

LEACHING METHODS APPLIED TO THE CHARACTERIZATION OF COAL UTILIZATION BY-PRODUCTS

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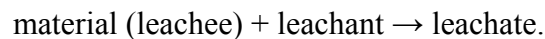
Abstract

There are more than one hundred leaching methods, but there is no agreement on which method is most appropriate to estimate the environmental consequences of the use or disposal of coal utilization by-products (CUB). Leaching methods are often categorized by whether the leaching fluid is a single addition (static extraction tests) or is renewed (dynamic tests). Methods can also be classified as batch leaching in which the sample is placed in a given volume of leachant solution, as column or flow through systems, and as bulk or flow around systems for monolithic samples. Commonly used methods developed by EPA or promulgated by ASTM, as well as methods developed specifically for CUB, are discussed.

Introduction

Leaching is a method to remove soluble components from a solid matrix. A survey of the literature identified over 100 leaching methods (Hesbach and Lamey, 2001). Given the plethora of methods, the question often arises as to what is the “best” method. The simplest answer is “It depends.”

Describing leaching by a very simple equation:



It can be assumed that the material to be leached is known, although its physical and chemical/mineralogical properties will affect the final result. The purpose or what you expect to find in the leachate will determine the selection of a leachant and also the conditions of the test.

Several common leaching methods are regulatory methods, mandated to characterize materials; others are approved by organizations for establishing compliance to particular specifications. Some methods are intended to mimic natural conditions or to obtain information about the nature of the extractable material within a particular solid. The methods vary in the mass and particle size of the sample, the type and volume of leachant solution(s), the leachant delivery method, and time. Most procedures are performed at ambient temperature, although a few decrease the time required to solubilize components by increasing the temperature. Although many were developed for application to municipal solid waste or industrial wastes, most leaching methods have been applied to a variety of materials, including coal utilization by-products (CUB).

For a simple, one compound material, leaching is relatively simple. It depends on the pH and composition of the leachant, the solubility of the chemical compound, and surface area of the

solid. This can be fairly well described by a shrinking core model (Batarseh et al, 1989) or and adsorption/desorption model (Chaiken, 1992). Most natural materials, including CUB, are not that simple, and leaching behavior is controlled by these and other variables.

With respect to leaching, it is important to recognize that CUB, particularly fly ash, is not a homogeneous material. Its elemental and mineralogical composition and its physical properties are a function of the original coal, the combustion temperature and post-combustion cooling rate (Kim, 2002). Volatilization, melting, decomposition, and the formation of new minerals, as well as oxidation, are the mechanisms that transform the minerals in coal.

Concentration is one of several factors in determining leaching potential; volatility, and solubility also influence leaching potential. Most elements, particularly trace elements, in CUB are only slightly soluble (Kim et al, 2003). Heavy metals are most soluble in acidic solutions, while those elements that form oxyanions are more soluble at high pH. There is no particular element or group of elements that is characteristic of CUB leaching potential. Elemental solubility appears to be an independent variable.

Solubility is also a function of speciation within the ash. The primary minerals in CUB are silicates (quartz), alumino-silicates (clays and feldspar), oxides (hematite), and sulfates (gypsum, anhydrite). Silicates and alumino-silicates are comparatively insoluble. Oxides tend to be only slightly soluble, while sulfates are more soluble. The silicate/non-silicate distribution of various cations has been shown to influence elemental solubility (Kim & Kazonich, 2004); the condensation of mixed particles (Osgood-Kutchko and Kim, 2002) also affects elemental solubility. Volatile inorganic elements may condense as compounds forming surface coatings on other particles.

The physical characteristics of combustion residues include particle size, particle shape or morphology, hardness, and density. These properties are a function of the particle size of the feed coal, the type of combustion, and the particulate control device. Due to the high temperature of P.C. combustion, fly ash particles tend to melt and condense as spheres with a diameter of less than 0.010 mm. The spherical shape of fly ash particles results in a minimum surface area, which reduces the potential number of leaching sites.

Choosing the most effective leaching method for CUB must consider the chemical and physical properties of fly ash particles which are a function of the mineral matter in the coal, the combustion conditions and post-combustion cooling. Since these factors may be unknown, a variety of leaching methods have been proposed based on the type of information desired or on particular conditions the method simulates.

Leaching Methods

Summaries of many of the more commonly used leaching methods have been given by Sorini (1997), Wilson (1995), and Kim (2003). The International Ash Working Group (IAWG) based in Europe has done extensive work on the integration of a variety of tests into a comprehensive leaching system (Eighmy and van der Sloot, 1994; van der Sloot, 1998).

Leaching methods are often categorized by whether the leaching fluid is a single addition (static extraction tests) or is renewed (dynamic tests). Methods can also be classified as batch leaching in which the sample is placed in a given volume of leachant solution, as column or flow through systems, and as bulk or flow around systems for monolithic samples. Results are generally reported as a concentration, sometimes as the concentration in the leachant solution (mg/L) or as the leached concentration from the solid (mg/kg). In many methods, the liquid to solid ratio (L/S) is used to quantify the volume of leachant with respect to the amount of solid sample, usually as mL/g or L/kg. Representative methods are listed in Table 1.

Batch Methods

Batch leaching methods are those in which a sample is placed in a given volume of leachant solution for a set period of time. Most of these methods require some type of agitation to insure constant contact between the sample and the leachant. At the end of the leaching period, the liquid is removed and analyzed.

The most commonly used batch leaching methods are the Toxicity Characteristic Leaching Procedure (TCLP), the Extraction Procedure Toxicity Test (EPTOX), the Synthetic Precipitation Leaching Procedure (SPLP), the Standard Test Method for Shake Extraction of Solid Waste with Water (ASTM-D3987), and the California Waste Extraction Test (CA WET). The Leachate Extraction Procedure approved by the Canadian General Standards Board (CGSB, 1987) and the Leachate Extraction Procedure (LEP, 1993) of Ontario are very similar to EPTOX. The parameters for these methods are compared in Table 2.

In serial batch methods, a sample of waste is leached successively with fresh aliquots of the same leaching fluid. This method is intended to eliminate the effect of concentration on solubility and to simulate long-term exposure to the leachant solution. These methods include EPA's Multiple Extraction Procedure (MEP), the Standard Test Method for Sequential Batch Extraction of Waste with Acidic Extraction Fluid (ASTM D 5284), and the Standard Test Method for Sequential Batch Extraction of Waste with Water (ASTM D 4793).

Sequential leaching tests use a single sample that is leached by a series of different leaching fluids. The Availability Test for Granular Materials (NEN 7341), a Dutch Standard leaching test, leaches a material at pH 4 and at pH 8; the two pH's are intended to bracket the range found naturally in the environment. In more complex sequential extractions, the constituents extracted with a particular leachant are associated with a mineral phase or chemical species. Palmer at the USGS developed a Sequential Leaching Method as a rapid indirect method of determining the modes of occurrence of trace elements in coal (Palmer et al, 1999). Like the later USGS sequential extraction procedure, Tessier (1979) uses a series of four extractant fluids to dissolve metals associated with particular ligand phases in a complex sample. A modified Tessier procedure uses aqua regia in place of hydrofluoric/nitric acid (Raksasataya et al, 1996). A three-step sequential extraction procedure (Quevauviller et al, 1997) developed by the commission of the European Communities Bureau of Reference has also been modified to include aqua regia digestion of the residual material (Raksasataya et al, 1996). A Short Sequential Procedure uses two steps to assess the lability of heavy metals in soil particles (Maiz et al, 2000).

Column Methods

Column leaching tests are designed to simulate the flow of percolating groundwater through a porous bed of granular material. The flow of the leaching solution may be in either down-flow or up-flow direction, and continuous or intermittent. The flow rate is generally accelerated when compared to natural flow conditions. However, it should be slow enough to allow leaching reactions to occur. A basic assumption in column leaching is that the distribution of the leaching solution is uniform and that all particles are exposed equally to the leachant solution.

Precipitation or sorption within the column may affect the results.

The Standard Test Method for Leaching Solid Material in a Column Apparatus (ASTM D-4874) is intended to maximize the leaching of metallic species from a solid. The aqueous fluid passes through particles of known mass in a saturated up-flow mode. The Dutch Standard Column Test (NEN 7343) is also an up-flow application, and the Nordtest Column Method (NORDTEST, 1995) is similar to the Dutch Column test, except that column dimensions are optional. The up-flow column procedures are designed to insure that the leachant solution is equally distributed throughout the column. However, gravity flow columns can also be used to study leaching of porous media. Column experiments more closely approximate the particle size distribution and pore structure, leachant flow and solute transport found in the field (Zachara and Streile, 1990).

The NETL column leaching system is a continuous gravity flow system in which five leachant solutions are used to simultaneously leach 1 kg samples of a CUB (Kim and Sharp, 1995; Kazonich and Kim, 1997). The leachant solutions are sulfuric acid; synthetic precipitation, sodium carbonate, acetic acid, and deionized (DI) water.

Column experiments can be conducted in both saturated and unsaturated conditions.

Unsaturated conditions are usually intended to mimic vadose zone placement. Intermittent addition of a given volume of leachant solution at the top of the column can provide uniform distribution of the fluid and approximate a constant fluid front moving through the unsaturated column. Saturated columns are obtained by a constant fluid flux, and allowing the fluid to pond at the top of the column. Variables, such as leachate collection, sampling frequency, leachant flow rate, and duration of the experiment, are determined by the experimental objectives.

Monolithic and Bulk Methods

Monolithic leaching methods are used to evaluate the release of elements from a material that normally exists as a massive solid, cement for example, and are frequently used to characterize the release of pollutants from stabilized waste materials. The release of an element is a function of the exposed surface area as opposed to the mass. Flow-around systems relate solubility to the surface area of a particular volume. Flow-through systems also consider the internal pore surface. And some systems take into account the rate of diffusion of the leachant solution into the pores.

In static monolithic leaching, a particle of regular geometry and known surface area is immersed in a volume of leachant solution. The same leachant solution is sampled at defined intervals and replaced with fresh solution (Hoberg et al, 2000).

The flow through leaching test (Poon et al, 2001) is used to characterize leaching from a waste that is more permeable than the surrounding material. The solid sample is placed in a flexible wall permeameter, and in this method, the leaching solution is DI water at a mean flow rate of .0166 mL/min at a pressure of 400 kPa.

Bulk leaching generally refers to leaching large samples, either in a large column or in heaps. They are either hydro-metallurgical systems (Fleming, 1996) or are used in a research setting to leach a non-homogeneous sample with a large particle size (Dalverny et al, 1996). Neither system is particularly applicable to CUB.

The ASTM Static Leaching of Monolithic Waste Forms (ASTM C 1220) is intended to evaluate the durability of radioactive waste in glasses and ceramics. The testing period varies from 7 days to 1 year or more. Water, de-aerated water, brine, and silicate water (NaHCO_3 and silicic acid) or site-specific repository waters may be used as the leachant solution. The sample is a single piece of regular geometry so that the surface area can be determined. The volume of the reaction vessel, between 20 mL and 1 L, sets the size of the sample. The International Standards Organization also has a leaching protocol developed for application to solidified radioactive waste (ISO 6961, 1982).

Combined Methods

The International Ash Working Group (IAWG) has designed a combined leaching protocol to quickly determine the total leachable elements in a material and to estimate metal release in a normal environmental setting (van der Sloot et al, 1994; van der Sloot, 1998). It combines the sequential batch availability test (NEN 7341) with a serial batch extraction using water. A two-step availability test of fine-grained material, at a L/S of 50 and controlled pH's of 4 and 8, is used to determine leachability at the upper and lower pH limits found in natural environments. From the total acid consumption, the acid neutralization capacity of the material is estimated. Total elemental release as a function of time is estimated by leaching at several L/S values between 1 and 100 in a serial batch test with water. The release of contaminants is usually expressed in mg/kg leached against the L/S ratio.

The US Environmental Protection Agency Office of Solid Waste (EPA/OSW) has proposed a three-tier approach to the environmental assessment of CUB, particularly for the release of mercury (Kosson, 2002). It includes batch leaching at 11 pH's between 3 and 12 at a L/S of 10, batch leaching with DI water at 5 L/S ratios, and a monolithic procedure using 10 mL of DI water per cm^2 of surface area of a solidified cylinder or cube. A similar combination of pH dependent release and long term estimated release has been developed at NETL (Hesbach and Kim, 2005)

Conclusion

The selection of a leaching method is not a simple or trivial task. The ASTM E 50.03 committee is currently developing a standard guide or procedure to identify the best available leaching tests for specific materials or material types (Pflughoefft-Hassett, 2004). In the absence of an

accepted protocol, the project objective and the type of data desired determine what method is most appropriate. Critical variables include the sample size and particle size distribution, the leachant volume and pH, and the duration of the leaching test. The use of regulatory or standard methods by different laboratories does not always produce duplicate results. Even when tests are performed with the same methods, extraneous variables, such as analytical sensitivity and sample inhomogeneity, may influence the reproducibility of the results. Also, compliance tests and standard methods are not necessarily appropriate as leaching tests to simulate natural processes, to obtain data on reaction mechanisms or to unravel complex solubility relationships. Limited comparative studies of leaching methods (Heaton, et al, 1981; Mason and Carlile, 1986; Zachara and Streile, 1990) found that various methods could generate reproducible data, but there was no consistent correlation in the data generated by various methods. To evaluate four commonly used or proposed leaching methods for CUB, five laboratories are participating in an informal inter-laboratory comparison (Hesbach et al, 2005). The results will be compared on total elemental solubility and on internal and comprehensive reproducibility.

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Table 1. Representative Leaching Methods

| | Regulatory | Standard | Research (Other) |
|-------------------------------|------------|------------|---------------------|
| Batch <i>Static</i> | TCLP | ASTM 3987 | |
| | SPLP | | |
| <i>Dynamic</i> | Ca WET | | |
| | | | |
| <i>Serial</i> | MEP | ASTM D5284 | |
| | | ASTM D4793 | |
| <i>Sequential</i> | | | |
| | NEN7341 | | Palmer (USGS) |
| | BCR | | Tessier |
| Column | NEN7343 | ASTM D4874 | Kim (NETL) |
| | NORDTEST | | |
| Bulk <i>Static</i> | | ASTM C1220 | |
| | | ISO 6961 | |
| <i>Dynamic</i> | | ANS-16.1 | Heap |
| | | | Trickle Bed Reactor |

Table 2. Characteristic Parameters of Static Batch Leaching Methods

| Method | Leachant | Sample size, g | pH | L/S ¹ | Time, hr |
|-------------|------------------------------------------------|----------------|------|------------------|----------|
| ASTM D-3987 | Water | 70 | | 20 | 18 |
| EPTOX | Water | 100 | 5.0 | 20 | 24 |
| SPLP | Water acidified with nitric and sulfuric acids | 100 | 4.2 | 20 | 18 |
| TCLP | Acetic Acid or Acetate Buffer | 100 | 2.88 | 20 | 18 |
| CA WET | 0.2 M sodium citrate | 50 | 5.0 | 10 | 48 |
| LEP | Water acidified with 0.5 N acetic acid | 50 | 5.0 | 16 | 24 |

¹L/S: Liquid to Solid ratio, L/kg