SENSITIVITY ANALYSIS OF ACID MINE DRAINAGE PREDICTION LEACHING TESTS[1]

by

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Abstract. A statistical sensitivity analysis was conducted to identify predominant factors controlling leachate quality from humidity cell, soxhlet, and column overburden leaching tests. Soxhlet and weathering cell tests exhibited the greatest sensitivity to (1) *storage condition* (temperature and humidity); and (2) *leaching interval* (time between leachings). The factors of *particle size* and *leachant temperature* played secondary roles in influencing leachate quality. High temperature, oven sample storage selectively promoted pyrite oxidation over carbonate dissolution, with pyrite oxidation reactions that were continuous and thermally enhanced, and discontinuous calcium carbonate dissolution which varied with the water content of the overburden sample. For short leaching intervals, (2 days), pore water was retained for a greater percentage of the total storage interval, and acidity produced by pyrite oxidation was neutralized by carbonate dissolution. For longer leaching intervals (7 days), pore water was retained for a smaller portion of the total storage interval, and acidity was not neutralized by carbonate dissolution. Weathering cells and columns exhibited sensitivity to *particle size* which controlled the rate of water movement through the overburden material. Short residence times for pore water in the large grained overburden samples favored low alkalinity production. Fine grained overburdens, with small pore sizes, resulted in slower water movement, favoring greater alkalinity production and acid neutralization. Acid and sulfate production rates exhibited logarithmic correlation with particle size. Acid production rates for high NP overburdens were positively correlated with the logarithm of particle size, while acid production rates for low NP overburdens were negatively correlated with particle size. Sulfate production rates were negatively correlated with particle size. The results of this study aid in the selection and design of overburden leach tests for acid mine drainage prediction.

Key Words: acid mine drainage, leaching tests, weathering cell, soxhlet, leaching column

Introduction

Overburden leaching tests, such as humidity cells, columns, and soxhlets are commonly used overburden analytical techniques used to evaluate and screen mine overburden for potential acid mine drainage problems. Leaching tests are thought to provide the kinetic data that static testing techniques such as acid-base accounting (ABA, Sobek, *et al*, 1978) fail to provide. Dynamic tests result in empirical data collected by subjecting mine overburden samples to simulated weathering conditions which, in theory, mimic natural weathering conditions. The rates of pyrite oxidation and the release of weathering products are then measured quantitatively to determine whether a sample will produce acidic leachate, and if so, estimate the magnitude of potential acid loads.

The use of these techniques is not without disadvantages, however. The procedures are labor intensive, typically require twelve or more weeks to complete, and are thus relatively expensive to perform. In addition, the relative rate of laboratory weathering compared to conditions encountered at actual minesites, and the amount of time in the field that is represented in laboratory tests is uncertain. Also strongly contended is the argument that whether results gathered from a laboratory test that may involve as little as 100 grams of finely crushed mine overburden in a soxhlet or weathering cell leach test, can accurately be extrapolated to characterize overburden weathering at an actual mine.

Another important obstacle to the use of leaching tests is that the tests often appear to provide contradictory results. Ostensibly identical overburden samples have often been observed to produce acidic results in one type of test, and alkaline results in another (Bradham and Caruccio, 1991). These contradictions between the various types of leaching tests appear to be related to the differences in the conditions created for overburden weathering among the tests. Humidity cells and columns, for example, weather overburden samples at relatively low temperatures (~20°C) and at high humidity; compared to soxhlet extraction tests which are conducted at high temperatures (~105°C) in a drying oven (low humidity). Overburden sample particle

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size can vary dramatically among the various leaching tests, with larger particles used in column tests, and finely crushed particle sizes used in soxhlet extraction tests.

To address some of these concerns this study was designed to enable direct comparison of the manner in which conditions created for overburden weathering affected the production of leachate quality. The leaching tests and interpretation techniques were designed to test the sensitivity of each test to variations of the weathering conditions created by commonly used leaching tests such as:

1) ***storage***, the temperature and humidity at which an overburden sample was weathered,

> Humidity cells and columns weather overburdens under ambient temperature (~20°C), high humidity conditions (Caruccio, 1968), while the soxhlet extraction process weathers overburdens in a drying oven under conditions of high temperature (~105°C) and low humidity (Renton, *et al*, 1988);

2) ***temperature,*** the temperature of the leachant water,

> Humidity cells and columns are leached with cold (~20°C) water, while the soxhlet extraction process utilizes cyclic leachings with hot (~80°C) water;

3) ***particle size***, the particle size to which an overburden is crushed,

> Humidity cells typically utilize overburden samples crushed to pass 4 mm, columns use larger particles up to 2 cm in size (Hood and Oertel, 1984), while soxhlet extraction samples are typically crushed smaller than 250 μm;

4) ***leaching interval***, time interval between leaching events,

> Overburden samples are typically leached on a repeating seven day schedule, but both shorter and longer leaching intervals have been used.

Materials and Methods

Ten coal mine overburden samples, predominately siltstones and shales (samples A, B, C, D, F, G, H, & J), with two coal refuses (samples E & I), were collected from surface mines in Pennsylvania, Tennessee, and West Virginia. Five of the samples, A-E, were crushed and sieved into two particle size fractions. The fraction ranging in size from 2.26 mm to 4 mm was designated *large*, while the fraction ranging in size from 125 μm to 250 μm was designated *small*.

Representative sample subsplits were packed into cellulose soxhlet extraction thimbles, and stored under the following conditions of ***storage***:

1) ***ambient*** (ambient temperature (~20°C) and humidity of a climate controlled laboratory),
2) ***humid*** (high humidity, ambient temperature; created by a specially constructed humidity chamber), and
3) ***oven*** (high temperature (~105°C), zero humidity; in a drying oven).

Table 1: Overburden Samples A-E Acid Base Accounting Results

Sample ID	**% Pyrite**	**NP**
A Large	1.7	11.8
A Small	2.4	9.6
B Large	0.2	176.9
B Small	0.2	110.1
C Large	0.6	25
C Small	0.6	25.9
D Large	0.8	28.4
D Small	1	25.9
E Large	3	53.4
E Small	3.5	53.7

Both overburden sample size fraction subsplits were then leached twelve (12) times at intervals of 7 days; under two conditions of ***temperature***:

1) ***cold*** (20°C) deionized water, and
2) ***hot*** (80°C) deionized water.

Sample subsplits of both size fractions were also leached; again half with *cold* deionized water, and half with *hot* water deionized water, in varying ***leaching intervals*** of:

1) ***2*** days,
2) ***4*** days, and
3) ***7*** days

A second phase of leaching tests examined the sensitivity of acid production in weathering cells and leaching columns to variations in ***size*** and ***sorting efficiency*** of overburden particles. In this part of the study, mine overburden samples F-J were crushed, and sieved into three ***particle size*** fractions;

1) a ***large*** size fraction, consisting of particles ranging in size from 12.7 mm to 50 mm,
2) a ***medium*** size fraction of particles ranging from 6.4 to 12.7 mm, and
3) a ***small*** size fraction, consisting of particles smaller than 6.4 mm.

Differing **sorting coefficient** subsplits were created by blending the three sieved size fractions into three combinations:

1) three ***well sorted*** subsplits, an equal mixture of the large and small size fractions,
2) two ***medium sorted*** subsplits, and equal mixture of the medium and small size fractions, and
3) one ***poorly sorted*** subsplit, an equal mixture of the large, medium, and small size fractions.

Table 2: Sensitivity Analysis Leaching Tests

	Leachant Temperature			
	Cold (20 C)		**Hot (80 C)**	
	Particle Size		**Particle Size**	
Condition of Storage	**Large**	**Small**	**Large**	**Small**
Ambient	A-E	A-E	A-E	A-E
Humidity	A-E	A-E	A-E	A-E
Oven	A-E	A-E	A-E	A-E
Weathering Cell	A-E	A-E		
Oven (2 day)	A-E	A-E	A-E	A-E
Oven (4 day)	A-E	A-E	A-E	A-E
Oven (7 day)	A-E	A-E	A-E	A-E
Weathering Cell (2 day)	A-E	A-E		
Weathering Cell (4 day)	A-E	A-E		
Weathering Cell (7 day)	A-E	A-E		

Table 3: Overburden Samples F-J Acid Base Accounting Results

	% Pyrite			**Neutralization Potential**		
	Sorting Efficiency			**Sorting Efficiency**		
Sample ID	**Well**	**Medium**	**Poor**	**Well**	**Medium**	**Poor**
F Large	1.1	1	1.1	31	30.4	31.3
F Medium	1			30		
F Small	1.2	1.1		33	31.6	
G Large	0.4	0.4	0.4	19	22	24.1
G Medium	0.4			25		
G Small	0.5	0.4		28	26.6	
H Large	0.2	0.2	0.2	14	16.2	17
H Medium	0.2			19		
H Small	0.3	0.2		19	18.6	
I Large	0.4	0.9	1.2	7.9	8	9.9
I Medium	1.5			8.1		
I Small	1.8	1.6		14	11	
J Large	0.2	0.2	0.2	39	42.5	46.3
J Medium	0.2			46		
J Small	0.1	0.2		54	50.1	

Table 4: Sensitivity Analysis Leaching Tests

	Well Sorted		
	Large	**Medium**	**Small**
Column	F-J	F-J	F-J
Weathering Cell	F-J	F-J	F-J
	Medium Sorted		
	Large + Medium	**Medium + Small**	
Column	F-J	F-J	
Weathering Cell	F-J	F-J	
	Poorly Sorted		
	Large+Medium+Small		
Columns	F-J		
Weathering Cell	F-J		

Representative subsplits were packed into weathering cells and leaching columns, and leached with deionized water at seven day intervals for a minimum of twelve weeks. Leachate quality data from all of the leaching tests were quantified as:

1) mean contaminant loads (titratable acidity, sulfate),
2) initial contaminant loads (acid or sulfate load from initial leach),
3) contaminant production rates (mathematical acid or sulfate production rate for a given time period), and
4) total contaminant loads (sum of acid or sulfate loads for all twelve (or ten) leachings).

The quantified data were then evaluated utilizing analysis of variance (ANOVA), Bonferonni means comparisons tests, linear regression, and graphical interpretation to determine the effect of each factor (storage condition, particle size, leachant temperature, or leaching interval) on contaminant production.

Results

The factors expressing the greatest degree of influence over variations in contaminant production were ***storage*** and ***leaching interval***. Compared to *oven* storage conditions, the *ambient, humid,* and *weathering cell* storage conditions created statistically similar ranges of contaminant production. Differences in storage conditions, between the *oven* (high temperature/no humidity) and *weathering cell* (low temperature/high humidity) resulted in 18 to 85% of the variability in acid production rates. Variations in *leaching interval* of 2,4, and 7 days resulted in up to 100% variability in contaminant production. Compared to the effects of *storage* and *leaching interval,* the factors of *particle size* and *leachant temperature* (cold or hot) played secondary, less significant roles in influencing leachate quality variation.

The factors of *storage* and *leaching interval* also exhibited interactive effects on leachate quality variability. In several instances these interactions produced acidic leachate for an overburden sample weathered under one combination of *storage* and *leaching interval* conditions, and alkaline results when weathered under another combination. High temperature, oven storage (100°C/low or no humidity), characteristic of soxhlet extraction, appeared to selectively promote pyrite oxidation over carbonate dissolution. As indicated by figures 1 and 2, pyrite oxidation (acid production) reactions, as indicated by sulfate production rates (slope of cumulative sulfate curves), were continuous, appearing to occur even in the absence of water, and were thermally accelerated (54% to 68% higher than that of low temperature (20°C) storage). Calcium carbonate dissolution (alkalinity production), on the other hand, appeared to be a discontinuous process, occurring only in the presence of water, during the period of leaching, and during the first seven hours of oven storage; after which the sample was dry. When ***leaching intervals*** were increased from 2, to 4, and to 7 days, the length of time that the overburden samples retained porewater, however, occupied an increasingly smaller portion of the total storage period. Acid production **rates** decreased as intervals of oven storage became longer (7 days)(figure 1), indicating that the rate of pyrite oxidation decreased as porewater evaporated. Total acid **loads**, however, were higher for longer (7 days) intervals of oven storage (figure 2), due to a greater length of time for acid production reactions to occur.

Dwarfed by the presence of dominating factors of storage and leaching interval, ***particle size*** played a subordinate, although important role in controlling leachate quality. Pyrite oxidation rates were slightly higher in the *small* (125-250 µm) size fraction than in the *large* (2.26-4 mm) size fraction. This effect tended to supplement the main effects of storage condition and leaching interval. Longer intervals of oven storage tended to promote acid over alkalinity production, and the ***small*** particle size subsplits of those overburden samples tended to be even more acidic than were the *large*. Because weathering cell overburden storage tended to promote alkaline leachate quality by allowing carbonates to dissolve and neutralize acidity, the ***small*** size fraction subsplits of those samples were more alkaline than were the *large*.

When temperature or leaching interval effects were absent, however, as was the case in the second phase column and weathering cell tests, leachate quality variability for the leaching tests was sensitive to the factor of particle size. Both acid and sulfate production rates were linearly correlated with the logarithm of particle size. Acid production rates for overburden samples with high NP's (~>20‰), such as the "G" sample depicted in figure 5, exhibited a positive correlation with the logarithm of particle size, indicating that, for these samples, acidity decreased with decreasing particle size. Acid production rates for overburden samples of high pyrite/low NP (~10‰), such as the "I" sample in figure 6, however, increased with decreasing particle size, and were thus negatively correlated with particle size. Sulfate production rates, however, for all of the overburden samples, were negatively correlated with particle size.

Leachant temperature (*hot* (100°C), or *cold* (20°C)) only minimally influenced contaminant production when compared to the other factors. Acidity and sulfate production tended to be slightly enhanced by hot water leaching; although not consistently. In the case of two of the overburden samples, the cold leached subsplits produced acidic leachate, while the hot leached subsplits produced

Figure 1: Acid Production Rates for Overburden Samples A-E

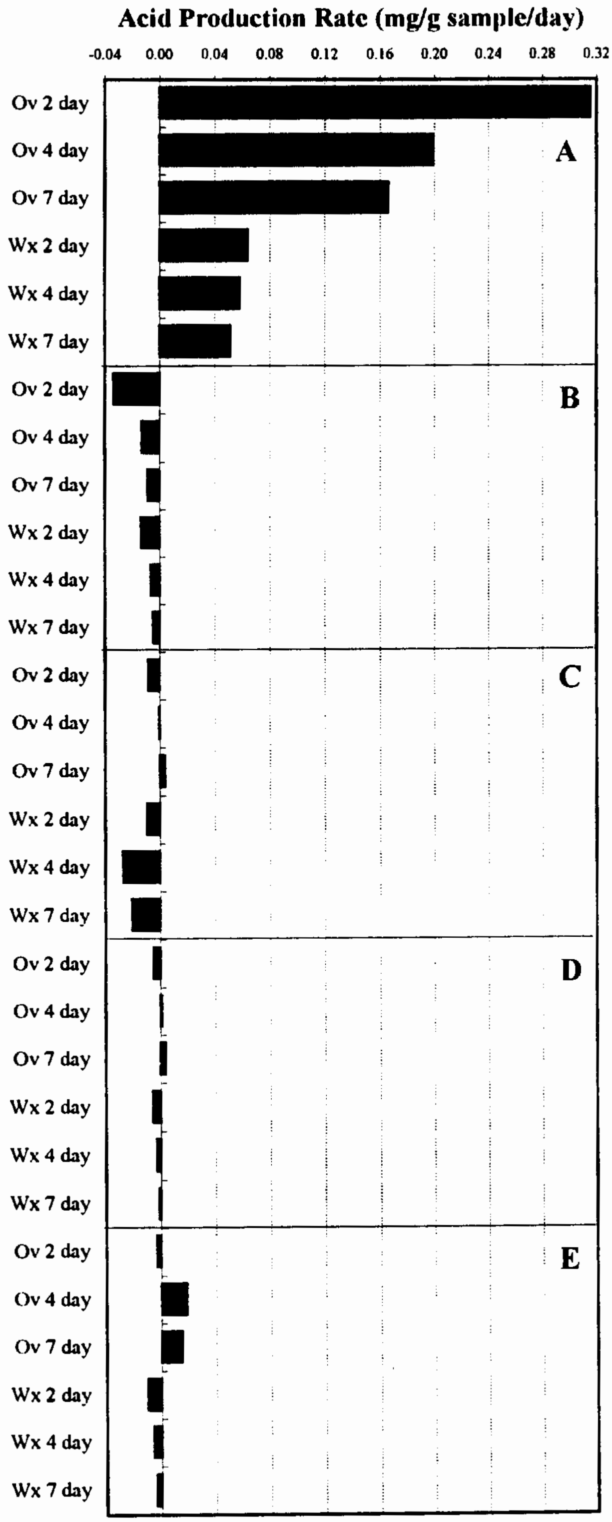


Figure 2: Total Acid Loads for Overburden Samples A-E

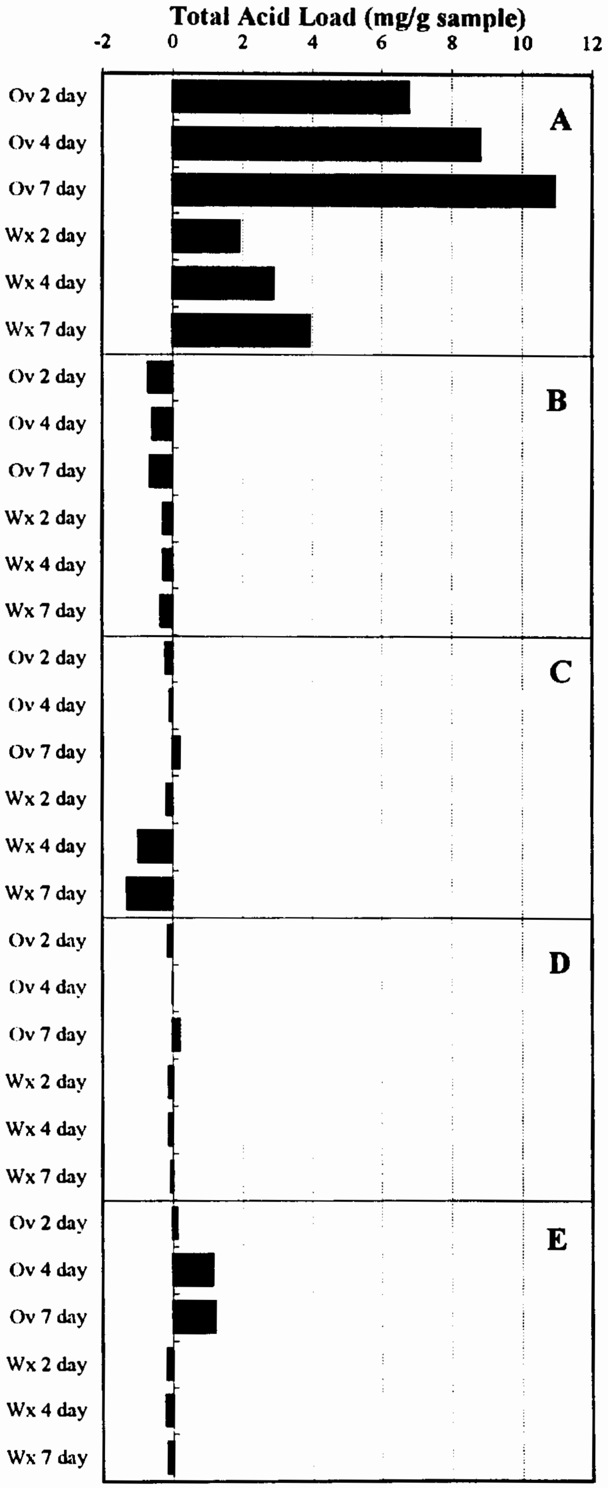


Figure 3: Cumulative Sulfate for Oven Stored Overburden Sample C

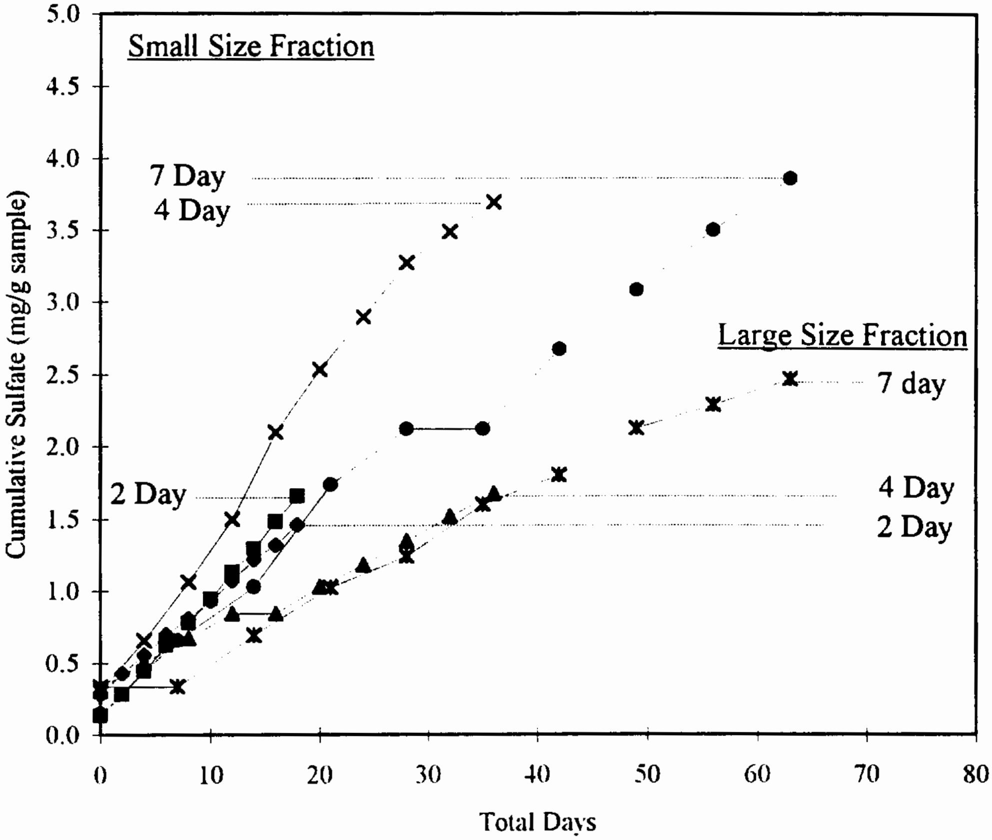


Figure 4: Cumulative Sulfate for Oven Stored Overburden Sample D

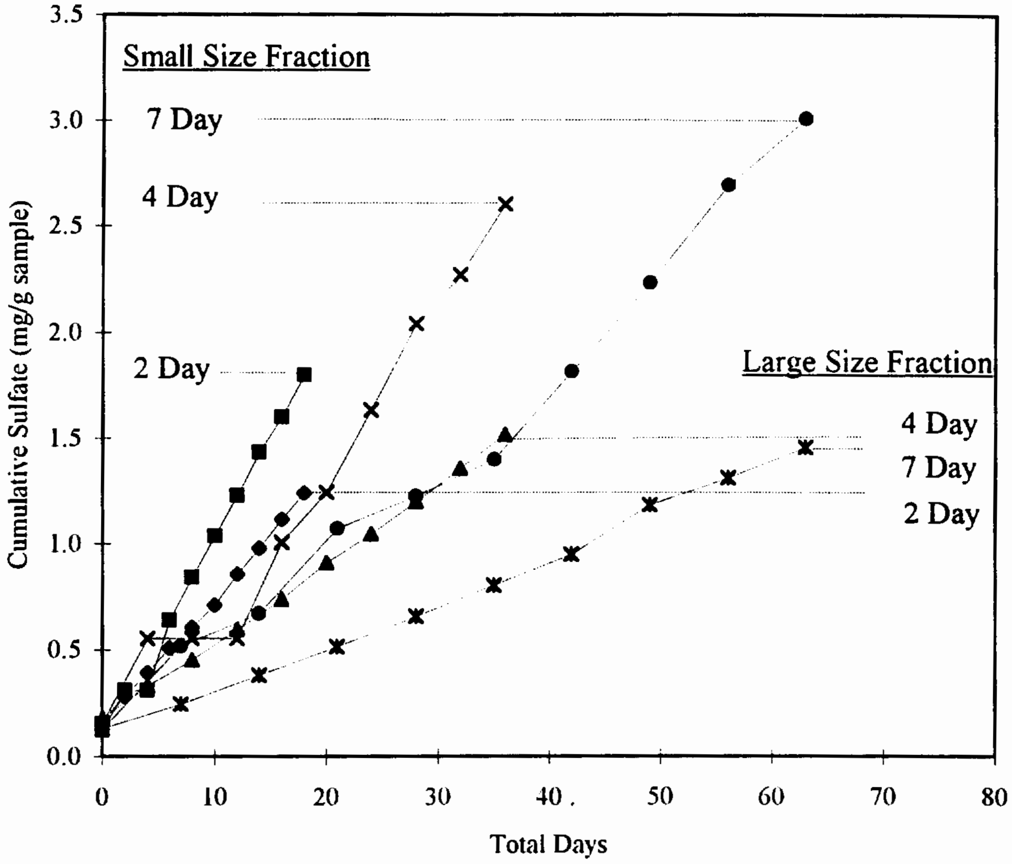


Figure 5: Acid Production Rate vs. Particle size
for Overburden Sample G

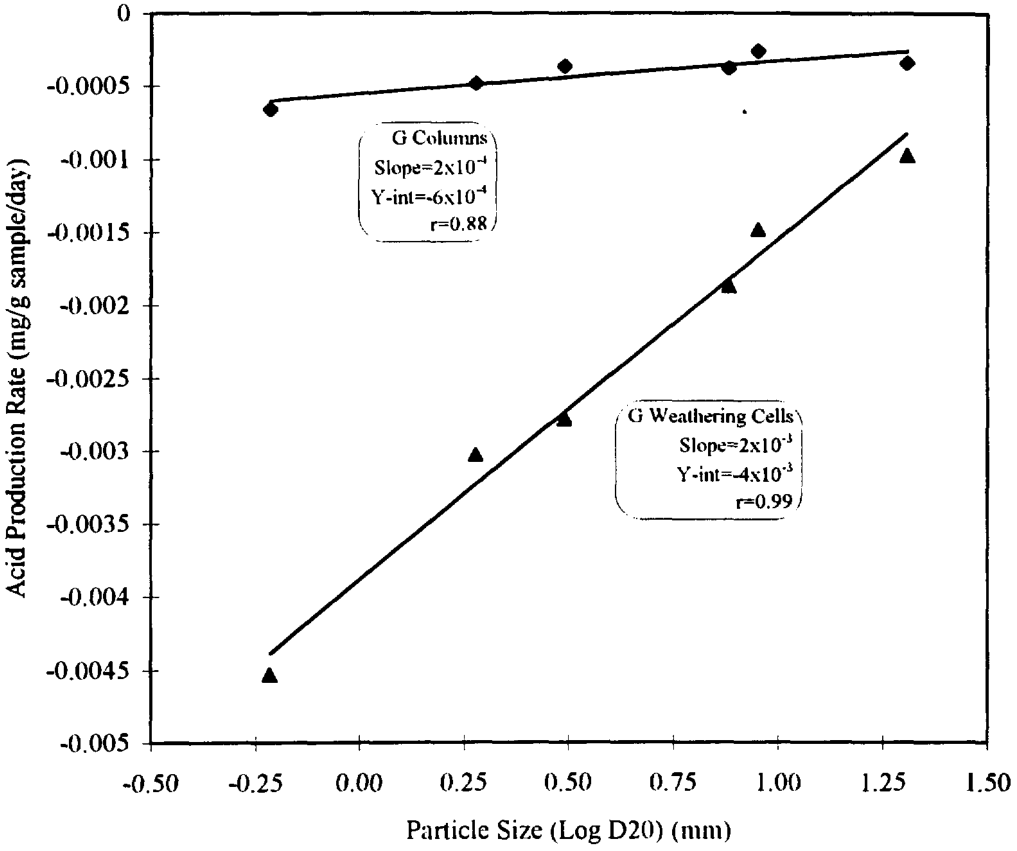
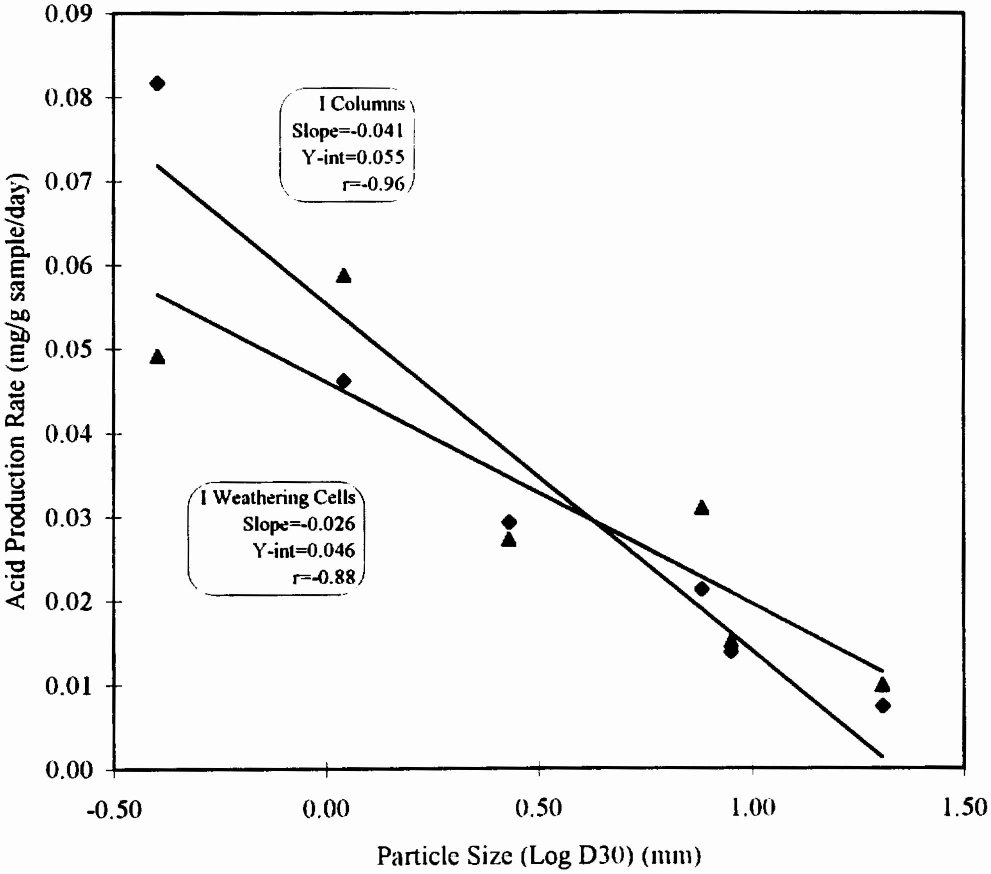


Figure 6: Acid Production Rate vs. Particle Size for Overburden Sample I



alkaline leachate. Due to the overwhelming and obscuring influence of the other factors in the study, however, the actual mechanism by which leachant temperature affected contaminant production was not easily discernible.

Discussion

The interactive effects of *storage* and *leaching interval* produced unique results depending on overburden type. For overburden samples with moderate to high NP (25 to 55‰), such as the "C" and "D" overburden samples depicted in figures 7 and 8, *high temperature* storage in oven conditions for short time periods, such as 2 days, resulted in the samples retaining porewater for a greater percentage of the total storage interval. The acidity produced by pyrite oxidation in these samples was neutralized by carbonate dissolution, and the resulting leachate quality was highly alkaline, as indicated by the downward trending acid production curve for the 2 day sample. As periods of oven storage became longer, however, to 4 and to 7 days, the length of time that the overburden sample retained pore water was a much smaller portion of the total storage interval, and the increased acidity produced by pyrite oxidation (which did not appear to require water) was not neutralized by carbonate dissolution (which does require water). The resulting leachate quality became increasingly acidic, as intervals of oven storage became longer (see figures 5 & 6). The net result was that identical overburden subsplits produced strongly alkaline leachate when leached under certain conditions (high temperature, short leaching intervals (frequent leaching), and strongly acidic leachate when leached under another set of conditions (high temperature, longer leaching intervals (less frequent leaching). Presumably, even longer intervals of oven storage would promote even more acidic conditions.

By contrast, overburden samples leached under *low temperature* (~20°C) conditions, such as for the "C" and "D" overburden humidity cells depicted in figures 9 and 10, retained pore water for longer periods of time, allowing carbonate minerals to dissolve continuously in response to pyrite oxidation. Base pyrite oxidation rates in these samples were lower, as a function of lower temperatures, and carbonate dissolution rates were sufficient to neutralize acidity. In general, all intervals of weathering cell storage allowed sufficient time for acid production and acid neutralizing reactions to reach equilibrium. As a result, few of the weathering cell leaching tests performed on overburden samples with NP's > 25 produced acidic leachate, regardless of pyrite content.

Conclusions

In practical application, the leaching test comparisons of the soxhlet extraction weathering cell techniques indicated that the selection of leaching technique may drastically affect the final result of the analysis. In leach testing of a suite of overburden samples; the high temperature, oven storage conditions of the soxhlet extraction technique result in a higher number of overburden samples being identified as acid producing, particularly if leaching intervals are 7 or more days. Conversely, ambient temperature/high humidity methods such as the humidity cell technique would result in a greater number of alkaline results. The soxhlet extraction technique will also result in higher estimates of acid or alkalinity (depending on sample chemistry) production rates, than would the weathering cell technique. Column leaching tests, due to the absence of the high temperature effects of the soxhlet technique and the high water/rock ratios of the weathering cell technique, would produce leachate quality intermediate between these two extremes and proportional to the particle size of the overburden samples.

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Figure 7: Cumulative Acidity for Oven Stored Overburden Sample C

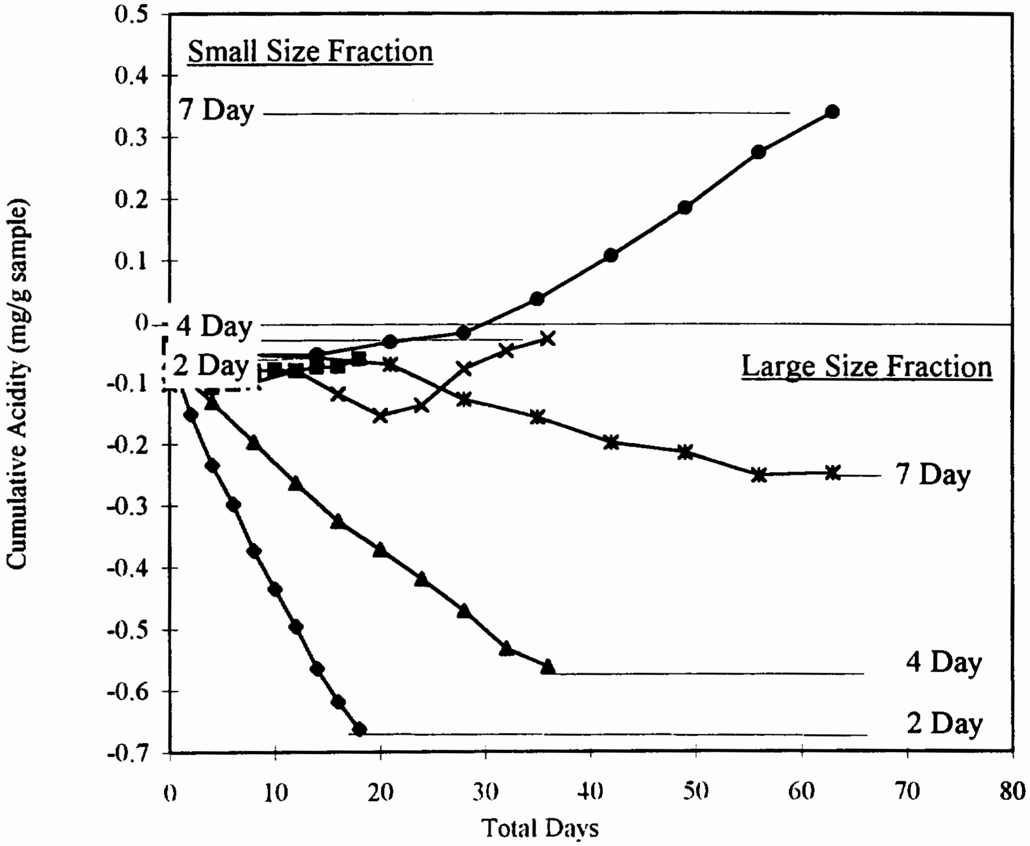

Figure 8: Cumulative Acidity for Oven Stored Overburden Sample D

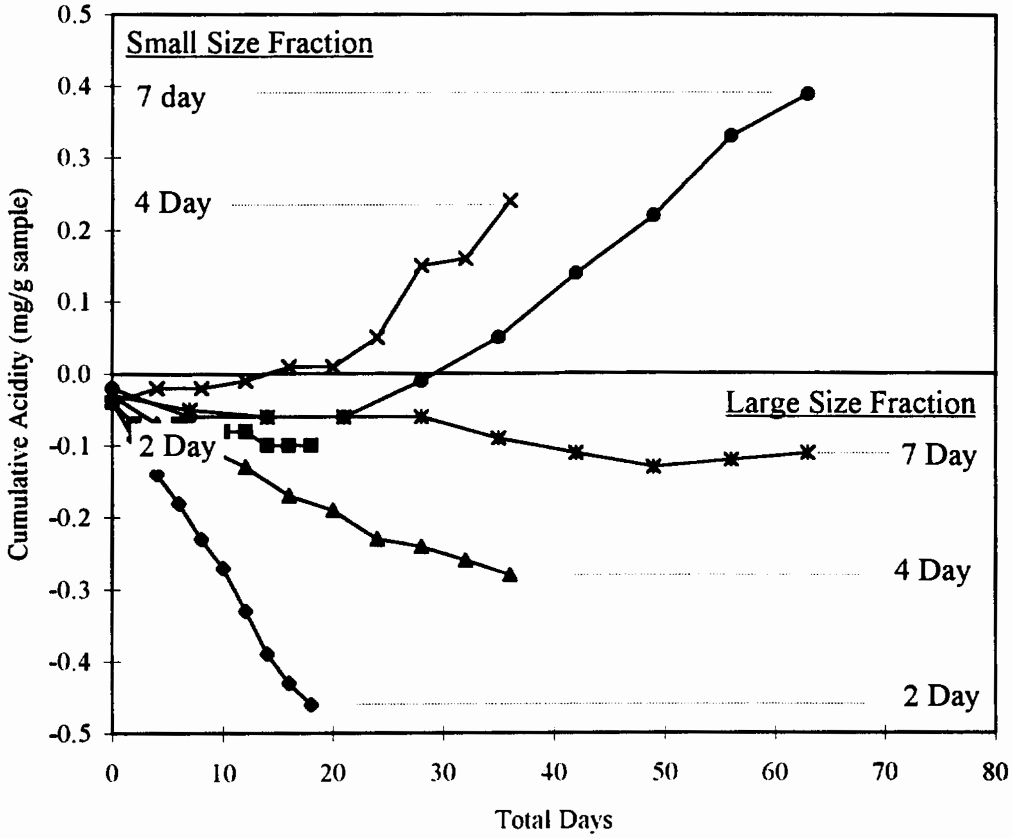

Figure 9: Cumulative Acidity for Weathering Cell Storage Overburden Sample C

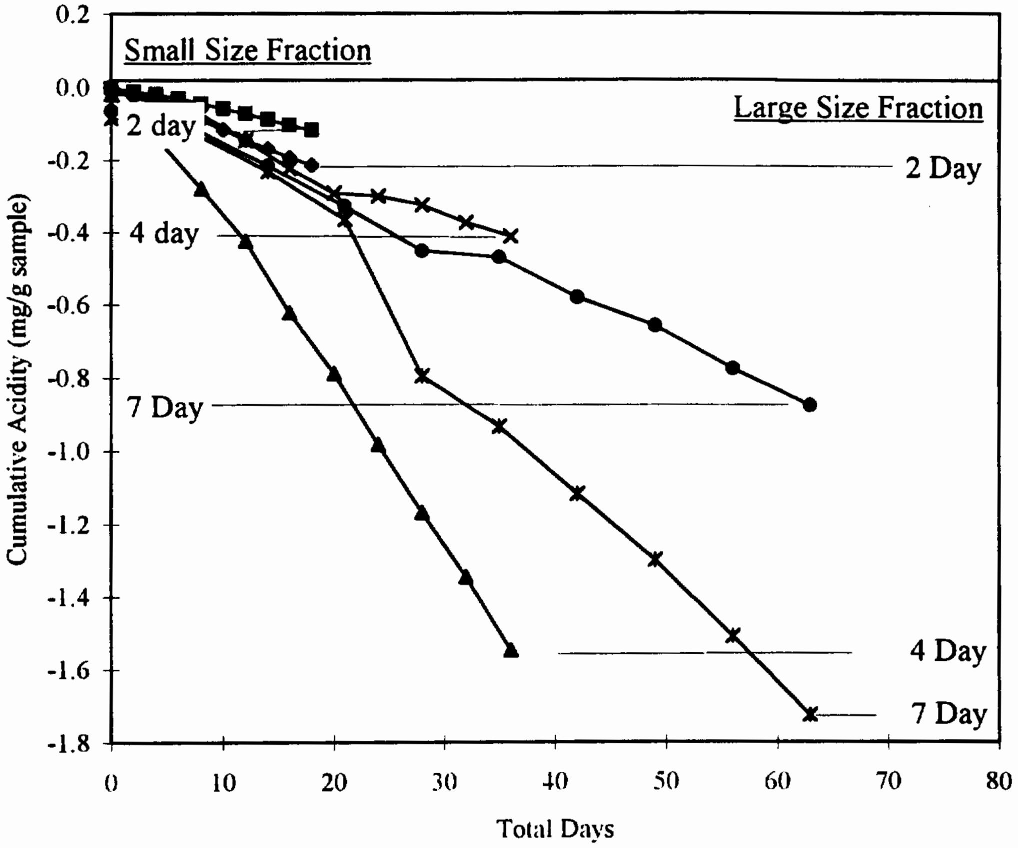

Figure 10: Cumulative Acidity for Weathering Cell Stored Overburden Sample D