Kirk-Othmer Encyclopedia of Chemical Technology.

EXTRACTION, LIQUID-SOLID

Liquid–solid extraction or leaching is a unit operation that predates large-scale industrial operations, with a history of known uses that goes back to Roman times. The early leaching process was known as lixiviation, a term used to describe the extraction of alkaline salts from wood (qv) and other plants. The term leaching, in use in the eighteenth century, was used to describe the process of percolating a liquid through a solid material. It is presumed that a soluble component was removed from the solid phase during the percolation so that the operation was distinguishable from sand filters which were quite widely used by that date, but which served a quite different function. Extraction is used in a wide variety of process industries and traditional terms have evolved in different fields. These include leaching, washing, percolation, digestion, steeping, lixiviation, and infusion, among others.

Many substances used in modern processing industries occur in a mixture of components dispersed through a solid material. To separate the desired solute constituent or to remove an unwanted component from the solid phase, the solid is contacted with a liquid phase in the process called liquid–solid extraction, or simply leaching. In leaching, when an undesirable component is removed from a solid with water, the process is called washing.

In the biological and food processing industries many products are extracted from their original structure by liquid–solid extraction (see Biotechnology; Food processing). Sugar (qv) is extracted from sugar beets using hot water; instant coffee (qv) is leached from ground roasted coffee using water; soluble tea (qv) is leached from tea leaves; pharmaceutical components, flavors, and essences are leached from plant roots, leaves, and stems (see Flavors and spices; Pharmaceuticals); and oil is extracted from peanuts, soybeans, sunflower and cotton (qv) seeds, and halibut livers by solvents such as hexane, acetone, or ether (see Solvents, industrial; Soybeans and other oilseeds). These are all examples of liquid–solid extraction.

Large-scale leaching also occurs in the metal processing industries, where useful metals frequently occur mixed with large quantities of unwanted matter, and leaching is used to remove the metals as soluble salts. For example, gold is leached from its ore using aqueous sodium cyanide solutions (see Cyanides; Gold and gold compounds); cobalt and nickel by sulfuric acid–ammonia–oxygen mixtures (see Cobalt and cobalt alloys; Nickel and nickel alloys); and copper salts by sulfuric acid or ammoniacal solutions (see Metallurgy, extractive metallurgy; Minerals recovery and processing).

1. Mechanisms of Extraction

If the solute is uniformly distributed through the solid phase the material near the surface dissolves first to leave a porous structure in the solid residue. In order to reach further solute the solvent has to penetrate this outer porous region; the process becomes progressively more difficult and the rate of extraction decreases. If the solute forms a large proportion of the volume of the original particle, its removal can destroy the structure of the particle which may crumble away, and further solute may be easily accessed by solvent. In such cases the extraction rate does not fall as rapidly.

In general, the following steps can occur in an overall liquid–solid extraction process: solvent transfer from the bulk of the solution to the surface of the solid; penetration or diffusion of the solvent into the pores of the solid; dissolution of the solvent into the solute; solute diffusion to the surface of the particle; and solute transfer to the bulk of the solution. The various fundamental mechanisms and processes involved in these steps make it impracticable or impossible to describe leaching by any rigorous theory.

Any one of the five basic processes may be responsible for limiting the extraction rate. The rate of transfer of solvent from the bulk solution to the solid surface and the rate into the solid are usually rapid and are not rate-limiting steps, and the dissolution is usually so rapid that it has only a small effect on the overall rate. However, knowledge of dissolution rates is sparse and the mechanism may be different in each solid (1).

The overall extraction process is sometimes subdivided into two general categories according to the main mechanisms responsible for the dissolution stage: (*1*) those operations that occur because of the solubility of the solute in or its miscibility with the solvent, eg, oilseed extraction, and (*2*) extractions where the solvent must react with a constituent of the solid material in order to produce a compound soluble in the solvent, eg, the extraction of metals from metalliferous ores. In the former case the rate of extraction is most likely to be controlled by diffusion phenomena, but in the latter the kinetics of the reaction producing the solute may play a dominant role.

1.1. Diffusion and Mass Transfer During Leaching

Rates of extraction from individual particles are difficult to assess because it is impossible to define the shapes of the pores or channels through which mass transfer (qv) has to take place. However, the nature of the diffusional process in a porous solid could be illustrated by considering the diffusion of solute through a pore. This is described mathematically by the diffusion equation, the solutions of which indicate that the concentration in the pore would be expected to decrease according to an exponential decay function.

To obtain an indication of the rate of solute transfer from the particle surface to the bulk of the liquid, the concept of a thin film providing the resistance to transfer can be used (2) and the equation for mass transferwritten as:

$$\frac{dM}{dt} = \frac{D^* A(c_s - c)}{\delta} \qquad (1)$$

where A = the area of the solid−liquid interface, c = the concentration of the solute in the bulk of the liquid at time, t, c_s = the concentration of the saturated solution in contact with the particles, D^* = a diffusion coefficient (approximated by the liquid-phase diffusivity), M = the mass of solute transferred in time t, and δ = the effective thickness of the liquid film surrounding the particles. For a batch process where the total volume V of solution is assumed to remain constant, $dM = V\,dc$ and

$$\frac{dc}{dt} = \frac{D^* A(c_s - c)}{\delta V} \qquad (2)$$

The time t taken for the concentration of the solution to rise from its initial value c_0 to a value c is obtained by integration of this equation, assuming that both A and δ remain constant, to give:

$$c = c_s - (c_s - c_0)\exp\left(\frac{-D^* At}{\delta V}\right) \qquad (3)$$

This simple analysis also shows that the bulk solution approaches a saturated condition exponentially. That A and δ are constant are both significant assumptions rarely met in extraction, although the change with time may be slow. The interfacial area tends to increase as extraction proceeds, and is of course dependent on the extent to which the solid material has been ground prior to extraction. D^* should be treated as an effective mass-transfer coefficient which would be sensitive not only to the composition and properties of the solution surrounding the particle, but also to the hydrodynamic conditions in the bulk of the solution. For larger particles, which are usually present in leaching, equations are available (3, 4) to predict the mass-transfer coefficient in agitated vessels.

2. Process Design

In most leaching operations the maintenance of constant fluid flows, pressures, and temperatures are important. These, together with the need to provide a sufficient contact time between the solvent and the solids, usually indicate a need for continuous, multistage, countercurrent processes in which fresh solvent is fed to the final stage while the solids are fed to the first stage. The objective is to be able to operate at steady conditions, and to be able to avoid extraction of undesirable material while preventing loss of solvent for both economic and safety reasons. This is usually achieved through the use of the usual control equipment, and recording instruments provide a useful means of studying plant performance. There are other factors which must be taken into account in the early stages of a design such as the particle size of the solid and the solvent employed.

2.1. Particle Size

The smaller the particle size, the greater is the relative interfacial area between the solid and the liquid and the shorter are diffusional path lengths, and therefore the higher is the rate of transfer of solute. However, smaller particle sizes tend to lead to lower drainage rates from the solid residue and can create problems in the solids flow through countercurrent extraction equipment. A compromise has to be made to select a particle size which offers an acceptable extraction rate but yet does not unduly impede flow of solvent through a percolation process or of solids through a countercurrent process. If the extractable material is a minor proportion of the starting material the disposal of the solids residue may present a problem. If the residues have some commercial value as a product after further processing, excessive grinding of the feed solids may render the residue unsuitable for the product and hence have an adverse effect on the economics of the extraction process.

2.2. Solvent

Solvent choice is determined by the chemical structure of the material to be extracted, and the rule that like dissolves like provides useful guidance. Thus vegetable oils (qv) consisting of triglycerides of fatty acids are normally extracted with hexane [110-54-3], whereas for free fatty acids, which are more polar than the triglycerides, more polar alcohols are used. Halogenated hydrocarbons and hexane are both widely used as solvents, and liquid carbon dioxide [124-38-9](qv) appears to be suitable for extracting flavor components from plants (5) (see Supercritical fluids). Where a choice of solvent other than water exists on the grounds of comparable solubility of the solute, the following criteria are likely to be considered.

2.2.1. Selectivity

Solvent selectivity is intimately linked to the purity of the recovered extract, and obtaining a purer extract can reduce the number and cost of subsequent separation and purification operations. In aqueous extractions pH gives only limited control over selectivity; greater control can be exercised using organic solvents. Use of mixed solvents, for example short-chain alcohols admixed with water to give a wide range of compositions, can be beneficial in this respect (6).

2.2.2. Physical Properties

Low surface tension facilitates wetting of the solids in the first extraction stage, and low viscosity assists diffusion rates in the solvent phase. A low solvent density is desirable to reduce the mass of solvent held up in the solid being extracted, but solvent choice is usually dictated by other factors. A high boiling solvent with a high latent heat of evaporation requires recovery conditions that may be adverse for thermally sensitive extracts and increases the cost of solvent recovery. In chemical leaching the thermodynamics of the leaching reaction must be considered in terms of the redox potential–pH diagram (Pourbaix diagram) which can be constructed from standard free-energy data (7). This provides the basis for choosing equilibrium leaching conditions (acidic vs basic, oxidizing vs reducing), leaving the need for a kinetic study to provide data on leaching rates.

2.2.3. Thermal Stability

At processing temperatures in both the extraction and recovery plants the solvent should be completely stable to avoid expensive solvent losses; contamination of the solvent by any solvent breakdown products must be avoided.

2.2.4. Hazards

The solvent should be nontoxic and nonhazardous; adequate design must take into account flammability and explosivity characteristics of the solvent.

2.2.5. Cost

The cost of fresh solvent is reflected in the operating costs in the form of solvent make-up charges. Avoidance of solvent losses, and hence a reduction of operating costs, may be obtainable through better plant design which is usually associated with increased capital costs.

2.3. Temperature

Both the solubility of the material being extracted and its diffusivity usually increase with temperature, and higher extraction rates are obtained. In some cases the upper limit for the operating temperature is determined by factors such as the need to avoid undesirable side reactions.

2.4. Agitation of the Fluid

Agitation of the solvent increases local turbulence and the rate of transfer of material from the surface of the particles to the bulk of the solution. Agitation should prevent settling of the solids, to enable most effective use of the interfacial area.

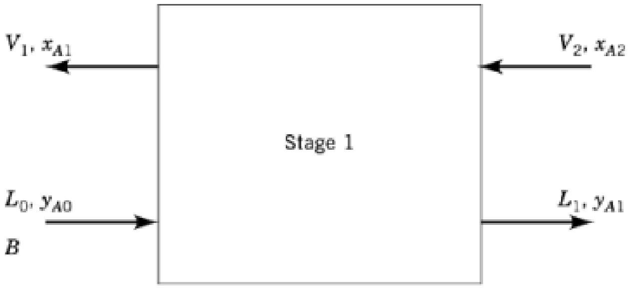

Fig. 1. Flow diagram for single-stage leaching.

3. Equilibrium Relationships and Mass Balances

The solid can be contacted with the solvent in a number of different ways but traditionally that part of the solvent retained by the solid is referred to as the underflow or holdup, whereas the solid-free solute-laden solvent separated from the solid after extraction is called the overflow. The holdup of bound liquor plays a vital role in the estimation of separation performance. In practice both static and dynamic holdup are measured in a process study, other parameters of importance being the relationship of holdup to drainage time and percolation rate. The results of such studies permit conclusions to be drawn about the feasibility of extraction by percolation, the holdup of different bed heights of material prepared for extraction, and the relationship between solute content of the liquor and holdup. If the percolation rate is very low (in the case of oilseeds a minimum percolation rate of 3×10^{-3} m/s is normally required), extraction by immersion may be more effective. Percolation rate measurements and the methods of utilizing the data have been reported (8, 9); these indicate that the effect of solute concentration on holdup plays an important part in determining the solute concentration in the liquor leaving the extractor.

3.1. Single-Stage Leaching

A single-stage leaching process is shown in Figure 1. The solution overflow rate is V kg/h; the mass fraction of solute in the overflow solution is x_A; and the liquid in the slurry is flowing at L kg/h, and has a composition y_A. The mass flow of dry inert solids in the slurry is B kg/h.

The material balance equationsare, for the total solution:

$$L_0 + V_2 = L_1 + V_1 = M \qquad (4)$$

where M is the total input flow rate of solution to the unit; for the solute component A:

$$L_0 y_{A0} + V_2 x_{A2} = L_1 y_{A1} + V_1 x_{A1} = M x_{Am} \qquad (5)$$

where $x_1 = y_1 = x_{Am}$; and for the solids:

$$B = L_0 N_0 = L_1 N_1 \qquad (6)$$

where N_i is the mass concentration of inert solids in the ith stream, ie, kg of inert solid per kg solution. From these balances the concentration of the discharged solution can be estimated.

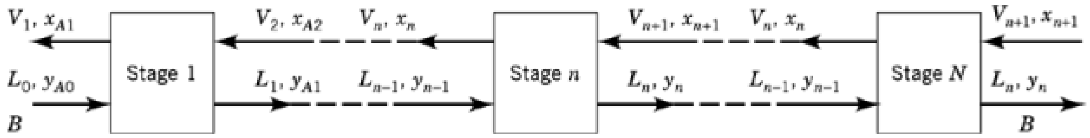

Fig. 2. Flow diagram for countercurrent multistage leaching.

3.2. Countercurrent Multistage Leaching

Countercurrent extraction offers the most economical use of solvent, permitting high concentrations in the final extract and high recovery from the initial solid but utilizing the least amount of solvent. In a multistage operation fresh solid enters the first stage and fresh solvent enters the final stage; the latter is gradually enriched in solute until it leaves the extraction battery as overflow from the first stage. The operation is usually discontinuous in that the solvent is pumped from one vessel to the next intermittently and allowed to remain until equilibrium extraction is approached. When the amount of solvent removed with the insoluble solid in the underflow is constant, it is convenient to define the ratio

$$R = \frac{amount\ of\ solvent\ in\ overflow}{amount\ of\ solvent\ in\ underflow} = \frac{V_n}{L_n} \qquad (7)$$

If perfect mixing occurs in each stage and the solute is not adsorbed preferentially at the surface of the solid, then the concentration of the solution in the underflow is the same as that in the overflow and

$$R = \frac{amount\ of\ solute\ in\ overflow}{amount\ of\ solute\ in\ underflow} = \frac{V_n x_{An}}{L_n y_{An}} \qquad (8)$$

Referring to Figure 2, by considering solute mass balances over n, $(n-1)$, … 2, 1 units in turn and eliminating intermediate solute mass fractions and flow rates, the amount of solute associated with the leached solid may be calculated in terms of the composition of the solid and solvent streams fed to the system. The resulting equation is (2)

$$L_0 y_{A0} = \frac{R^{n+1}-1}{R-1} L_n y_{An} - \frac{R^n - 1}{R-1} V_{n+1} x_{An+1} \qquad (9)$$

In many cases the amount of solute associated with the leached solid must not exceed a certain value, and it is possible to compute directly the minimum number of units needed by putting $n = N$.

Alternative approaches are to be found in the literature. Derivations of the above equations are given in numerous texts (2, 10–12), which also describe graphical or analytical solutions to the problem. Many of these have direct analogues in other separation processes such as distillation (qv) and liquid–liquid extraction, and use plots such as the McCabe-Thiele diagram or Ponchon-Savarit diagram.

3.3. Countercurrent Leaching With Variable Underflow

In practice most cases of interest exhibit a variable underflow rate, which is normally greatest at that point in the process where the solute concentration in the solvent is highest. In some leaching operations the viscosity and density of the solution changes appreciably as the solute concentration. Consequently, the operating line derived from mass balance equations for the McCabe-Thiele diagram has a slope which varies from stage to stage, and equation 9 is no longer valid. In the lower numbered stages the solute concentrations are higher and the underflows may retain more solution than the underflows from the higher numbered stages. Clearly, if the underflow rate L_n varies then so does the overflow rate. As in the previous case, each unit is assumed to be well mixed and the solute mass fractions in the overflow and underflow are related by:

$$x_{An} = y_{An} \qquad (10)$$

The solute mass fraction in the overflow solution from the first unit ($n = 1$) is

$$x_{A1} = \frac{V_{n+1}x_{An+1} + L_0 y_{A0} - L_n y_{An}}{V_{n+1} + L_0 - L_n} \qquad (11)$$

and the solute mass fraction in the solution fed to unit n is

$$x_{An+1} = \frac{V_{n+1}x_{An+1} - L_n y_{An} + L_n y_{An}}{V_{n+1} - L_n + L_n} \qquad (12)$$

It is often the case that all quantities in equation 11 except x_{A1} are known. If, instead of V_{n+1}, the concentration of the solution leaving the system is specified, then equation 11 can be used to calculate V_{n+1}.

4. Extractors

Calculations serve as a guide to the analysis of an extraction plant and, as for the analysis of equipment performance in any other sphere of process engineering, these may be supplemented by empirical correlations or process models pertinent to the particular equipment under consideration. Another process which usually deserves special attention is that needed for solvent regeneration and for solute recovery. Solvent recovery (qv) is often energy intensive and a full process energy analysis is recommended to reduce costs. Recovery of organic solvents from the exhausted solids is also important and can be more troublesome than recovery from a liquid, and consideration should be given to the use of superheated solvent vapor for this purpose (13) (see Process energy conservation).

Extractors rely on either percolation or agitation to ensure intimate contact between the solids and solvent. Percolation and extraction rate data provide guidance on whether extraction should be by percolation or immersion and what extraction time is needed to give an acceptable approach to equilibrium. For a percolation system to be viable the extraction rate needs to be high, as the solvent residence time is often relatively short, although where percolation rates are so high (residence times very short) that extraction becomes inefficient, upward flow through the bed of solids can sometimes be advantageous (14). A percolation process can be carried out either stagewise or in a differential contactor; for an immersion process stagewise contact is often more practicable, particularly where a low extraction rate requires an extended residence time or multiple contact with the solvent. Under these circumstances, or when extraction is accompanied by chemical reaction, a countercurrent multistage operation is often beneficial. When percolation and extraction rates are being measured for equipment specification and sizing purposes, the conditions in the test extractor should match as closely as possible those anticipated in the full-scale extractor in order to provide the most reliable data.

Extractors often contribute substantially to the capital and operating costs of a plant, which provides the impetus to seek ways to reduce the extraction load in order to increase extractor capacity and reduce specific solvent requirements. When the feed material is of plant origin and the solute is contained in cells that can be ruptured by heat or pressure, pre-treatment frequently involves removing part of the solute by pressing. The variety of extractors used in liquid–solid extraction is diverse, ranging from batchwise dump or heap leaching for the extraction of low grade ores to continuous countercurrent extractors to extract materials such as oilseeds and sugar beets where problems of solids transport have dominated equipment development.

4.1. Batch Extractors

Coarse solids are leached by percolation in fixed or moving-bed equipment. Both open and closed tanks (qv) having false bottoms are used, into which the solids are dumped to a uniform depth and then treated with the solvent by percolation, immersion, or intermittent drainage methods.

The pot extractor is a batch extraction plant in which extraction and solvent recovery from the exhausted solids can be carried out in a single vessel. These extractors are normally agitated vessels having capacities in the range of 2 to 10 m^3, beyond which the battery system becomes a preferred technical alternative.

The diffuser is a closed percolation vessel which is used when the pressure drop is too high for gravity flow of solvent, when evaporative losses of solvent would be too high, or when it is necessary to use elevated temperatures. The solvent is circulated through the tank by pumping, or leaching may be achieved without solvent circulation. A diffuser battery is a semibatch extraction system operating on a cyclical basis. The system comprises a battery of diffusers or vessels, each of which is charged with the solids to be extracted. Fresh solvent is fed to the first one or two vessels, sometimes with the solvent being heated before being fed to the unit. The underflow from one unit is fed to the next, again with the option of interunit heating available. The underflow from the final unit is a solute-rich solution. The actual number of diffusers in the battery depends on extraction and equilibrium conditions, but an additional diffuser above the estimated number is required to permit cyclical operation. When a battery consists of more than four units a close approximation to countercurrent flow is achieved, and owing to the cyclic nature of the operation each unit changes its position in the extraction sequence at each cycle changeover. In the cyclic operation, the most exhausted diffuser is bypassed and emptied, and an empty one is charged with fresh solids. This rather cumbersome plant layout is used, for example, for the extraction of coffee solubles using hot (150–180°C) water, but can be largely replaced by fully continuous devices.

4.2. Continuous Extractors

Continuous extractors are available in a variety of forms. The main difference between them is the way by which the solids are transported through the equipment. For convenience the method of solids transport is used as a means of equipment classification.

Moving-bed percolation systems are used for extraction from many types of cellular particles such as seeds, beans, and peanuts (see Nuts). In most of these cases organic solvents are used to extract the oils from the particles. Pre-treatment of the seed or nut is usually necessary to increase the number of cells exposed to the solvent by increasing the specific surface by flaking or rolling. The oil-rich solvent (or miscella) solution often contains a small proportion of fine particles which must be removed, as well as the oil separated from the solvent after leaching.

The Bollman extractor (15) (Fig. 3) is a moving-bed, perforated-basket type of extractor. The solids are loaded into baskets fixed to a chain conveyor in a closed vessel. Solid is fed to the top basket on the downward side of the conveyor and is discharged from the top basket on the upward side. Fresh solvent is sprayed on the solid about to be discharged, leaving some time for drainage from the basket before discharge is effected, and passes downward through the baskets to effect a countercurrent flow. The partially rich solvent (half-miscella) from the bottom of the upward side is pumped to the basket at the top of the downward side, from which solvent flows from basket to basket in cocurrent fashion. The final solvent solution, miscella, is collected from the bottom of the downward side. Control of flake size during pre-treatment is desirable, as is control of the thickness and bulk density of the bed. A typical extractor moves at about 0.3 m/s, each basket contains some 350 kg of seeds, about equal masses of seeds and solvent are used, and the miscella contains about 25% oil by mass (2). Advantages of this design of extractor are that a solids-free miscella can be obtained, the residue is well drained when the equipment is properly controlled, and large quantities of solids can be extracted continuously.

The Rotocel extractor (16) achieves countercurrent extraction through a sequence of discrete liquid–solid contacts. The solids to be extracted are fed continuously as a dry material or as a slurry to sector-shaped cells arranged around a horizontal rotor. Each cell has a perforated base which allows easy drainage of solvent into a basin at the base of the cell from which the solvent is pumped into the next cell in the countercurrent direction. Fresh solvent is supplied to the last cell, which also occupies a larger sector than the other cells to allow for drainage of the extracted solids prior to discharge. The miscella is filtered by the bed of solids in each cell, and miscella from such rotary-type extractors can be expected to contain less than 5 ppm suspended solids, sometimes effecting a saving on the cost of subsequent solid–liquid separation equipment.

Tipping pan and horizontal filters are also used for leaching: the modus operandi of the Rotocel extractor resembles that of a tipping pan filter, although the details of its design differ slightly.

An alternative tower design, the Bonotto extractor (15) (Fig. 4), is a series of slowly rotating horizontal trays equispaced vertically in a tall cylindrical vessel. The solid is fed continuously close to the outside edge on the top tray and a stationary scraper attached to the vessel causes the solid to cross the tray. The solid then falls through an opening onto the tray beneath, where another scraper moves the solid across the tray in the opposite direction toward a similar opening near the periphery of this tray. This sequence of moving the solid across each plate in opposite directions on alternate plates is continued until the solid reaches the bottom of the tower. It is then transported from the tower by a screw conveyor, although alternative types of solids conveyor could be used. The solvent is fed to the bottom of the vessel and flows upward to give a flow countercurrent to the solids flow direction. Clearly the upward velocity of the solvent should be lower than the fall velocity of the solids to prevent entrainment of the solids, and the density of the solvent may change markedly up the column as the concentration of solute increases.

Endless belt percolation extractors (Fig. 5) such as the uncompartmented de Smet belt extractor and the compartmented Lurgi frame belt extractor are similar in principle and closely resemble a belt filter, and are probably the simplest type of percolation extractor from a mechanical point of view. These are fitted with a slow-moving perforated belt. The belt is made from steel mesh cloths when the solids are fine, or coarser screens when the solids are larger, and is attached to chains which pass over sprockets at each end of the extractor. The solid is fed from a hopper at one end of the extractor to the moving belt, and the bed height is controlled by an adjustable damper at the outlet of the feed hopper. The two side walls of the extractor provide support for the bed on the moving belt. Fresh solvent is fed by spraying it onto the bed close to the discharge end of the belt, but leaving sufficient distance for adequate drainage of the bed prior to discharge. Miscella draining from the bed is collected in a pan below the belt and circulated back to be sprayed onto the bed at a point closer to the solids-feed end of the belt; this process is repeated to achieve extraction operating with a countercurrent flow. The top of the bed is scraped by a hinged rake which has two functions: (*1*) it prevents a layer of fine solids from accumulating at the top of the bed thereby reducing permeability, and (*2*) it form a solids pile which helps to prevent intermingling of miscella from different feed points at the surface of the bed.

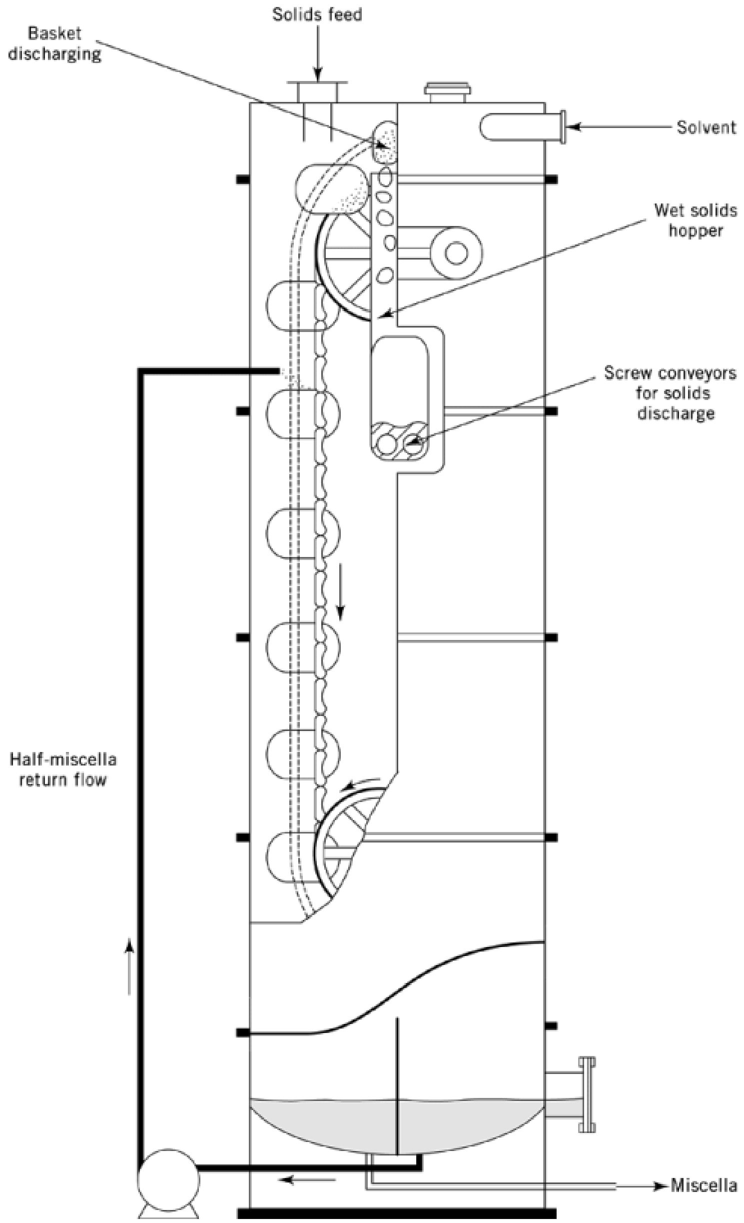

Fig. 3. Bollman moving-bed-type extractor.

The belt is effectively washed twice: once by fresh solvent just after the solids discharge point, and then at the other end of the belt return by miscella. The extraction time and percolation rate determine the belt speed and the amount of drainage area, and hence linear length of belt, required. These parameters control the plant capacity as the bed height is fixed by the mechanical design of the extractor.

Immersion extraction systems are useful in handling finely ground material or when the percolation rate through the material to be extracted is too rapid to allow effective diffusion from the solids. These systems

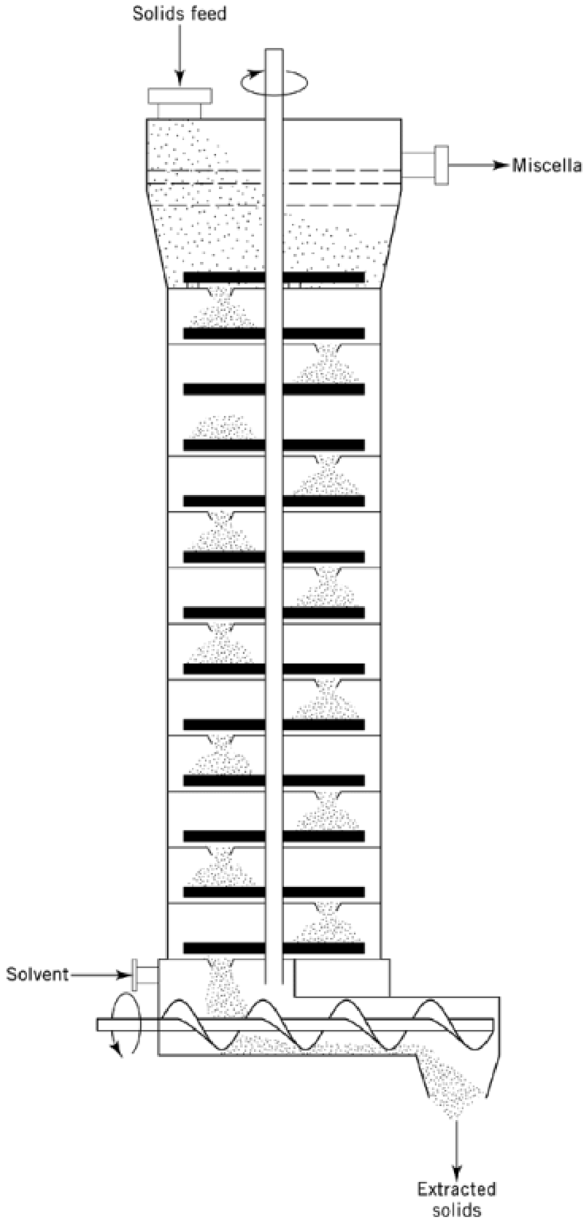

Fig. 4. Principles of the Bonotto-type extractor.

are applied extensively in the sugar industry, in extraction from oilseeds having a high oil content, and from plant materials. The stepwise extraction by immersion, or continuous countercurrent leaching systems, can be carried out by a number of techniques analogous to the mixer–settler widely used in liquid–liquid extraction. The method is only viable if the solids settle more or less completely so that a clear supernatant liquor remains for decantation and when the slurry formed is pumpable, but it is nonetheless a most important method of leaching. If this is not so, other solid–liquid separation stages, such as filtration (qv) or centrifugation (see

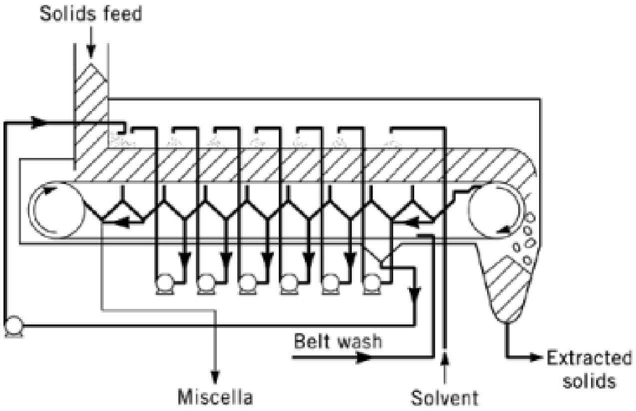

Fig. 5. Principles of the belt-type extractor.

Separation, centrifugal), have to be considered. However, such mixer–settler methods are continuous only by virtue of repeating a sequence of similar stages to achieve a given degree of extraction. More fully continuous methods of extraction were designed as tower systems and later as screw conveyor systems as effective methods of solids transport became reliable.

Continuous countercurrent decantation systems are not uncommon, employing a cascade of thickeners to wash solute from fine particles or to wash the solids formed by chemical reactions. The capacity of each thickener in the cascade is designed so that the residence time of the particles is long enough to allow the reaction to go to completion or to allow an acceptable degree of leaching to be achieved. Interthickener filtration permits use of much smaller volumes of solvent, sometimes also allowing greater removal of solute. The drawback of interstage filtering is that filters tend to be more expensive than equivalent thickeners, but this also has to be set against the smaller amount of space required for the whole installation.

The BMA diffusion tower (17), in common with some other tower systems, employs a central shaft fitted with a series of inclined plates or wings that direct movement of the solids. The tower shell is fitted with staggered guide plates for the same purpose. The solids are fed to the bottom of the tower and transported upward. Such units are found most widely in sugar beet refining. Tower heights of 10 to 15 m are used, with the diameter being dependent on the solids throughput capacity. For a capacity of 3000 metric tons of beet per day (17), a tower diameter of 5.5 m is required and the power consumption is of the order of 40 kW. In general, immersion extractors take up less space than the percolation types and have a lower power consumption.

Immersion-type extractors have been made continuous through the inclusion of screw conveyors to transport the solids. The Hildebrandt immersion extractor (18) employs a sequence of separate screw conveyors to move solids through three parts of a U-shaped extraction vessel. The helix surface is perforated so that solvent can pass through the unit in the direction countercurrent to the flow of solids. The screw conveyors rotate at different speeds so that the solids are compacted as they travel toward the discharge end of the unit. Alternative designs using fewer screws are also available.

The De Danske Sukkerfabriker (DDS) diffuser extractor (Fig. 6) is a relatively simple version of this family of machines, employing a double screw rotating in a vessel mounted at about 10° to the horizontal. The double screw is used to transport the solids up the gradient of the shell, while solvent flows down the gradient. Equipment using a single screw in a horizontal shell for countercurrent extraction of solids under pressure has been described (19).

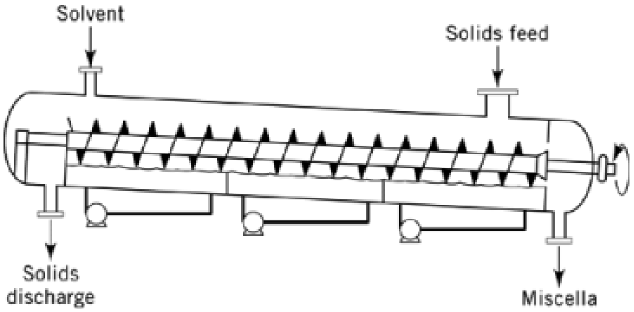

Fig. 6. Principles of the De Danske Sukkerfabriker (DDS) diffuser extractor.

5. Safety and Environmental Considerations

Solvent flammability, the solvent, and dust loading in the atmosphere of the working environment and of the products in the case of edible materials are the main factors that constitute health and safety hazards in extraction plants (20). General safety and environmental standards must therefore be applied (see Plant safety) and due recognition taken of the most recently published national regulations relating to acceptable threshold limit values (TLVs) for solvents and dusts. The permissible levels of solvent residues and emissions have repeatedly been lowered in recent years, making it important to ensure that the most up-to-date regulations are available to and acted upon by plant designers and engineers.

Disposal of exhausted solids can be easily overlooked at the plant design stage, particularly when these have no intrinsic value; alternative disposal methods might include landfill of inert material or incineration, hydrolysis, or pyrolysis of organic materials. Liquid, solid, and gaseous emissions are all subject to the usual environmental considerations.

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Related Articles

Extraction, liquid-liquid; Metallurgy, extractive; Solvents, industrial; Mineral recovery and processing