## Lecture 13

## Leaching and Washing

Leaching, sometimes called solid-liquid (or liquid-solid) extraction, involves the removal of a soluble fraction (the solute or leachant) of a solid material by a liquid solvent. The solute diffuses from the solid into the surrounding solvent. Either the extracted solid fraction or the insoluble solids, or both may be the valuable product. Leaching is widely used in the metallurgical, natural product, and food industries. In the former, leaching may involve oxidation or reduction reactions of the solid with the solvent. Equipment is available to conduct leaching under batch, semicontinuous, or continuous operating conditions. Effluents from a leaching stage are essentially solids-free liquid, called the oveflow, and wet solids, called the undeflow. To reduce the concentration of solute in the liquid portion of the underflow, leaching is often accompanied by two or more countercurrent-flow washing stages. The combined process produces a final overflow, called the extract, which contains some of the solvent and most of the solute; and a final underflow, called the extracted or leached solids, which are wet with almost pure, remaining solvent. Ideally, the soluble solids are perfectly separated from the insoluble solids, but solvent is distributed to both products. Additional processing of the extract and the leached solids is necessary to recover the solvent for recycle.

Some industrial applications of leaching include: (1) removal of copper from ore with sulfuric acid, (2) recovery of gold from ore with sodium-cyanide solution, (3) extraction of sugar from sugar beets with hot water, (4) extraction of tannin from tree bark with water, and (5) the removal of caffeine from green coffee beans with supercritical  $CO_2$ .

As an example of leaching, consider the extraction of vegetable oil from soybeans with a commercial hexane solvent in a pilot-plant size countercurrent-flow, multistage leaching unit, as described by Othmer and Agarwal. Although edible oils can be extracted from a number of different field and tree crops, including coconuts, cottonseeds, palm, peanuts, grapeseed, soybeans, and sunflower seeds, the highest percentage of edible oil is from soybeans. In 1979-1980, world production of soybean oil was 14 million metric tons. Oil from soybeans is high in polyunsaturated fats and, thus, is less threatening from a cholesterol standpoint. When the oil content of seeds and beans is high, some of the oil can be removed by compressing the solids in a process known as expression, as discussed in Perry and Green. For soybeans, whose oil content is typically less than 0.30 lb per lb of dry and oil-free solids, leaching is a more desirable technique than expression because a higher yield of oil can be achieved by leaching.

The soybeans are dehulled, cleaned, cracked, and flaked before being fed to the extractor. Typically, the cleaned soybeans contain 8 wt% moisture and 20 wt% oil. The dry and oil-free soybeans have a particle specific gravity of about 1.425 and the oil has a specific gravity of 0.907 with a viscosity at 25°C of 50 cP. Approximately 50% of the flake volume is taken up by oil, moisture, and air. It might be expected that whole soybeans, rather than flakes, might be fed to the extractor, with leaching

taking place by molecular diffusion of the solvent uniformly into the seed, followed by diffusion of the oil through the solvent and out of the seed. If so, the mass-transfer process within the seed could be modeled with Fick's second law. However, experiments by King, Katz, and Brier found that although extraction of oil with solvent in uniformly porous inorganic solids, like porous clay plates, obeyed Fick's law of molecular diffusion, extraction of oil from soybeans did not, presumably because of the complex internal structure of soybeans. Furthermore, Othmer and Aganval, using whole and carefully cut half soybeans, found that diffusion was extremely slow. After 168 hours in contact with hexane, less than 0.08% of the original oil in the whole beans and less than 0.19% of the oil in the half beans was extracted. Such a slow diffusion rate for particles that are approximately 5 mm in diameter is probably due to the location of the oil within the insoluble cell walls, requiring that the oil pass through the walls by slow osmotic pressure differences.

The extent and rate of extraction of the oil is greatly enhanced by flaking the soybeans to thicknesses in the 0.005-0.02-in. range. The flaking process ruptures the cell walls, greatly facilitating contacting of the oil with the solvent. Using trichloroethylene or n-hexane as the solvent, with flakes of diameters ranging from 0.04 to 0.24 in., approximately 90% of the oil can be extracted in 100 minutes. The ideal solvent for commercial applications of the leaching of soybeans should have a high solubility of the oil to minimize the amount of solvent needed, a high volatility to facilitate the recovery of solvent from the oil by evaporation or distillation, nonflammability to eliminate the chance of fire and explosion, low cost, ready availability, chemical stability, low toxicity, and compatibility with inexpensive materials of construction to minimize or eliminate corrosion. Although in a number of respects, especially nonflammability, trichloroethylene is an ideal solvent, it is now considered to be a very hazardous chemical because of its high toxicity. The favored solvent is commercial hexane (mostly n-hexane), which presents a fire hazard but has a low toxicity.

The pilot-plant leaching unit used by Othmer and Agarwal, and known as the Kennedy extractor, is shown in Figure 1. The soybeans enter continuously at the low end and are leached in a countercurrent cascade of tubs by hexane solvent, which enters at the upper end. The flakes and solvent are agitated and the underflows are pushed uphill from one tub to the next by slowly rotating paddles and scrapers, while the overflows move downhill from tub to tub. The paddles are perforated to drain the solids when they are lifted above the liquid level in the tub by the paddle. The cascade can contain as many tubs as required; Othmer and Agarwal used 15. Soybean flakes of 0.012 in. average thickness and containing, on the average, 10.67 wt% moisture and 0.2675 g oil/g dry oil-free flakes were fed to the Kennedy extractor at a continuous flow rate of 6.375 lbh. The solvent flow rate was 10.844 lb/h. Leaching took place at ambient conditions. After 11 hours of operation, a steady state was achieved that delivered an extract, called a miscella, of 7.313 lb/h, containing 15.35 wt% oil. The leached solids contained 0.0151 g oil/g dry oil-free flakes. Thus, 94.4% of the oil was extracted. The residence time in each tub was 3 minutes, giving a total residence time of 45 minutes. From these data, a mass-balance

check can be made for oil and solvent, and the liquid-to-solids ratio in the leached solids can be estimated. These calculations are left as an exercise.



Figure 1 - Kennedy extractor for leaching of oil from soybeans