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USDOJ Office of Surface Mining Reclamation and Enforcement

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IMPROVED STATIC TEST PREDICTION OF ACID GENERATION POTENTIAL: A SURFACE ANALYSIS APPROACH

Hylton G. McWhinney¹, P. T. Behum³, J. E. Brandt², and Tony Grady¹

Prairie View A&M University, Prairie View, Texas¹; Railroad Commission of Texas, Austin, Texas²;
Office of Surface Mining, Mid-Continent Region, Alton, Illinois³

Project Description and Objectives:

X-Ray photoelectron spectroscopy (XPS) a surface analytical tool is applied in the characterization of minerals common to mining operations. Samples collected from mine sites were also analyzed with XPS indicate that the technique holds strong potential as a complimentary method in explaining the chemistry of oxidic, sulfur-bearing species such as jarosite, barite, anhydrite, as well as reduced forms of sulfur (pyrite and galena). Semi-quantitative results from XPS compare favorably with bulk results determined by traditional acid-base accounting (ABA) methods.

Applicability to Mining and Reclamation:

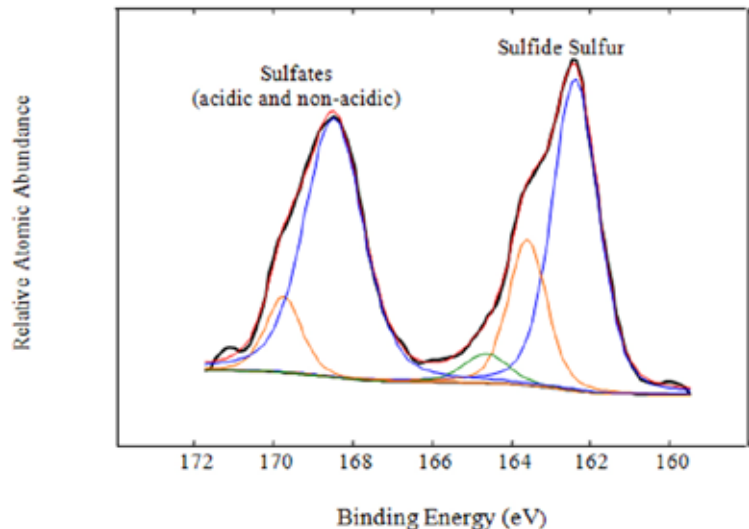
Acid mine drainage or acid rock drainage (AMD/ARD) is one of the costliest environmental problems. Currently, reliance is placed on acid-base predictive tests and modeling for the prevention and mitigation of AMD. Acid-base accounting (ABA) methods have shortcomings which can lead to over or underestimation of future acid generation. Sources of errors associated with ABA are

- Over compensation for reactive sulfur content by including sulfate from gypsum and other non-acid forming sulfates in the total sulfur determination.
- In the determination of neutralization potential (NP), strong acids may cause the dissolution of stable minerals giving rise to erroneous results.
- The overestimation of NP when siderite (FeCO_3) is present.
- The underestimation of NP when dolomite is present.

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Sample ID	LECO Analyzer Bulk Sulfur (%)			XPS Surface Sulfur (%)
	Pyritic	Total S	Pyritic/Total S	Relative %/Total S
BW6(1A)#2	0.03	0.34	9.0%	8.0%
BW2A1208C	0.11	0.45	24.0%	20.0%
Store-1	0.02-0.06	0.49	8.0%	6.0%

ABOVE TABLE: Comparison of Bulk Sulfur Ratios with XPS Surface Species of Selected Mine Samples.



ABOVE FIGURE: XPS spectrum of oxidized surface of pyrite showing the reduced and oxidized regions.

Applicability to Mining and Reclamation (continued):

- The formation of metal hydroxides in titrations involving sodium hydroxide produces additional errors in calculating NP.
- Jarosite, a common secondary mineral at mine sites, is acid-forming under certain environmental conditions, but can be under-quantified in potential acidity (PA).
- Galena (PbS) may be included in the PA, but as a mineral with a low solubility may not contribute to acidity at a mine site.

Mineral speciation is proving to be helpful in understanding the complex chemistry of soils and rocks associated with mining. Great importance is being placed on the application of cutting edge characterization techniques that can assist in reducing errors in ABA.

Methodology:

XPS is an analytical technique capable of elemental and chemical speciation at depths of approximately 5-150 Angstroms in the surface of a sample. The quantitative information gained is given as relative % of chemical specie versus the mass weight. In very fine powders, quantitative results are observed to be comparable to bulk concentrations. We investigated the application of XPS:

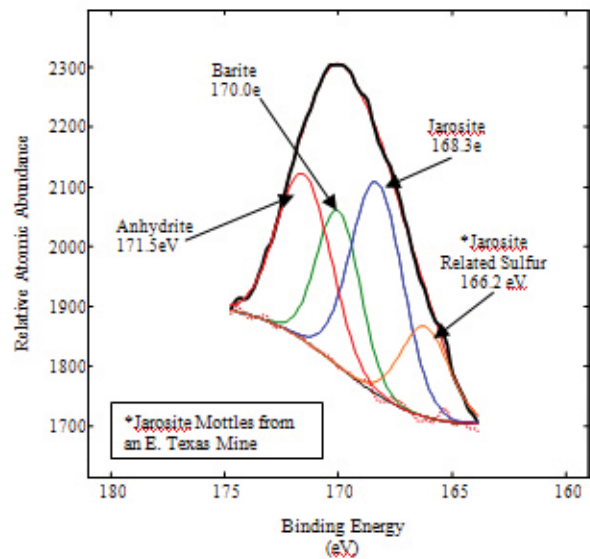
- For routine elemental and mineral composition characterization of mine site materials.
- To identify and quantify reactive sulfide (i.e., pyritic) species relative to all other types of sulfur in mine samples.
- Distinguish among sulfur mineral compounds (barite, anhydrite, and jarosite).
- To understand its limitation in mine sample characterization.

Surface analysis was carried out with a PHI 5400 chemical imaging XPS. Bulk sample analyses for total sulfur, pyritic, sulfates and organic sulfur were carried out at Energy Labs Inc. in College Station, Texas. A LECO Sulfur Analyzer was employed to calculate bulk % sample concentration based on standard operating

procedures, (EPA -670/2-74-070: Mine Spoil Potential (EPA-600/2-78-054: Field and Lab Methods).

Results/Findings:

XPS spectra of the sulfur 2p regions for minerals and field samples studied are shown in Figures 1 and 2. Figure 1 is an XPS binding energy S 2p spectrum of air oxidized planar polished euhedral pyrite and establishes the basis on which we identified the general sulfur species in terms of sulfates and pyritic sulfur. Figure 2 shows the S 2p region of a mixture barite mineral, anhydrite mineral and a field-collected jarosite sample. The de-convoluted spectrum shows distinct signature for the minerals corresponding to their predetermined binding energies. A comparison of the bulk pyrite/total sulfur ratio versus the XPS determined pyrite/total sulfur ratio for three analyses is presented in Table 1. There appears to be good correlation between semi-quantitative XPS results and bulk traditional results. Irrespective of the sample type, the XPS spectra did not show improved resolution at the higher bulk concentrations over the lower concentrations. It appears that the lower limit for XPS analysis of these types of samples is ~ 0.3% total sulfur.



ABOVE FIGURE: XPS of mixture of barite, anhydrite and jarosite minerals (S 2p chemical shifts).

Website Information:

The final project report can be found at <http://www.techtransfer.osmre.gov/NTTMainSite/appliedscience/2006appscience/CompletedProjects/PVAMVAcidGenPotential06FR.pdf>

Principal Investigator:

Hylton G. McWhinney
Prairie View A&M University
936.261.3112
hgmwhinney@pvamu.edu

OSM Project Technical Representatives:

Len Meier
618.463.6463 x5109
lmeier@osmre.gov

Paul Behum
618.463.6463 x5122
pbehum@osmre.gov



For Further Information About OSM's Applied Science Programs:

Kimery Vories - kvories@osmre.gov - (618) 463-6463, Ext. 5103