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Project Summary

Evaluation of an ESCA/Leachate Analytical Scheme to Characterize Process Stream Wastes

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An ESCA/leachate analytical scheme was evaluated for its ability to characterize solid waste from combustion processes and hazardous waste incinerators. Samples were analyzed for surface elemental composition by electron spectroscopy for chemical analysis (ESCA) before and after aqueous leaching. Selected elements were subjected to oxidation state studies by ESCA, and leachates were analyzed for anions by ion chromatography and for trace metals by inductively coupled plasma spectrometry. The results of ESCA before and after leaching compared favorably with leachate data. Although aqueous leaching did not significantly affect the metal species present in the samples used in this study, it did extract considerable amounts of water-soluble ions: sodium, calcium, chloride, sulfate. Essentially all samples showed an increase in oxygen after leaching that was attributed to hydration by the aqueous extraction medium.

ESCA can successfully speciate chromium, lead, and zinc when these elements are sufficiently abundant in the sample. The technique is limited, however, by its ability to detect only those elements present at greater than 0.1 to 1.0 percent atomic. Most process wastes contain very low concentrations of metals, thereby minimizing the suitability of ESCA for process waste characterization. However, municipal and hazardous waste incinerators produce bottom and baghouse ashes containing significant amounts of hazardous trace metals. ESCA in conjunction with process data could prove useful in determining metal species present and their potential for release from particulate.

This Project Summary was developed by EPA's Industrial Environmental Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Solid waste from refuse incineration presents a landfill disposal problem owing to its potentially hazardous nature. Significant leaching from fly ash and bottom ash has been shown to occur when these materials are exposed to neutral or slightly acidic solutions simulating rainwater. In a landfill disposal situation, ground water quality would be affected by the amounts and types of compounds thus leached.

Incinerator residue leachates and acid digestions have been characterized for anion and cation content, but the actual solid waste has not been studied before and after leaching to determine changes in its composition. Electron spectroscopy for chemical analysis (ESCA or XPS) is a surface analysis technique and was expected to be useful for obtaining such information. With ESCA, only the top 20 to 100 Å of the solid or particulate surface are analyzed, and in leaching studies, it is this part of the sample that is of interest. ESCA provides elemental composition data for all elements except hydrogen and helium. In addition, for a number of elements, ESCA can be used to determine

oxidation state and bonding information.

This project was designed to evaluate a combined ESCA/leachate analytical scheme for characterizing the leachability of wastes from process streams. ESCA was used to analyze waste samples (fly ash, bottom ash) before and after leaching, and to provide an estimate of oxidation state (speciation) for several elements. Chemical analyses of the leachates were used to provide supporting data for the ESCA/leachate results.

Experimental Program

The experimental program for evaluating the utility of an ESCA/leachate analytical scheme consisted of replicate analyses of actual source particulate samples using ESCA, Level 1 aqueous leachate generation, ion chromatography, and inductively coupled plasma emission spectroscopy.

Conclusions

Several conclusions were drawn from the data obtained in this study:

- Elemental identifications by ESCA are reproducible for major components (>5 percent atomic), but minor elements are often misidentified as noise or vice versa.
- Quantification by ESCA is fairly precise (15 percent RSD) for most elements present as major components or for which ESCA has high sensitivity. This precision holds only for homogeneous samples or samples for which representative aliquots can be mounted on ESCA substrates. Elements producing low photoelectron counts show much greater variation in quantification which is attributable to subjective decisions by the analyst.
- The material used as substrates to mount samples in the ESCA appears to introduce no consistent bias in the concentrations of elements measured in the samples.
- The Level 1 aqueous leaching procedure has a rather low extraction efficiency for many species found in process waste samples. It also suffers from poor precision when inhomogeneous samples or nonrepresentative aliquots are analyzed.
- ESCA can be used to speciate chromium, lead, and zinc when these elements are present in fairly high concentrations. At the detection limit, they do not produce sharp enough peaks for the analyst to determine a precise binding energy for the element. As learned from previous ESCA work, it is also possible to speciate sulfur and carbon in samples of this type.
- The ESCA/leachate analytical scheme can be used to detect changes in sample composition caused by aqueous leaching. As expected, water soluble components are easily extracted from the sample while metals show an apparent increase in concentration after leaching. This is primarily due to the removal of the soluble surface components. The species of metals present in the samples did not seem to change after leaching.

Recommendations

The following recommendations are based on the results of this experimental program:

- The combined ESCA/leachate analytical scheme should probably not be pursued as a means for determining the degree of hazard associated with disposal of process wastes. The combined procedure requires a great deal of analytical time, and the benefits of post-leaching ESCA analyses appear minimal based on the samples used in this study. Although other samples may behave differently, it must be recognized that these samples (containing metals at concentrations detectable by ESCA) were extremely difficult to obtain and will probably not be routinely encountered in practice.
- The use of ESCA alone to predict aqueous leachability has advantages. Its focussing on the surface of particulate matter and its ability to detect elements and species of high aqueous solubility recommend it for this purpose. This statement, supported by the literature, is based on the supposition that, under the conditions of the Level 1 leaching procedure, only small amounts of trace metals will be leachable from the waste.
- The use of ESCA to determine trace metal oxidation states could be useful in a study to correlate the metal compounds formed during incineration with combustion conditions and the type of waste. Similar work has been performed using metal compound volatility to predict if the metal will accumulate in flyash, bottom ash, or scrubber slurry, or be emitted from the stack. Adding the speciation capability of ESCA to this evaluation could prove quite interesting.

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The complete report, entitled "Evaluation of an ESCA/Leachate Analytical Scheme to Characterize Process Stream Wastes," (Order No. PB 85-116 192; Cost: $14.50, subject to change) will be available only from:
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