

In the name of God

Manual

Solid-Liquid Extraction

Datis Energy Company

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Datis Energy



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Goal

Solid-phase extraction (SPE) is an extractive technique by which compounds that are dissolved or suspended in a liquid mixture are separated from other compounds in the mixture according to their physical and chemical properties. Analytical laboratories use solid phase extraction to concentrate and purify samples for analysis. Solid phase extraction can be used to isolate analytes of interest from a wide variety of matrices, including urine, blood, water, beverages, soil, and animal tissue.

Introduction

Solid-liquid extraction (or leaching) is the separation of a solid solute from a mixture of solids by dissolving it in a liquid phase. Basically, there are three components in leaching: solid solute, insoluble solids and solvent. In most cases, the diffusion of intra-particle soluble component(s) controls the extraction rate. Therefore the process is often called as diffusion extraction. Solid-liquid extraction is widely used in food (e.g. extraction of sugar from sugar cane or sugar beet; isolation of vegetable oils from different seeds) and pharmaceutical industries (e.g. Extraction of active components from medicinal plants), and in hydrometallurgy (e.g. leaching of metal ions from ores). For mathematical modeling of leaching an ideal solid-liquid extraction is defined. The solvent added to the dry raw material is partly taken up by the solid material and the soluble ingredients are instantaneously dissolved. Often all the solute is already dissolved (e.g. Sugar in sugar beet). The solution is then split into two parts. The external solution is called extract and the internal one raffinate (in the case of plant materials this is cell liquor).



Solid Liquid Extraction



Extraction uses the property of solubility to transfer a solute from one phase to another phase. Solid-phase extraction (SPE) is an extractive technique by which compounds that are dissolved or suspended in a liquid mixture are separated from other compounds in the mixture according to their physical and chemical properties. Analytical laboratories use solid phase extraction to concentrate and purify samples for analysis. Solid phase extraction can be used to isolate analytes of interest from a wide variety of matrices, including urine, blood, water, beverages, soil, and animal tissue.

SPE uses the affinity of solutes dissolved or suspended in a liquid (known as the mobile phase) for a solid through which the sample is passed (known as the stationary phase) to separate a mixture into desired and undesired components. The result is that either the desired analytes of interest or undesired impurities in the sample are retained on the stationary phase. The portion that passes through the stationary phase is collected or discarded, depending on whether it contains the desired analytes or undesired impurities. If the portion retained on the stationary phase includes the desired analytes, they can then be removed from the stationary phase for collection in an additional step, in which the stationary phase is rinsed with an appropriate eluent.

It is possible to have an incomplete recovery of the analyses by SPE caused by incomplete extraction or elution. In the case of an incomplete extraction, the analyses do not have enough affinity for the stationary phase and part of them will remain in the permeation. In an incomplete elution, part of the analyses remain in the sorbent because the eluent used does not have a strong enough affinity.

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Many of the adsorbents/materials are the same as in chromatographic methods, but SPE is distinctive, with aims separate from chromatography, and so has a unique niche in modern chemical science.

Solid-liquid extraction methods

The main methods of solid-liquid extraction are:

- Stationary solid-bed leaching,
- Countercurrent,
- Volatile,
- Moving-bed leaching,
- Bollman extractor
- Bucket.

These methods have 4 basic steps:



- Physical extraction of oil from seeds in the device
- Desalinating from soybean meal then drying and cooling
- Distillation to separate oil from solvent
- Recycling solvent for reuse

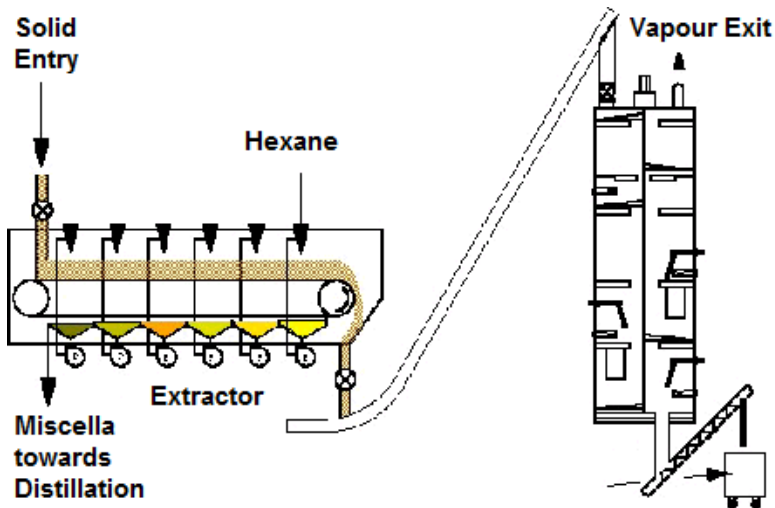


Figure 1 : De-Smet device

The main evaporator

The pressure in primary and main evaporators are the same, so fast evaporation is considerable. The exhausted sensibio which leaves the primary evaporator (65-75% density) at 50°C, is heated while entering the main evaporator to 75°C.

The density of exhausted sensibio H₂O from evaporator reaches 93% and the temperature reaches 80°C.

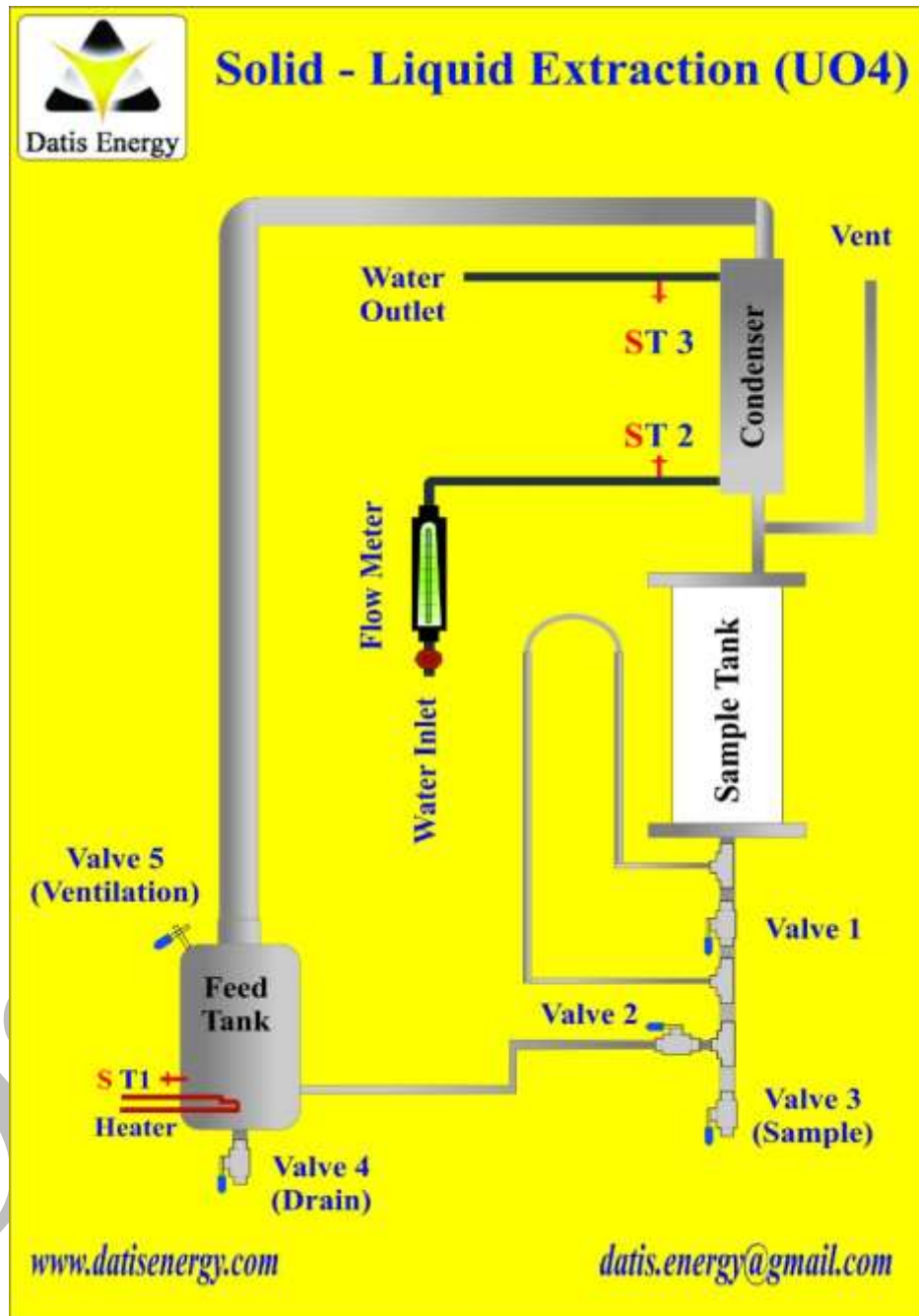


Solvent separators

To separate the remained Hexane, the temperature must be increased or the pressure be decreased, the crude oil cannot go higher than 105°C (decrease in quality), so steam distillation is used. The oil must be preheated until it reaches 90-100°C. Steam is injected from bottom of tower to increase the heat coefficient and separation of liquid and steam happen at the top of separator. Density of exhaust oil is 99.5-99.2%.



Apparatus parts





Solid Liquid Extraction



- Stainless steel boiler –power 2.5 KW– Thermocouple to measure temperature of liquid solvent
- Pressure gauge to measure the level of fluid in reservoir
- Drain valve for boiler discharge
- Feed valve
- Sampling valve
- Condenser –0.25 m² – heat transfer surface
- Pyrex extraction reservoir
- Thermometer (boiler, entrance and exhaust water of condenser, holder reservoir)

Description

As mentioned before, Solid-liquid extraction is used for oil extraction from seeds. The aim of this experiment is the extraction of oil from soybeans/sunflower seed, therefore the soybeans are placed in the solid bed and the solvent is Hexane. The condenser, condenses the vapor and then leads the condensed solvent vapor into extraction reservoir. After contact of Normal Hexane with the sample powder, by mass transfer, the oil which exists in solid phase and according to solidity difference enters the solvent. The reservoir liquid which consists of Normal Hexane and oil fills the reservoir, after getting to desired height, exits extraction reservoir and then by closing V2 and V3 the liquid returns to the reservoir.



Summarized principle of the apparatus

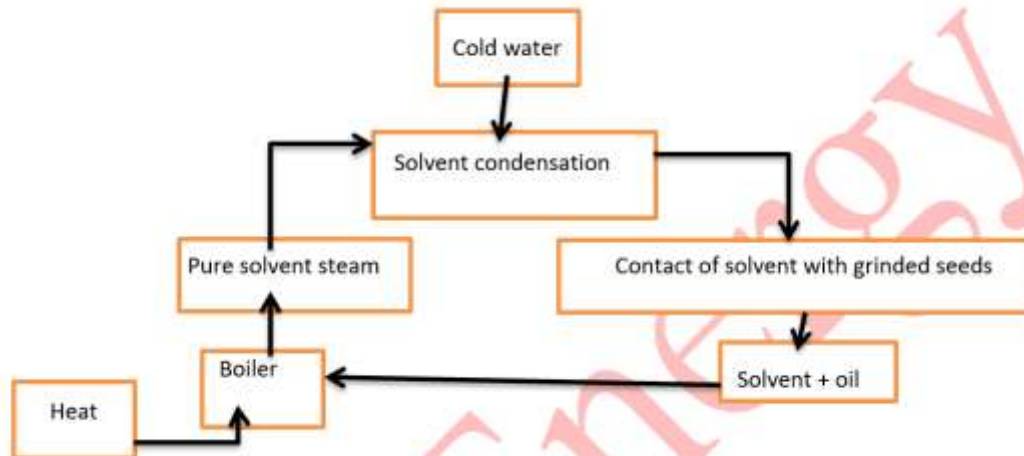


Figure 2: system flow chart

Test instruction

Measurement of extracted oil

- ✓ Weigh about 200 to 500 grams of grinded Soybeans/sun flower seeds. The size of each grinded seed mustn't be less than 2 millimeters.
- ✓ Put the sample powder in a metal mesh and put the metal mesh in interior mesh.
- ✓ Put the mesh in the extraction reservoir using its handle.
- ✓ Pour the solvent into the boiler until 2/3 of reservoir is filled.
- ✓ Connect the condenser to cold water.
- ✓ Turn on the heater.
- ✓ Open Rotameter's needle valve to set the discharge of water.
- ✓ The solvent inside the pot, evaporates and enters the condenser and turns into liquid, then penetrates into extraction reservoir (or samples mesh).



Solid Liquid Extraction



The solvent enters extraction reservoir from top of it, after touching the sample, exits at the bottom, then enters boiler again.

- ✓ The device is atmospheric. The sample mesh is provided to prevent the powder from entering the device. Normal Hexane is the solvent in this experiment.
- ✓ After depleting the extraction reservoir, the solvent enters the boiler again, while depleting you can take a sample from the valve which is located under the reservoir. (First step sample)
- ✓ For each step of taking sample, you must wait until the amount of depleted Normal-Hexane from condenser which has been entered the sample tank, reaches a height close to capillarity level in the tube. Then close V2 and open V1 and V3 in order to take sample. After taking sample at first close V3, then open V2 and at the end close V1.
- ✓ The oil sample should get to environment temperature. Then, start the next step and after depleting the reservoir get the second step's sample. You should take note of the volume of the sample too.
- ✓ After 5 steps of the experiment turn off the heater to stop the solvent's cycling. Open the exhaust valve (V4) which is under the reservoir and deplete the solvent.
- ✓ Wait until the solvent depletes, then take out the metal mesh and deplete it, too.
- ✓ Dry the sample and weigh it.
- ✓ Complete the table during the experiment.

NOTE: In order to do the experiment in automatic state (5 steps of taking sample be done automatically) V1 and V3 must be closed and V2 be opened.



Solid Liquid Extraction



Make sure of depleting all air properly in automatic state. To do so, when Normal-Hexane starts to enter the sample tank and its height reaches almost 5 centimeters , while V2 is opened , open V1 . Then open V3 for a short time and once the liquid starts to extract close V3 immediately. At this point air depleting is done properly.

Taking sample

To take sample from extracted oil

When the experiment is done, turn off the boiler and then give system time to cool.

The oil and the Normal-Hexane will be separated, using the valve under the boiler take sample from oil and test it.

Solid sample specification	
name	
Amount consumed	
Solvent specification	
name	
Consumed amount	
density	

Table\ : first test



Solid Liquid Extraction



Solvent specification	
Boiler Temperature(°C)	
Entrance water temperature (°C)	
Discharged condenser water temperature(°C)	
Sample's pot temperature(°C)	
Condenser's fluid flow rate	

Table 2

Sample #	Weight of Pico-meter(empty)	Weight of Pico-meter(full)	Mass fraction	density	efficiency
1					
2					
3					
4					
5					

Table 3 : first test

After measuring and calculating density of samples, using interpolation, calculate the mass fraction of extracted oil.

Considering $x=0$



Solid Liquid Extraction



$$\rho_{mix} = \rho_{cyclohexane} = 0.66 \frac{gr}{cm^3}$$

X	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1
$\rho \left(\frac{gr}{cm^3} \right)$	0.6	0.68	0.71	0.74	0.76	0.79	0.82	0.84	0.87	0.90	0.9
	6	7	4	1	8	5	2	9	6	3	3

Table 4 : mass fraction-density

Measure the solvent density before the experiment and fill in the table.

Efficiency calculation

$$E = \frac{\text{mass of extracted oil}}{\text{mass of (premier oil - previous extracted oil)}} \times 100$$

Pycno meter

Pycnometers are used for measuring and comparing densities or specific gravities of liquids and solids, including soil.



The volume of Pycnometer is written on it. Clean the Pycnometer before using the acetone and dry with compressor or blower. Weigh the liquid before adding it to the Pycnometer. Fill the pycnometer and weight it again. Calculate the difference in weight between the filled and the empty state.

For instance empty Pycnometer weight 25 gr and filled weight 67gr. consider the volume of Pycnometer 50 cc then:

$$M = 67 - 25 = 42 \text{ gr}$$

$$V = 50 \text{ cc}$$

$$\rho = \frac{m}{V}$$

$$\rho = 42/50 = 0.84$$



Attention

Error factors in experiment

- ✓ Measuring sample weight before chilling
- ✓ Mismeasuring the volume of vessel
- ✓ Mismeasuring the weight of vessel
- ✓ Error in the device
- ✓ Error in calculation

Safety tips

- ✓ After turning on the heater flow the condenser's water
- ✓ Be careful with the solvent
- ✓ During the test ,pressure gauge should display zero
- ✓ During the experiment, do not fire anything near the device
- ✓ Do not open the drain valve of boiler While the heater is turned on
- ✓ Do not open boiler's entrance door, while performing the operation
- ✓ When you want to add feed in boiler, make sure it's off.
- ✓ The valve which is at the top of condenser should always be open
- ✓ The solvent's vapors are so toxic. While recycling the solvent or extracting use hood