compartments of the heater are brought to 25 °C, then 35 °C and finally 45 °C, and the concentration data are taken for every 10 seconds with the computer program to be used.

DISCUSSION

- Investigate the effects of solid/solvent ratio in solid-liquid extraction.
- Describe the effects of the pre-treatments applied to the solid in the solid-liquid extraction on the extraction yield.
- State the importance of solvent selection in solid-liquid extraction. Explain in detail the features that need to be considered in the choice of solvent.
- Describe the effects of solvent flow rate on extraction yield in solid-liquid extraction.
- Investigate the effects of solvent temperature on solid-liquid extraction.

QUESTIONS

1. Describe the types of extractors that can be used in solid-liquid extraction?

2. Derive the equations used to determine the ideal number of stage in extraction?

3. Describe by drawing the solid-solvent flow types in solid-liquid extraction?

4. Draw flow charts by giving examples of processes in which solid-liquid extraction is used in chemical engineering applications?

5. Using kerosene, 2 tons of waxed paper per day is extracted with a continuous counter-current in a system with ideal stages. 25% of wax paper is paraffin and 75% is pulp. The extracted paper is dried in a dryer to evaporate the kerosene. The maximum amount of unextracted wax remaining after evaporation will be 0.2 kg per 100 kg of paper. Kerosene used in extraction contains 0.05 kg of wax per 100 kg. Experiments have shown that every 1 kg of pulp carries 2 kg of kerosene. There is 5 kg of wax in 100 kg of kerosene in the extract from the extractor. How many stages are needed under these conditions?

6. Benzene is used as a solvent in the production of soybean oil. Determine the amounts and components of the upstream and downstream streams that leave an ideal extractor when 1 kg of grounded soybean (50% soluble, 50% inert) is extracted with 1 kg of pure benzene. Calculate the percentage of oil taken up in the benzene overhead stream. In previous experiments

conducted in the pilot plant, when equilibrium is reached, it was observed that the bottom stream contains 65% (by weight) of solids.

REFERENCES

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APPENDIX 1.

Table 1. Solvents commonly used in extraction and their properties

Solvent	Dielectric constant[a]	Boling point (°C)	Density (g/mL)	Flammability[b]	Toxicity[b]	Compatibility
Hexane	1.9	68.7	0.65	+++	+	It is a weak solvent for polar compounds; can be removed easily.
Benzene	2.3	80.1	0.87	+++	+++	It is extremely toxic for general uses; prone to emulsion; less toxic than toluene.
Ether	4.3	34.6	0.71	+++	+	It is a good solvent especially for oxygen-containing compounds; tends to peroxide formation in long term storage.
Chloroform	4.8	61.7	1.48	-	+++	It can be removed easily; is toxic; it tends to emulsion.
Ethyl acetate	6.0	77.1	0.89	+++	+	Suitable for polar compounds; can absorb water
Dichloromethane	8.9	39.7	1.31	-	++	It is a good solvent for general use; slightly tend to emulsion formation.
1-Butanol	17.5	117.7	0.81	++	+	It is used as a last solvent for higher polar components.

[a] Although the dielectric constant gives information about the polarity of the solvent, it does not always indicate that the solvent can dissolve polar organic compounds.

[b] + = least flammable/toxic, +++ = most flammable/toxic