IMPROVED LEACHING TEST METHODS FOR ENVIRONMENTAL ASSESSMENT OF COAL ASH AND RECYCLED MATERIALS USED IN CONSTRUCTION

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SUMMARY: Changes in air pollution control at coal-fired power plants will result in lower emissions of mercury and other pollutants. Fly ash, flue gas desulfurization gypsum, and other air pollution control residues are used in agricultural, commercial, and engineering applications. Research is being conducted to evaluate the fate and environmental stability of mercury and other metals found in coal combustion residues as a result of changes in air pollution control at coal-fired power plants in the U.S. The pathway of most concern is leaching of metals to groundwater and surface water. Improved leaching test methods are being used that evaluate the range of field conditions that coal ash is exposed to. Results are considered more representative of actual field conditions than single-point pH tests which have historically been used in evaluating use of CCRs and other secondary materials. This paper provides an overview of the leaching test procedures being used in this research and application to a range of CCRs resulting from more stringent air pollution control at coal-fired power plants. The results of this research are helping to ensure that the use of fly ash or other secondary materials are being managed in a manner that is protective of human health and the environment.

1. INTRODUCTION

In the U.S., approximately 40% (51 out of 126 million tons) of all coal combustion residues (CCRs) produced as of 2006 are being "beneficially" used versus managed in landfills or surface impoundments. The physical and chemical characteristics of CCRs make them suitable as replacements for materials used in a wide range of products including cement, concrete, road base, wallboard, and even toothpaste. For example, high fly ash-content concrete can be used for high performance, long-life pavements which are designed to last 50 years which is twice the lifetime of conventional pavements (U.S. EPA, 2009). Recycling CCRs can help conserve natural resources and energy, as well as decrease the amount of CCRs being land disposed.

Figure 1 presents a summary of the primary uses by CCR type (e.g., fly ash, FGD gypsum, bottom ash).

Of the 72 millions tons of fly ash produced in 2006, 44% (or 32 million tons) was used in commercial applications including the production of cement-related products, structural fill, and highway construction [American Coal Ash Association (ACAA), 2007]. The U.S. EPA has a partnership with federal and state governments, industry, and trade associations to encourage use of CCRs because it is a high-volume waste stream (i.e., 126 million tons in 2006) and can replace raw materials with CCRs that are reported to out perform existing materials. The U.S. EPA is hoping to increase the use of CCRs to 50% by 2011 from 32% in 2001. (U.S. EPA, 2009)

Changes are occurring at U.S. coal-fired power plants that will reduce emissions of mercury, nitrogen oxides, particulate matter, and sulphur dioxide. Figure 2 provides a diagram of a coal-fired power plant showing the types of technologies that are being implemented to reduce emissions of mercury, nitrogen oxides, particulate matter, and sulfur dioxide. Coal-burning power plants are the largest anthropogenic source of mercury emissions to the air in the U.S., accounting for over 40 percent of all domestic anthropogenic mercury emissions. EPA has estimated that about one quarter of U.S. emissions from coal-burning power plants are deposited within the contiguous U.S. and the remainder enters the global cycle.

The major mercury-containing input to a power plant is coal with an average mercury content of 01. ug/g (Kilgroe et al., 2002). As flue gas cools, some elemental mercury can be adsorbed on fly ash (or suspended particles) which consists of inorganic ash and unburned carbon. The fate of mercury is tied to its form [i.e., gaseous elemental (Hg o), gaseous oxidized (Hg $^{2+}$), or particulatebound(Hg $_{p}$)].

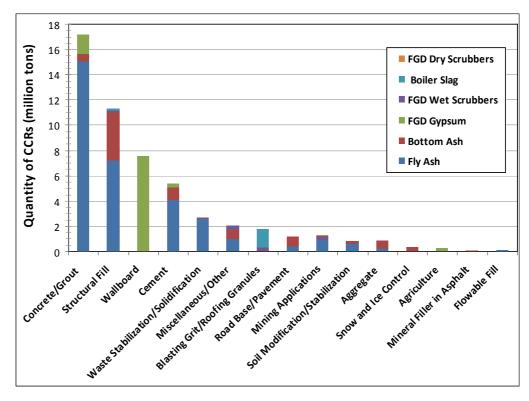


Figure 1. Uses of CCRs based on 2006 Industry Statistics (ACAA, 2007).

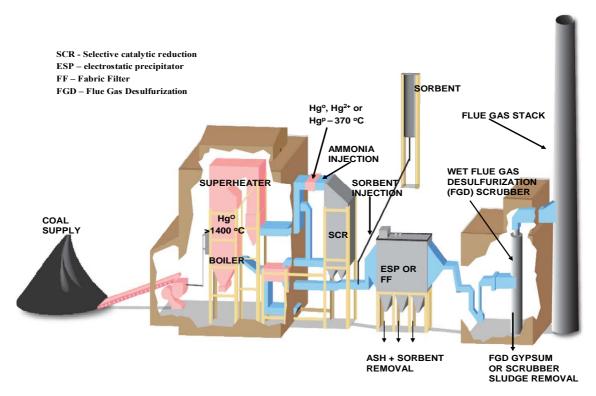


Figure 2. Diagram of Coal-Fired Power Plant Showing Technologies to Control Emissions of Mercury, Nitrogen Oxides, Particulate Matter, and Sulfur Dioxide

For example, catalysts are used to oxidize elemental mercury so that it is easier to control. The type and configuration of technologies in use at a power plants can vary such as the type of particulate control (i.e., fabric filter or cold-side or hot-side electric precipitator) and scrubber (wet or dry) as well as the variety of sorbents and catalyts in use.

With the addition of mercury specific controls, less mercury is released from the flue gas and transferred to either the fly ash or scrubber solids and liquid effluent. If dedicated mercury control technologies are applied, the activated carbon is collected with the fly ash. Some facilities are adding a fabric filter to the existing PM control device, so that the activated carbon that is injected into the flue gas can be collected separately. This preserves the use of fly ash by keeping it separate.

The type of coal can also varies across coal plants and affects the characteristics of the fly ash and scrubber residues. Even within a plant, different blends of coal are used at different units and can vary over time. Coal is classified according to its heating value and relative content of elemental carbon. Other elements such as nitrogen and sulfur will vary by coal type. Table 1 provides the mercury content from sampling of different coal types (Kilgroe et al., 2002).

The potential for the leaching of metals is a principal concern in assessing protection of groundwater and surface water. A major challenge in this research was identifying the appropriate leaching test method to use that would span the range of materials and potential management conditions.

Fuel Type	Number of Samples	Hg content, μg/g dry basis				
		Mean	Range			
Bituminous	27,793	0.11	0.0 - 1.3			
Subbituminous	8,180	0.07	0.008 - 0.9			
Lignite	1,047	0.11	0.02 - 0.75			

Table 1. Mercury content of U.S. coal-fired fired power plants in 1999 (Kilgroe et al., 2002).

Historically, single-point pH leaching tests (e.g., Toxicity Characteristic Leaching Procedure) have been used to support CCR management decisions. The U.S. EPA's Science Advisory Board and the National Academy of Sciences raised concerns over the use of single-point pH tests that do not reflect the actual conditions under which CCRs are typically managed (U.S.EPA, 2006; National Academy of Sciences, 2006). Because metal leaching rates change with changing environmental conditions (especially pH), the concern is that the existing leaching tests being used for CCR management decisions may not be the most accurate predictor of potential environmental release of mercury or other metals.

With changes occurring in air pollution control at coal-fired power plants, are there changes occurring that will result in CCRs being unsuitable for beneficial use? What is the fate of mercury and other metals that are being captured and controlled in the flue-gas at coal-fired power plants? Is there a change in the environmental stability of mercury and other metals found in CCRs as a result of more stringent air pollution control? These questions have been a major focus for the research being conducted by U.S. EPA's National Risk Management Research Laboratory. The objective is to identify any potential cross-media transfers to ensure that mercury and other pollutants being reduced at the power plant are not later released to the environment.

2. DEVELOPMENT OF LEACHING TEST METHODS

A first step in this research was to conduct a review of available data to evaluate fate of mercury and other metals found in fly ash and other air pollution control residues and the range of management practices currently being implemented. (EPA, 2002) After the initial review, it became apparent that most of the available data predates changes in air pollution controls. Another major obstacle was the lack of a single, consistent leaching test to be able to compare across CCRs and management practices. We also found that many of the leach tests in use were not published or did not include quality assurance and control necessary for reviewing the data. The other major issue is that the available data was based on single-point pH tests which do not consider the range of possible conditions under which CCRs are actually managed.

In response to these concerns and those raised by the National Academy of Sciences and EPA's Science Advisory Board (SAB), the "Leach Testing Framework" was selected to evaluate the potential for leaching of mercury and other metals due to the fact it considers the waste form, buffering capacity, and field conditions through a more detailed analysis. The framework includes a suite of different test methods that account for: (i) pH and LS (liquid-to-solid ratio) dependent leaching, (ii) percolation-based release using column testing, and (iii) diffusion-limited release from monoliths and compacted granular materials that behave as monoliths after placement. EPA consulted the SAB on the proposed approach and its application to evaluation of CCR leaching potential. The SAB was supportive and encouraged the collection of information

that would allow comparison of laboratory and field leach data, as a validation exercise. This suggestion has been incorporated into this research through use of EPA and industry-supplied data

A background document is being drafted to verify the scientific theory supporting the methods and how to interpret the results in environmental decision making. The report will also contain data that allows comparison of field versus laboratory leach data for a range of materials. The anticipated release of the final EPA report is spring 2010.

A set of draft EPA methods have been prepared and are currently being validated to be a part of SW-846 which is EPA's official compendium of analytical and sampling methods that have been evaluated and approved for use evaluating solid waste (http://www.epa.gov/osw/hazard/testmethods/sw846/index.htm). The draft methods are:

- Draft Method 1313 Liquid-Solid Partitioning as a Function of Eluate pH using a Parallel Batch Extraction Test
- Draft Method 1314 Liquid-Solid Partitioning as a Function of Liquid-Solid Ratio Using an Up-flow Column Test
- Draft Method 1315 Mass Transfer in Monolithic or Compacted Granular Materials Using a Semi-dynamic Tank Leach Test
- Draft Method 1316 Liquid-Solid Partitioning as a Function of Liquid-Solid Ratio Using a Parallel Batch Test
- Draft Method 1317 Concise Test for Determining Consistency in Leaching Behavior

In the next sections a description is provided of the equilibrium-based methods (1313 and 1314) being used to evaluate the leaching potential of metals found in CCRs. The U.S. EPA plans to provide a guidance document along with the release of the methods. The guidance will identify which methods to use for different applications and how to interpret the results for use in environmental decisions for beneficial use and other applications.

3. ALKALINITY, SOLUBILITY AND RELEASE AS A FUNCTION OF PH

The alalkinity, solubility and release of constituents of potential concern as a function of pH is determined using a precusor of the Draft Method 1313. The protocol consists of a series of parallel batch extractions of particle-size reduced material in dilute acid or base solutions. Particle-size reduction facilitates the approach to solid-liquid equilibrium during the test duration, however, particle size reduction of small grain materials (e.g., fly ash) is minimal. Each batch extraction consists of (i) placement of a mass of solid material equivalent to a specified dry mass (actual value depends on sample heterogeneity and particle size) into an extraction bottle, (ii) addition of deionized water to a final liquid-solid ratio of 10 mL/g, and (iii) pipetting of diluted acid or base based on a pre-test titration procedure in order to yeild a range of final extractions from pH 2 to 13. The extraction vessels are tumbled in an end-over-end fashion for a specified contact time that depends on the particle size of the sample. Liquid and solid phases are separated via settling or centrifugation and an aliquot is removed for measurement of eluate pH and conductivity. The remainder of the eluate is filtered (0.45 µm filter) by pressure or vacuum filtration and saved for chemical analysis.

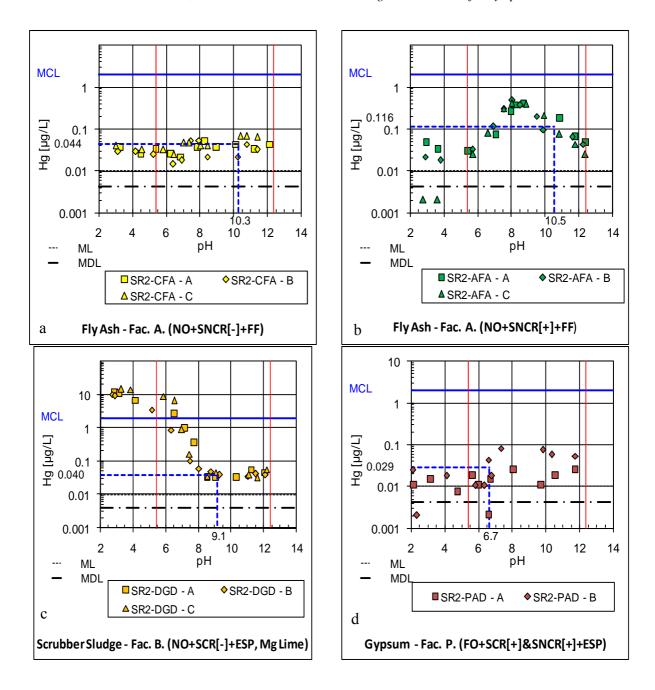


Figure 3. Examples of leaching behavior of mercury as a function of pH (U.S. EPA 2006 and 2008)

The titration curve of the material is produced by plotting the final eluate pH as a function of the amount of acid added (base additions shown as negative acid additions). The solubility and release of constituents of concern is represented as a liquid-solid partitioning (LSP) curve created by plotting eluate concentrations of constituents of interest as a function of eluate pH. Figure 3 shows an illustration of LSP curves for Hg in four coal combustion materials. The shape of the LSP curve is indicative of the speciation of the analyte in solution and as a function of pH. For example, the LSP curve of cationic [e.g., Cd(II)] and amphoteric species [e.g., Pb(II), Cr(III), As(III)] would show a minimum release in the moderately alkaline range whereas oxyanion species [e.g., Cr(VI), As(V)] show a maximum in the same range. The release (i.e., mass of species release per unit mass of solid materia) at the maximum concentration value can be used as an estimate of the available or mobile fraction of a species under environmental

conditions. For cationic and amphoteric species, maximum concentration is often observed as an asymptote at low pH (e.g.,pH < 4) making low pH extraction values very important to the interpretation of environmental leaching interpretation.

4. SOLUBILITY AND RELEASE AS A FUNCTION OF LS RATIO

Solubility and release as a function of LS ratio are determined using the Draft Method 1316 which was designed to provide the LSP of constituents in a granular solid material using five pparallel batch extractions of particle-size reduced at LS ratios of 10, 5, 2, 1, 0.5 mL/g dry material. Deionized water is used as the extractant such that the eluate pH reflects the natural pH of the material. The amount of solid material used in each extraction varies from 40 to 200g based on the LS ratio and the volume of eluate required for chemical analysis. The extraction vessels are tumbled in an end-over-end fashion for a specified contact time that depends on the particle size of the sample. Liquid and solid phases are separated via settling or centrifugation and an aliquot is removed for measurement of eluate pH and conductivity. The remainder of the eluate is filtered (0.45 μ m filter) by pressure or vacuum filtration and saved for chemical analysis.

Eluate concentrations may be plotted as a function of eluate pH in order to check consistency with pH-dependence test data. Two types of behavior are typical: (i) LS test data following the LSP curve indicates that solubility controls the release with LS ratio, (ii) eluate concentrations will cross the LSP curve indicating that the effect of LS ratio is non-linear. When eluate concentation data is plotted as a function of LS ratio, the effect of LS ratio on constituent release may be directly investigated. Highly soluble constituents which are not sensitive to pH (e.g., Na, K, and somtimes B or Tl) tend to increase dramatically when LS ratio decreases due to ionic strength effects at low LS ratio. The LS ratio dependence of constituents shown to be a strong function of pH will be controlled by the change in pH as LS ratios decrease. Therefore, eluate pH from the LS test should be plotted as a function of LS ratio.

5. APPLICATION OF LEACHING TEST METHODS ON COAL ASH

The U.S. EPA is currently considering how to regulate coal ash. Data from use of improved leaching test methods will be used to help evaluate risks for CCR disposal. Recent damage cases have highlighted potential environmental concerns from CCR disposal and reuse. For example, in December 2008, a major spill (4 million cubic meters) from a coal ash impoundment occurred covering 121 hectares at a TVA coal-fired power plant. In spring 2009, data was released from a golf-course in Virginia that was constructed using 1.5 million tons of fly ash. Although the site is less than two years old, monitoring date showed leaching of metals at levels of potential environmental concern. Historically, decisions have been tied to single-point pH tests that do not reflect likely range of field conditions. These damage cases highlight the need to provide better environmental assessment of the materials as well as management facility.

To date, approximately 80 CCRs have been collected from 30 U.S. coal fired power plants for characterization using the improved leaching test methods. The CCRs collected include 34 fly ashes and the rest are scrubber residues including flue-gas desulfurization gypsum, sludge (mostly calcium sulfite), filter cake, and "as managed" sludge which is typically a mixture of lime, fly ash and sludge. The CCRs collected are intended to span the range of coal types and air pollution control configurations.

The use of these results are intended as inputs to groundwater transport and fate models which take into account dilution and attenuation as well as other factors important in determining levels of potential concern to human health and the environment. To provide context of the leach

results being found, comparison is made to the threshold for hazardous waste determination [toxicity characteristic (TC)] and to health indicators for drinking water [e.g., maximum concentration limit (MCL) or the drinking water equivalent (DWEL)].

The results are being documented into a series of reports:

- Report 1 Characterization of Mercury-Enriched Coal Combustion Residues from Electric Utilities Using Enhanced Sorbents for Mercury Control (EPA, 2006) -Collected 12 fly ashes from 6 utilities with and without use of enhanced Hg control
- Report 2 Characterization of Coal Combustion Residues from Electric Utilities Using Wet Scrubbers (EPA, 2008) -Collected 23 CCRs from 8 utilities with wet scrubbers
- Report 3 Characterization of Coal Combustion Residues from Electric Utilities Using
 Multi-Pollutant Control Technology (to be available by Nov 2009) Collected 43 CCRs
 from 16 utilities with scrubbers (wet and dry)
- Report 4 Probabilistic Assessment of Potential Leaching of Mercury and Other Metals Based on Plausible Management Practices - (to be available by spring 2010)

In the first report identified above, fly ashes were evaluated from samples collected from six facilities with and without the use of sorbents for enhanced mercury capture (U.S. EPA, 2006). Table 2 provides results for mercury, arsenic, and selenium. The results show that mercury is strongly retained by the fly ash and unlikely to be leached at levels of environmental concern based when compared to TC or MCL. The results for report 1 indicate there could be a potential concern for increased mobility of arsenic and selenium when compared to TC or MCL. Report 1 also includes an illustration of how results can be used to conduct probablistic assessment of plausible management practices using methodolgy in Sanchez et al., 2005.

A second report provides data for a wider range of metals from evaluation of twenty-three samples collected from eight facilities that use wet flue gas desulfurization (FGD) scrubbers (U.S. EPA, 2008). Results for fly ash and FGD gypsum indicate that although there may not be a concern for leaching of mercury based on comparison to MCL or TC values, other metals may be of concern (Tables 3 and 4). Results also suggest that there may be more of a concern in terms of potential leaching of metals for fly ash and scrubber sludge than for FGD gypsum. The blocks that are highlighted in Tables 3 and 4 indicate where there may be potential concern when comparing the leach results to health based levels (i.e., MCL or TC). For five out of eight facilities there were sampled, CCRs were obtained with and without use of air pollution controls for nitrogen oxides (NOx). The results suggest that post-combustion NOx control may be a factor in release of Cr and other metals. Additional sampling has been conducted to verify that Cr⁺⁶ is being formed so resulting Cr in CCRs is more toxic and leachable. These results will be documented in report 3.

A third report is being drafted from analysis of forty-three samples obtained from sixteen facilities with multi-pollutant controls in use at coal-fired power plants. This report is expected to be released within the next six months. A fourth report is also under development. Using the methodology identified in report 1 providing a probablistic assessment of plausible management practices, all 80 CCRs will be evaluated to identify specific materials or management practices that may be of concern in comparison to health based indicators such as MCL or DWEL. The fourth report is intended to meet the commitment made to document the fate of mercury and other metals found in CCRs resulting from coal-fired power plants with more stringent air pollution control. (U.S. EPA, 2005).

With the use of improved leaching test methods, a data management tool is needed. A protootype of this tool has been developed based on collaboration with the developers of LeachXS (http://www.leachxs.com/lxsdll.html). The proposed name for the tool is LeachXS-Lite. It will be available for use by state and local regulatory authority, industry, and others. The

tool will provide access to available data and filters data by different categories including fly ash class, air pollution control, and material type (i.e., fly ash, sludge, gypsum). The tool currently has the ability to compare new materials to leaching test data in Reports 1 through 3. This will enable identifying classes of leaching behavior by material, metals, and field conditions. Finally, the tool also provide the ability to add in new data from the use of improved leaching test methods (1313 through 1317). The results from this tool will provide more realistic leach data as input to future risk assessments and to help ensure protection of human health and the environment for future CCR management decisions.

Additional information on the fate of mercury and other metals at plants using more stringent air pollution control technologieswill be reported from EPA research program as part of the work outlined in the EPA Mercury Road Map (U.S. EPA, 2005).

Table 2. Results of leach testing analysis for coal fly ash from six facilities using ACI for enhanced Hg capture (U.S. EPA, 2006).

	Hg	As	Se
Total in CCR material (mg/kg)	0.1 -1	20 - 500	3 - 200
Leach results (µg/L)	Generally 0.1 or lower	<1 - 1000	5 – 10,000
MCL^{1} (µg/L)	2	10	50
$TC^2 (\mu g/L)$	200	5,000	1,000
Variability relative to pH ³	Low	Moderate to High	Moderate

¹MCL is the maximum concentration limit for drinking water.

Table 3. Leach Data for Fly Ash (U.S. EPA, 2008).

	Hg	As	Se	Sb	Ba	В	Cd	Cr	Co	Pb	Mo	Tl
Total in Material (mg/kg)	0.04- 0.6	70-90	2-30	3-15	600- 1,500	NA	0.7- 1.5	100- 200	20-50	40-90	10-20	3-13
Leach results (µg/L)	<0.01 - 0.4	7-300	7-400	<0.3 - 200	90- 4,000	200 - 300,0 00	<0.2 - 30	1- 4,000	<0.3 - 200	<0.2 -2	100- 40,00 0	<0.3 - 300
MCL ¹ (μg/L)	2	10	50	6	2,000	7,000 DWE L2	5	100	-	15	200 DWE L	2
TC ³ (μg/L)	200	5,000	1,000	1	100,0 00	1	1,000	5,000	1	5,000	1	1
Variabilit y relative to pH ⁴	Low to High	Low to Med	Low to Med	Med to High	Low	Med to High	High	Low to Med	High	Med	Low to Med	Med

¹MCL is the maximum concentration limit for drinking water.

²TC is the toxicity characteristic and is a threshold for hazardous waste determinations.

³ Variability defined as low is <1 order of magnitude difference; moderate is 1 to 2 orders of magnitude difference; and high is >2 orders of magnitude difference.

²DWEL is the drinking water equivalent level.

³TC is the toxicity characteristic and is a threshold for hazardous waste determinations.

⁴Variability defined as low is <1 order of magnitude difference; moderate is 1 to 2 orders of magnitude difference; and high is >2 orders of magnitude difference.

	Hg	As	Se	Sb	Ba	В	Cd	Cr	Co	Pb	Mo	Tl
Total in Material (mg/kg)	0.01- 0.5	2-4	2-30	2-6	3-60	NA	0.3- 0.5	6-20	1-4	1-12	2-12	0.6-2
Leach results (µg/L)	<0.01 -0.6	0.5- 10	4- 3,000	<0.3- 10	40- 400	40- 70,00 0	<0.2- 50	<0.3- 50	<0.2- 10	<0.2- 10	1-600	<0.3- 20
MCL ¹ (μg/L)	2	10	50	6	2,000	7,000 DWE L2	5	100	15	15	200 DWE L	2
TC^3 (µg/L)	200	5,000	1,000	ı	100,0 00	ı	1,000	5,000	ı	5,000	ı	ı
Variabilit y relative to pH ⁴	Low to Med	Low to Med	Low to Med	Low	Low	Low to Med	High	Med to High	Low	Low	Low	Low

Table 4. Leach Data for FGD Gypsum (U.S. EPA, 2008)

⁴Variability defined as low is <1 order of magnitude difference; moderate is 1 to 2 orders of magnitude difference; and high is >2 orders of magnitude difference.

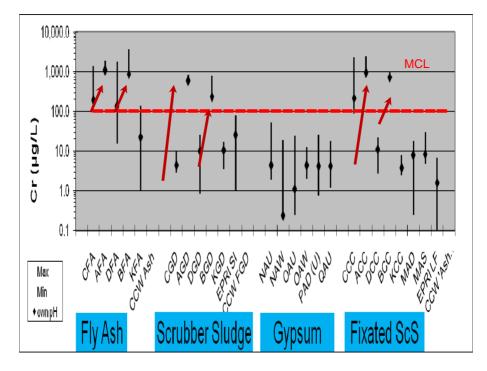


Figure 4. Results of chromium leaching concentration results (5.4<pH<12.4) for different CCRs evaluated in "Report 2" (EPA, 2008).

6. CONCLUSIONS

The addition of improved multi-pollutant control technology at coal fired power plants will result in reducing air emissions of concern. However, depending upon how the CCRs are managed, there could be a potential for cross-media transfer. To help prevent this from occurring, improved leaching test methods will enable more informed decisions to be made

¹MCL is the maximum concentration limit for drinking water.

²DWEL is the drinking water equivalent level.

³TC is the toxicity characteristic and is a threshold for hazardous waste determinations.

regarding CCR management. These leaching test methods are applicable to other materials being used in commercial and engineering applications. With the increased interest in protecting scarce resources and conserving energy, there is more wide-spread interest in use of coal ash and other secondary materials. However, it is important that improved leaching test methods be used to ensure protection of human health and the environment through more comprehensive environmental assessment.

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