Minespoil Amendment with Dry Flue Gas Desulfurization By-Products: Element Solubility and Mobility

R. C. Stehouwer,* P. Sutton, R. K. Fowler, and W. A. Dick

ABSTRACT

Greenhouse column studies of 8 mo duration investigated the solubility and mobility of salts and trace elements in dry flue gas desulfurization (FGD) by-products used for minespoil reclamation. Three minespoils were amended with two dry FGD by-products (lime injection multistage burners ash, LIMB; and, pressurized fluidized bed combustion ash, PFBC) using amounts from 0 to 320 g kg^{-1}. Two of the minespoils also received sewage sludge amendment of 60 g kg^{-1}. Columns were planted with 'Kentucky 31' tall fescue (*Festuca arundinacea* Schreber). Leachate analyses and pH determinations from column mixes were done at the beginning and the conclusion of the experiments. Both FGD by-products were effective in raising pH of the spoil materials. The largest LIMB amendment raised pH to near 12 and resulted in the formation of ettringite [$(Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26\ H_2O)$]. Leachate pH, electrical conductivity, dissolved organic C, Ca, Mg, and S tended to increase with increased FGD amendment, while Al, Fe, Mn, and Zn decreased. Changes in leachate As, B, Cu, Ni, and Se depended on interactions among the type of FGD, the type of spoil, and the presence of sewage sludge, with pH being the most important variable. Overall, with FGD amendments of 120 g kg^{-1} or less, leachate concentrations of most elements of environmental concern were less than drinking water standards. The amount of FGD by-product that can be applied to a minespoil is probably limited by soluble salts and initially high pH levels rather than by trace element loading of spoil or water.

THE 1990 AMENDMENTS to the Clean Air Act mandating a two-stage, 9.07 million Mg reduction in SO_2 emissions in the USA by the year 2000, together with federal and state clean coal technology programs, have spurred the development of a number of dry FGD technologies. These dry FGD technologies are generally smaller in scale and in engineering requirements than wet FGD processes, and require lower capital investment. Dry FGD scrubbers are suitable for retrofit on existing coal-fired power plants and, therefore, present electric utilities with an option for bringing older plants into compliance with clean air legislation. In addition to installation and operating expenses, utilities must also frequently bear increasing costs for landfill disposal of FGD by-products. Development of beneficial uses for FGD by-products demonstrated to be environmentally benign or even beneficial would significantly reduce the costs of SO_2 emission control.

Dry FGD by-products are removed from flue gasses by the particulate emission control systems and, therefore, are a mixture of conventional coal combustion ash (either bed or fly ash), the SO_2 reaction product (primarily anhydrite, $CaSO_4$), and unspent sorbent (generally lime, limestone, or dolomite). Because of the presence of unspent sorbent, dry FGD by-products are usually highly alkaline with significant neutralization potential (Terman et al., 1978; Korcak, 1980; Fowler et al., 1992). Dry FGD by-products, however, may contain large concentrations of soluble salts and also contain some trace elements of environmental concern (Fowler et al., 1992).

One potential high volume use for dry FGD by-products is as an alkaline amendment for reclamation of minelands that contain acid spoil materials. It has been estimated that >0.5 million ha of surface coal mined areas in the eastern portion of the USA need to be reclaimed (Sutton and Dick, 1987). Such areas are frequently strongly acidic because the spoil materials contain Fe disulfides that oxidize to produce large amounts of acid and soluble salts (Hill, 1978). Lack of vegetative cover causes these areas to be highly erosive and drainage waters from such sites can cause severe off-site environmental damage because of acidity, large salt content, and sedimentation (Sutton and Dick, 1987). One type of dry FGD by-product (atmospheric fluidized bed combustion) has been effective as an alkaline amendment for acidic agricultural soils (Terman et al., 1978; Korcak, 1980). These studies, however, did not consider possible environmental impacts of trace elements in the FGD by-products. Furthermore, the amount of alkaline amendment needed for reclamation of acidic minespoils can be 5 to 10 times greater than is used in agricultural soils. Salt and trace element loading from minespoil amendment with FGD by-products will, therefore, also be much greater than what has been previously investigated. The purpose of this greenhouse study was to investigate the efficacy of dry FGD by-products as alkaline amendments for acidic minespoil, and to investigate the potential for adverse environmental impacts from salts and trace elements when FGD by-products are applied in amounts needed for acidic minespoil reclamation.

MATERIALS AND METHODS

Materials

Overburden and underclay materials were sampled from an abandoned mined land (AML) site, and overburden and topsoil were sampled from an active surface coal mine site for use in the greenhouse. Both mine sites were located in east-central Ohio. Overburden refers to the material that was above the mined coal seam and was moved to access the coal. Topsoil refers to the upper 20 cm of the soil profile that was separated from the overburden at the active mine site for replacement on the spoil surface during post-mining reclamation. Underclay refers to material that occurred below the mined coal seam at

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Abbreviations: AML, abandoned mined land; DOC, dissolved organic carbon; EC, electrical conductivity; FGD, flue gas desulfurization; LIMB, lime injection multistage burners; PFBC, pressurized fluidized bed combustion; PVC, polyvinyl chloride.

the AML site and was exposed upon removal of the coal. The term *spoil* will be used to refer collectively to both overburden and underclay materials. These samples were air dried and passed through a 10-mm sieve prior to use in the greenhouse experiments. Characteristics of the spoil materials are given in Table 1.

Dry FGD by-products were sampled from two different scrubber processes: LIMB and PFBC (Table 2). In the LIMB process the sorbent (hydrated lime, $Ca(OH)_2$) is injected into the boiler where it calcines to CaO and reacts with SO_2 in the combustion gasses to produce anhydrite ($CaSO_4$). Temperatures at the point of injection approach 1260°C, and reaction with SO_2 occurs between this temperature and about 870°C. The reaction product and any unspent sorbent are collected with the coal fly ash in electrostatic precipitators. In the PFBC system, the sorbent (dolomite, $CaMg\,(CO_3)_2$) and crushed coal are introduced together into the boiler bed where they are fluidized or suspended by jets of air. Reaction of the sorbent with SO_2 occurs in the boiler at temperatures generally somewhat less than 870°C. This temperature is high enough for $MgCO_3$ to calcine to MgO, but is not high enough to calcine $CaCO_3$. Two FGD by-product streams result from the PFBC process; a granular bed ash material, and smaller particles suspended in the flue gas, which are removed with the particulate emission control system. Both by-product streams contain reaction products, unspent sorbent, and conventional coal bed ash or fly ash. The PFBC by-product from the fly ash stream was used in this experiment.

Reclamation research on acidic spoil materials has shown inclusion of organic amendments, such as sewage sludge, produces superior revegetation results (Sutton and Dick, 1987). For this reason digested sewage sludge (from Rahway, NJ; Table 2), was used together with varying amounts of the FGD by-products, as an amendment for the AML spoils. The sewage sludge was air dried and passed through a 10-mm screen before being used.

Table 1. Characterization of coal minespoil samples used in greenhouse column studies.

Parameter	AML† overburden	AML underclay	Active mine overburden
Particle size, g kg^{-1}			
Sand, 0.05 to 2 mm	173	50	144
Silt, 2 to 50 μm	347	610	549
Clay			
0.2 to 2 μm	408	289	260
<0.2 μm	72	52	47
Extractable cations, mg kg^{-1} (1 *M* ammonium acetate)			
Ca	50	130	1180
Mg	27	38	370
K	65	76	181
pH, 1:1, water	3.1	3.4	5.8
Potential acidity, cmol$_c$ kg^{-1}	33.6	5.3	5.3
Total chemical analysis			
Major elements, g kg^{-1}			
Al	87	95	92
Ba	0.5	0.3	0.4
Ca	<0.1	<0.1	<0.1
Fe	25	<0.1	13
K	20	13	23
Mg	<0.1	<0.1	1.8
S	10.2	2.7	10.6
Si	176	272	235
Trace elements, mg kg^{-1}			
As	46.3	14.4	13.7
Cd	0.8	0.8	0.8
Cr	94.4	95.6	79.8
Cu	26.8	37.3	79.0
Pb	78.0	35.0	30.5
Mo	14.0	1.0	<0.9
Ni	28.5	33.2	65.1
P	1027	351	371
Se	4.5	5.6	4.3
Zn	<0.3	<0.3	<0.3

† AML, abandoned mined land.

Abandoned Mined Land Experiments

The overburden (3 kg) and underclay (4 kg) materials from the AML site were each mixed with sewage sludge (60 g kg^{-1} by dry weight) and with each of the two dry FGD by-products in proportions of 0, 30, 60, 120, and 240 g kg^{-1} by dry weight. These proportions were based on a preliminary incubation study with the AML overburden and FGD by-products that indicated an FGD amendment of 60 g kg^{-1} would raise the overburden pH to 7. Amounts in excess of 60 g kg^{-1} were investigated because in mine reclamation it is desirable to provide excess pH buffering capacity and to encourage downward leaching of alkaline amendments. In addition to the treatments with sewage sludge, overburden material without sewage sludge was combined with the two dry FGD by-products in proportions of 0 and 120 g kg^{-1} by dry weight. This was done both to determine the effects of FGD amendment alone on leachate quality and to investigate interactions between

Table 2. Characterization of lime injection multistage burners (LIMB), pressurized fluidized bed combustion (PFBC), and sewage sludge amendments.

Parameter	LIMB	PFBC	Sewage Sludge
Particle size, g kg^{-1}			na†
Sand, 0.05 to 2 mm	0	255	
Silt, 2 to 50 μm	900	741	
Clay, <2 μm	100	4	
Mineralogy, g kg^{-1}			na
Anhydrite, $CaSO_4$	250	220	
Calcite, $CaCO_3$	150	110	
Dolomite, $CaMg\,(CO_3)_2$	nd‡	230	
Lime, CaO	210	nd	
Portlandite, $Ca(OH)_2$	50	nd	
Periclase, MgO	nd	130	
Fly ash	300	320	
$CaCO_3$ equivalency§	0.59	0.60	0
pH, 1:1, water	12.5	10.5	6.5
Total chemical analysis			
Major elements, g kg^{-1}			
Al	35.2	39.3	34.0
Ba	0.3	0.2	0.1
C (organic)	na	na	312
Ca	360	175	27.6
Fe	55.6	51.7	12.4
K	9.1	5.0	1.5
Mg	6.0	106	3.4
N	na	na	38
Na	3.3	10.3	0.7
P	0.2	0.2	17.8
S	57.7	52.1	14.1
Si	65.8	72.4	na
Trace elements, mg kg^{-1}			
As	55.1	75.0	<0.03
B	233.1	171.2	31.1
Cd	1.0	1.9	6.3
Cr	28.0	36.9	315.2
Cu	21.0	52.5	1174
Pb	16.0	16.0	16.1
Mo	5.9	6.6	11.2
Ni	31.1	52.1	166.4
Se	8.1	5.6	<0.3
Zn	86.0	74.0	1494

† Not analyzed.

‡ Not detected.

§ Neutralizing capacity expressed as a fraction of the neutralizing potential of $CaCO_3$.

FGD by-products and sewage sludge. The <10-mm fractions of all materials were mixed in an air-dry condition and poured into PVC columns (30 cm tall, 15 cm diam.). The columns were mounted on flat PVC plates with a nipple in the center to allow for leachate collection. A complete factorial experimental design was used with all treatments replicated four times and arranged in randomized complete blocks.

Active Mine Experiments

Spoil and topsoil were combined with the FGD by-products to simulate their placement during coal mine reclamation. Overburden (6.2 kg) from the active mine site was mixed with the two dry FGD by-products in proportions of 0, 40, 80, 160, and 320 g kg^{-1} by dry weight. The air-dry mixes were poured into PVC columns (60 cm tall, 15 cm diam.) mounted as previously described. The depth of the FGD-amended overburden ranged from 25 to 36 cm depending on the amount and type of FGD by-product. Topsoil (5.4 kg) was mixed with LIMB (4 g kg^{-1}) and PFBC (8 g kg^{-1}) and placed in the PVC columns above overburden treated with the same FGD by-product. This resulted in a 20-cm layer of FGD-amended topsoil in each column above the FGD-amended overburden. Rates of FGD amendment to the topsoil were based on a preliminary study with LIMB and PFBC that indicated these rates would raise the topsoil pH to 7. A complete factorial experimental design was used with all treatments replicated four times and arranged in randomized complete blocks.

Leachate and Soil–Spoil Analyses

After filling the columns with the air-dry mixes, deionized water was added to the surface of each column in 100-mL increments until between 150 and 200 mL of leachate was collected from the bottom of each column. Each 100-mL increment was allowed to become completely absorbed by the column mix before adding the next increment. Following this initial leaching, an ≈ 5-g soil–spoil sample was collected from the surface 1-cm depth of each column for pH determination, and columns were planted with 30 seeds of Kentucky 31 tall fescue. After an initial 3-mo growth period, fescue was harvested once each month for a total of six harvests. All columns were leached following the sixth harvest using the same procedure described for the first leachate collection. Between these leachings, columns were watered with only enough deionized water to replace evapotranspiration losses. Soil moisture was monitored by weighing the columns weekly and adjusting moisture content to ≈ 0.75 of field capacity on a gravimetric moisture basis. Field capacity was taken to be the gravimetric moisture content of the columns after the first leaching.

Following the final harvest and leachate collection, the columns were sampled at various depth intervals for pH determination. Selected samples were analyzed for crystalline mineral phases by x-ray diffraction and scanning electron microscopy. X-ray diffraction patterns were obtained from randomly-oriented powder mounts using Cu Kα radiation. Crystalline phase assignments were based on published literature, searches of the International Centre for Diffraction Data-international data base (Newton Square, PA) and comparative analyses of reference mineral samples. Scanning electron micrographs were obtained from natural-fracture peds of air-dried samples that were fixed on Al stubs and C coated with a planar magnetron sputter coater.

Leachates were analyzed for pH, electrical conductivity, for As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Se, Si, and Zn by inductively coupled plasma emission spectrometry, SO_4^{2-} by ion chromatography, and dissolved organic C using a C analyzer. The geochemical speciation model MINTEQA2/PRODEFA2 (Allison et al., 1990) was used to chemically speciate the leachate samples and to determine saturation indices for various possible phase controlling solids.

Data analysis was conducted using analysis of variance and single degree of freedom orthogonal contrasts for response variables within each experiment. Linear and quadratic regression analysis was used to assess the responses to amendment proportion within spoils and FGD by-products.

RESULTS AND DISCUSSION

Spoil Chemistry

Both FGD by-products were effective in raising the pH of the mine spoil materials (Tables 3 and 4). Because of its lime and portlandite ($Ca(OH)_2$) content, the LIMB by-product initially increased both spoil and leachate pH to higher levels than the PFBC by-product, in which alkalinity was primarily the result of Ca and Mg carbonates with some periclase (MgO) (Table 2). With both FGD amendments, higher pH values were observed in the AML underclay than in the AML overburden material. This was because the overburden had a higher potential acidity than the underclay due to its larger S content. Peroxide oxidation and titration (Sobek et al., 1978) showed that much of the overburden S was present in a reduced form, presumably as FeS_2, which oxidizes to produce considerable acidity (Caruccio and Geidel, 1978; Hill, 1978).

With amendments of 40 to 60 g kg^{-1} both FGD by-products increased minespoil pH to ≈7 and sustained this pH throughout the 8-mo experiment. Application of alkaline materials in excess of that needed to neutralize acidity in the treated zone may be desirable if downward leaching will increase the pH and base status of the subjacent spoil. Although the LIMB and PFBC materials used in this study had similar $CaCO_3$ equivalencies, it should be noted that dry FGD by-products vary considerably in their total neutralization potential (Fowler et al., 1992). Therefore, the amount of material needed to neutralize acidity in a given spoil will also vary from one type of FGD by-product to another.

Application of FGD by-products in proportions >60 g kg^{-1} initially raised the pH of most leachates and spoils above 8. These initially high pH values tended to decrease with time, with the largest decreases occurring near column surfaces. The decrease was apparently the result of carbonation (conversion to carbonates) of lime (CaO), portlandite, and periclase because the pH was tending toward that of free carbonates in equilibrium with atmospheric CO_2. In the AML overburden, pH was also decreased as a result of acid generation from oxidation of pyrite.

With LIMB amendments of 160, 240, and 320 g kg^{-1}, carbonation was apparently limited below the column surface because spoil pH remained above 8.3. One factor which may have caused this was the formation of secondary minerals. Conditions of high pH, large Ca and SO_4^{2-} concentrations, and adequate moisture led to the

Table 3. Leachate and spoil pH from columns of abandoned mined land (AML) overburden and underclay amended with sewage sludge and lime injection multistage burners (LIMB) and pressurized fluidized bed combustion (PFBC) by-products.

Treatment	Flue gas desulfurization (FGD) amount	Leachate		Overburden/Underclay		
				First	Final	
		First	Final	Surface	0 to 5 cm	15 to 20 cm
	g kg^{-1}	pH				
Overburden with 60 g kg^{-1} sewage sludge						
Overburden + sludge (0 FGD)		3.76	3.72	3.95	3.95	4.05
Overburden + sludge + LIMB	30	6.97	7.26	7.28	7.38	7.38
	60	8.20	7.62	7.68	7.68	7.78
	120	9.81	7.56	8.25	7.83	7.73
	240	11.97	8.52	9.58	7.78	8.55
Overburden vs. Overburden + LIMB†		**	**	**	**	**
Overburden + LIMB: Linear†		**	**	**	**	**
Overburden + LIMB: quadratic§		**	**	**	**	**
Overburden + sludge + PFBC	30	7.34	6.87	6.63	6.60	6.48
	60	7.28	7.47	7.08	7.43	7.45
	120	6.99	7.63	8.00	7.80	7.83
	240	7.85	7.73	8.63	7.98	8.10
Overburden vs. Overburden + PFBC†		**	**	**	**	**
Overburden + PFBC: linear‡		**	**	**	**	**
Overburden + PFBC: quadratic§		**	**	**	**	**
Overburden + LIMB vs. Overburden + PFBC†		**	**	**	**	**
Overburden without sewage sludge						
Overburden (0 sludge, 0 FGD)		3.07	2.81	na¶	3.03	2.98
Overburden + LIMB	120	11.11	7.86	na	7.80	8.08
Overburden + PFBC	120	7.29	7.98	na	7.90	7.95
LSD(0.05)#		1.10	0.55	–	0.15	0.14
Underclay with 60 g kg^{-1} sewage sludge						
Underclay + sludge (0 FGD)		4.73	3.65	4.18	4.63	4.55
Underclay + sludge + LIMB	30	8.18	7.50	7.63	7.78	7.73
	60	10.72	7.63	8.55	7.75	7.78
	120	11.87	8.54	9.63	7.90	8.48
	240	12.03	10.59	11.38	7.98	9.88
Underclay vs. Underclay + LIMB†		**	**	**	**	**
Underclay + LIMB: linear‡		**	**	**	**	**
Underclay + LIMB: quadratic§		**	**	**	**	**
Underclay + sludge + PFBC	30	7.41	7.33	7.30	7.50	7.45
	60	7.28	7.60	8.08	7.75	7.70
	120	7.69	7.79	8.38	7.95	8.15
	240	9.04	7.78	9.00	8.20	8.38
Underclay vs. Underclay + PFBC†		**	**	**	**	**
Underclay + PFBC: linear‡		**	**	**	**	**
Underclay + PFBC: quadratic§		ns	**	**	**	**
Underclay + LIMB vs. Underclay + PFBC†		**	**	**	ns	**
Spoil + FGD vs. Clay + FGD†		**	**	**	**	**

**, ns = Significance at the 0.05 probability level and nonsignificance, respectively.

† Single degree of freedom contrasts.

‡ Linear regression of the response variable against FGD amount.

§ Quadratic regression of the response variable against FGD amount and FGD amount squared.

¶ Not analyzed.

LSD (0.05) value for comparison of spoil treatments with and without sewage sludge with 0 and 120 g kg^{-1} FGD.

formation of ettringite [($Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26\ H_2O$)] (Fig. 1 and 2). Ettringite is highly expansive and cementitious, and thus severely restricted both water movement and root extension below the depth at which it occurred. With LIMB amendment of 240 g kg^{-1}, extensive ettringite formation occurred below 15 cm in the AML overburden and below 8 cm in the AML underclay. In the active mine material, 160 and 320 g kg^{-1} LIMB amendments resulted in ettringite formation throughout the overburden section of the columns. With the PFBC by-product, extensive cementation occurred only when 320 g kg^{-1} were added to the active mine overburden, and was due to the conversion of anhydrite to gypsum ($CaSO_4 \cdot 2\ H_2O$) (Fig. 1 and 2).

Leachate Chemistry

The two leachings of the columns were conducted to study salt and trace element solubility, and their potential for movement from the zone of FGD application both immediately following incorporation and after a long period of reaction, equilibration and plant growth. We did not attempt to simulate any particular patterns of precipitation or percolation. The first leachates were indicative of the most soluble phase present in the FGD by-products, whereas the final leachates were representative of steady-state soil solution chemistry. Because the final leachates provided a better indication of the long-term potential for solute transport we decided to focus

Table 4. Leachate, topsoil and overburden pH from columns of active mine topsoil and overburden amended with lime injection multistage burners (LIMB) and pressurized fluidized bed combustion (PFBC) by-products.

			Final pH		
	Leachate		Topsoil	Overburden	
Flue gas desulfurization (FGD) amount	First	Final	0 to 20 cm	20 to 30 cm	40 to 50 cm
g kg^{-1}	pH				
	LIMB amended topsoil and overburden				
0	6.51	5.93	6.62	6.33	5.91
40	11.94	7.57	6.58	7.76	8.16
80	12.00	9.46	6.37	8.02	8.95
160	11.98	9.67	6.10	7.79	9.19
320	12.14	9.76	6.84	8.45	9.19
Overburden vs. Overburden + LIMB†	**	**	ns	**	**
Overburden + LIMB: linear‡	**	**	ns	**	**
Overburden + LIMB: quadratic§	**	**	ns	**	**
	PFBC amended topsoil and overburden				
0	6.68	6.28	7.10	6.47	6.14
40	8.51	7.92	7.21	7.87	8.02
80	8.70	7.94	7.08	7.87	8.10
160	8.64	8.07	7.45	8.13	8.38
320	9.51	7.77	6.85	8.22	8.91
Overburden vs. Overburden + PFBC†	**	**	ns	**	**
Overburden + PFBC: linear‡	**	ns	ns	**	**
Overburden + PFBC: quadratic§	ns	**	ns	**	**
LIMB vs. PFBC†	**	**	**	ns	**

**, ns = Significance at the 0.05 probability level and nonsignificance, respectively.
† Single degree of freedom contrasts.
‡ Linear regression of the response variable against FGD amount.
§ Quadratic regression of the response variable against FGD amount and FGD amount squared.

our presentation and discussion of solution chemistry on the final leachates. The first leachates (data not given) tended to have somewhat larger maximum concentrations of Ca (with LIMB) and smaller concentrations of Mg and S (with PFBC) than those which occurred in the final leachates. In general, concentrations of most other solutes were somewhat smaller in the first leachates than in the final leachates.

Final leachate chemistry and solute mobility were affected by the type and amount of FGD amendment, the type of minespoil material, the presence of sewage sludge, and interactions among these components. To a large extent, spoil pH was the major factor that governed the interactions among these components and their effects on leachate chemistry.

Leachate dissolved organic C (DOC) increased with increasing LIMB and PFBC amendment in all AML spoil materials. Leachate DOC concentrations were also increased by sewage sludge amendment without FGD (Table 5). As with pH, LIMB amendment produced larger leachate DOC concentrations than did PFBC amendment, and AML underclay gave larger leachate DOC concentrations than did AML overburden. In fact the increases in DOC were strongly correlated with increases in pH above 7 (r^2 = 0.82). These results were expected because the solubility of organic matter increases substantially as pH increases above the neutral range (Stevenson, 1982). Similar responses in leachate DOC were seen with the active mine overburden (Table 6), although overall DOC concentrations were smaller than those of the AML spoils with sewage sludge amendment.

Increasing FGD amendment increased leachate soluble salt concentrations (as determined by EC) with the largest increases resulting from PFBC amendment (Tables 5 and 6). Similar patterns were seen in the FGD amendment effects on concentrations of Ca, Mg, and S, the major elements in the leachates. Although it must be recognized that the leachate samples were not equilibrium solutions, geochemical speciation modeling did show that most of the leachates from FGD-amended minespoils were slightly oversaturated with respect to gypsum (saturation indices ranged from 0.12 to 0.49). This, together with x-ray diffraction and scanning electron microscopy identification of gypsum in the columns (Fig. 1 and 2), indicated that gypsum was the solid that controlled Ca and S solubility in most columns. Exceptions to this were those columns with LIMB amendment of 160, 240, and 320 g kg^{-1}, the same columns in which ettringite had formed. Leachate samples from these columns were undersaturated with respect to gypsum (saturation indices of −0.93 to −2.51), although still oversaturated with respect to ettringite. Ettringite is highly insoluble (log $K_{sp} = -111.32$; Myneni and Logan, 1993) and precipitation of this mineral probably accounted for the decreased Ca^{2+} and SO_4^{2-} activities in leachate samples from these columns. Amendment with PFBC caused large increases in leachate Mg and S concentrations. Chemical speciation showed increasing proportions of Mg and S were in solution as the $MgSO_4$ ion pair. All PFBC leachates were undersaturated with respect to epsomite ($MgSO_4 \cdot 6\,H_2O$), but approached saturation for this very soluble salt (≈300 times more soluble than gypsum; Weast, 1972) in leachates with the largest concentrations of Mg and S. Thus, the presence of Mg in the PFBC by-product gave it a greater potential for salt loading than the LIMB by-product.

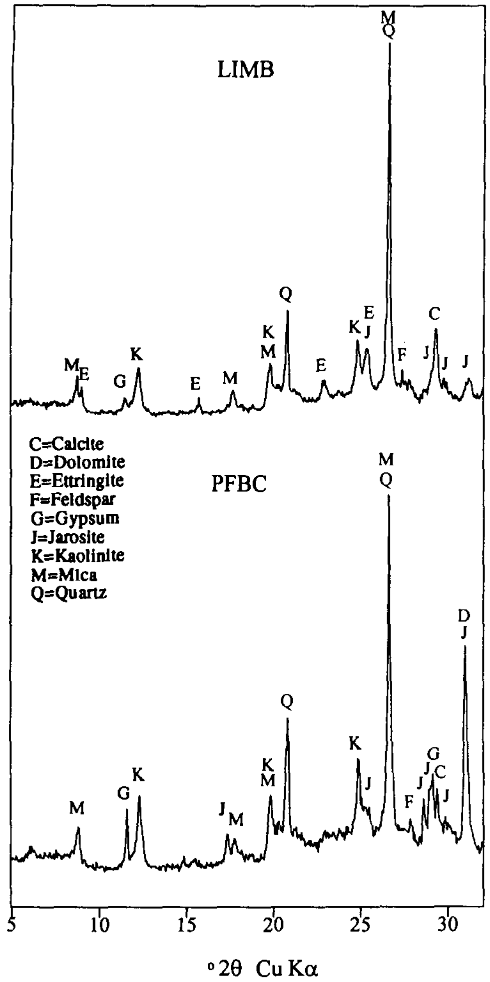

Fig. 1. X-ray diffraction tracings of 15- to 20-cm depth samples of abandoned mined land (AML) overburden amended with 240 g kg^{-1} lime injection multistage burners (LIMB) and pressurized fluidized bed combustion (PFBC).

Leachate concentrations of Al, Fe, Mn, and Zn, metals that are frequently phytotoxic in acid spoils, all decreased with FGD amendment (Tables 5, 6, 7, and 8). This was expected because the solubility of each of these metals is greatest under acid conditions and decreases rapidly with increasing pH (Bohn et al., 1985). Concentrations of Al were greater in leachates from active mine spoil with 160 g kg^{-1} LIMB than from spoil with smaller LIMB amendments (Table 5), which reflects the increased solubility of Al above pH 9. The subsequent decrease in leachate Al with 320 g kg^{-1} LIMB may have been the result of Al incorporation into ettringite. Amendment of the AML overburden with sewage sludge resulted in large decreases in leachate Al and Fe even though the corresponding pH increase was small (from 2.81 to 3.72) relative to pH increases from FGD amendment. This reflected the ability of organic C to form strong complexes with these elements (Stevenson, 1982), and the inverse relationship between soil organic matter and Al^{3+} solubility and phytotoxicity (Kamprath, 1970; Hue et al., 1986). The same effect was not observed with Mn and Zn because the concentrations of these metals were relatively large in the sewage sludge (Table 2).

Tables 7 and 8 give leachate concentrations of elements that are of environmental concern and have been regulated with respect to land application of sewage sludge (USEPA, 1993). Not listed are Hg and Pb. Mercury was below detection limit (<0.04 mg L^{-1}) in all of the column leachates. Lead was detected at a level of 0.12 mg L^{-1} only in leachates from unamended active mine overburden. In all other leachates Pb was below detection limit (0.04 mg L^{-1}). All leachates had very small Cd concentrations (Tables 7 and 8). There was no detectable FGD effect in the AML spoils, while FGD amendment of the active mine spoil resulted in decreased leachate Cd. Thus, there appeared to be little potential for groundwater contamination by these metals as a result of using FGD by-products as minespoil amendments.

In both the AML and active mine spoils, leachate Cu and Ni generally remained constant or decreased when

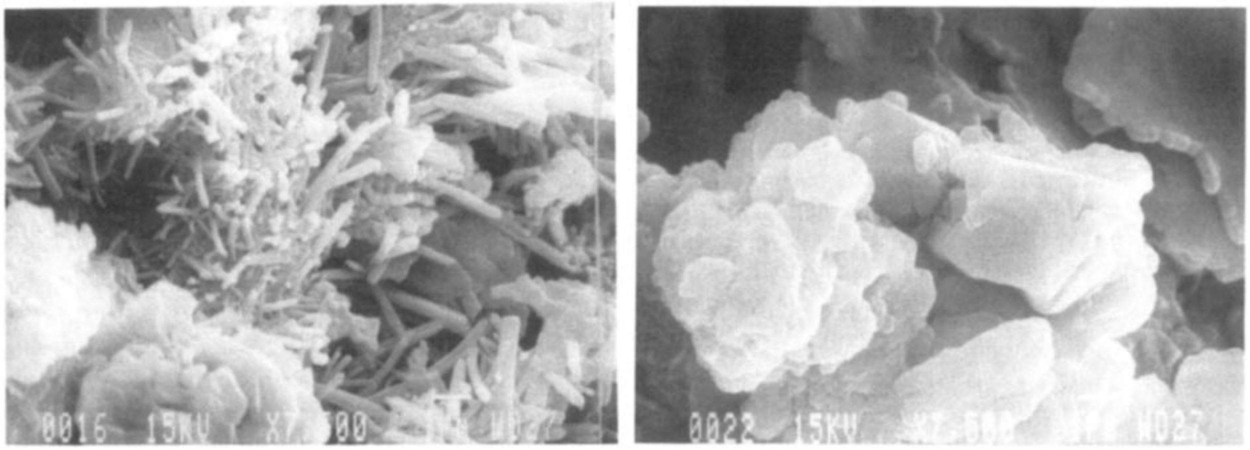
LIMB X7,500 ▬ 1 µm PFBC X7,500 ▬ 1 µm

Fig. 2. Scanning electron micrographs of 40- to 50-cm depth samples of active mine overburden amended with 320 g kg^{-1} lime injection multistage burners (LIMB) and pressurized fluidized bed combustion (PFBC). The LIMB sample shows the characteristic needle-shaped ettringite crystals extending from the surface of a coal fly-ash particle. The PFBC sample shows gypsum crystals.

Table 5. Electrical conductivity and major element composition of final leachates from columns of abandoned mined land (AML) overburden and underclay amended with sewage sludge and lime injection multistage burners (LIMB) and pressurized fluidized bed combustion (PFBC) by-products.

Treatment	Flue gas desulfurization (FGD) amount	EC†	DOC‡	Ca	Mg	S	Al	Fe	Mn
	g kg^{-1}	dS m^{-1}	mg L^{-1}						
Overburden with 60 g kg^{-1} sewage sludge									
Overburden + sludge (0 FGD)		0.96	14	82	35	170	3.04	2.27	2.53
Overburden + sludge + LIMB	30	2.39	57	705	34	557	<0.05	0.14	0.10
	60	2.35	53	692	23	524	<0.05	<0.01	0.01
	120	2.88	151	770	58	631	<0.05	<0.01	0.01
	240	5.44	1050	1280	4	970	0.07	0.02	<0.01
Overburden vs. Overburden + LIMB§		**	**	**	ns	**	**	**	**
Overburden + LIMB: linear¶		**	**	**	ns	**	**	ns	**
Overburden + LIMB: quadratic#		ns	**	ns	**	ns	**	ns	**
Overburden + sludge + PFBC	30	3.83	74	461	560	1060	<0.05	0.08	0.06
	60	4.21	96	477	674	1170	<0.05	<0.01	0.04
	120	6.34	130	452	1300	1760	<0.05	<0.01	0.05
	240	21.97	632	425	7090	7490	0.09	0.07	0.10
Overburden vs. Overburden + PFBC§		**	**	**	**	**	**	**	**
Overburden + PFBC: linear¶		**	**	ns	**	**	**	ns	**
Overburden + PFBC: quadratic#		**	**	**	**	**	**	ns	**
Overburden + LIMB vs. Overburden + PFBC¶		**	ns	**	**	**	ns	ns	ns
Overburden without sewage sludge									
Overburden (0 sludge, 0 FGD)		2.24	5	50	16	48	50.17	1.15	0.87
Overburden + LIMB	120	2.92	55	696	11	648	<0.05	<0.01	<0.001
Overburden + PFBC	120	6.36	52	391	1270	1900	<0.05	<0.01	<0.001
LSD(0.05)‡‡		1.17	61	50	296	368	15.85	ns	0.81
Underclay with 60 g kg^{-1} sewage sludge									
Underclay + sludge (0 FGD)		1.56	39	278	55	373	2.89	20.30	8.28
Underclay + sludge + LIMB	30	2.37	47	655	52	540	<0.05	0.06	0.04
	60	2.06	77	553	41	410	<0.05	0.04	0.04
	120	6.39	2250	1600	10	988	0.17	0.11	0.02
	240	6.23	4490	1160	3	205	<0.05	<0.01	<0.01
Underclay vs. Underclay + LIMB§		**	**	**	ns	ns	**	**	**
Underclay + LIMB: linear¶		**	**	**	**	ns	**	ns	**
Underclay + LIMB: quadratic#		ns	**	**	ns	**	**	**	**
Underclay + sludge + PFBC	30	4.81	84	486	788	1170	<0.05	0.01	0.30
	60	4.99	94	454	1140	163	<0.05	0.01	0.09
	120	9.20	132	448	2230	2760	<0.05	0.01	0.12
	240	21.50	323	465	6420	6760	<0.05	0.04	0.32
Underclay vs. Underclay + PFBC§		**	ns	**	**	**	**	**	**
Underclay + PFBC: linear¶		**	**	ns	**	**	**	ns	**
Underclay + PFBC: quadratic#		**	**	ns	**	**	**	**	**
Underclay + LIMB vs. Underclay + PFBC§		**	**	**	**	**	ns	ns	ns
Overburden + FGD vs. Underclay + FGD§		**	**	**	ns	ns	ns	ns	ns

**, ns = Significance at the 0.05 probability level and nonsignificance, respectively.
† EC, electrical conductivity.
‡ DOC, dissolved organic carbon.
§ Single degree of freedom contrasts.
¶ Linear regression of the response variable against FGD amount.
# Quadratic regression of the response variable against FGD amount and FGD amount squared.
†† LSD (0.05) value for comparison of spoil treatments with and without sewage sludge with 0 and 120 g kg^{-1} FGD.

amended with FGD by-products, except for the largest amendments. These results were consistent with most studies of trace metal behavior in soils which have concluded that trace metal solubility and mobility decrease as pH increases from the acid range (Chaney et al., 1987; Woodbury, 1992). Increases in leachate Cu and Ni with increasing FGD amendment occurred only when spoils were amended with sewage sludge and where pH was also high. This was most notable with LIMB amended underclay, where highly basic conditions increased the solubility of organic matter that was added in the sewage sludge. The first leachates from the 240 g kg^{-1} LIMB amended AML spoils had pH near 12 (Table 3) and very large concentrations of Cu (57.87 mg L^{-1} from underclay, 14.03 mg L^{-1} from overburden) and Ni (8.96 mg L^{-1} from underclay, 3.77 mg L^{-1} from overburden). Copper and Ni were present at much larger concentrations in the sewage sludge than the FGD by-products (Table 2); thus, a relatively constant amount was added to all the AML columns. Apparently, organic matter to which these metals were bound was solubilized and the metals were held in solution through complexation with dissolved organic C. Organic ligands will form relatively stable complexes with these metals (Stevenson, 1982).

Table 6. Electrical conductivity and major element composition of final leachates from columns of active mine topsoil and overburden amended with lime injection multistage burners (LIMB) and pressurized fluidized bed combustion (PFBC) by-products.

Flue gas desulfurization (FGD) amount	EC†	DOC‡	Ca	Mg	S	Al	Fe	Mn
g kg^{-1}	dS m^{-1}	mg L^{-1}						
		LIMB amended topsoil and overburden						
0	9.56	8	404	1180	2990	2.57	1.37	144
40	6.29	86	647	7.00	1320	<0.05	<0.01	0.10
80	5.91	169	225	3.09	719	0.58	<0.01	0.03
160	5.92	278	2.37	2.59	335	5.87	<0.01	0.01
320	15.92	255	2.93	2.64	2030	1.04	0.02	<0.01
Overburden vs. Overburden + LIMB§	ns	**	**	**	**	ns	**	**
Overburden + LIMB: linear¶	**	**	**	ns	ns	**	**	ns
Overburden + LIMB: quadratic#	**	**	**	**	**	**	**	**
		PFBC amended topsoil and overburden						
0	10.58	12	419	1220	3260	0.03	0.06	135
40	20.19	74	438	4740	7550	<0.05	<0.01	1.92
80	24.68	97	443	6290	9530	<0.05	<0.01	0.30
160	29.53	81	448	7630	11300	<0.05	<0.01	0.12
320	8.82	36	442	40	2060	<0.05	<0.01	0.03
Overburden vs. Overburden + PFBC§	**	ns	ns	**	**	ns	ns	**
Overburden + PFBC: linear¶	ns	ns	ns	ns	ns	ns	ns	ns
Overburden + PFBC: quadratic#	**	**	**	**	**	ns	ns	**
LIMB vs. PFBC§	**	**	**	**	**	**	ns	ns

† EC, electrical conductivity.
‡ DOC, dissolved organic carbon.
§ Single degree of freedom contrasts.
¶ Linear regression of the response variable against FGD amount.
# Quadratic regression of the response variable against FGD amount and FGD amount squared.

Leachate concentrations of the oxyanions As, B, and Se generally tended to increase with increasing FGD amendment, with the largest concentrations occurring in leachates from active mine overburden. Concentrations of As and B decreased with the largest LIMB amendments where ettringite had formed in the columns. These elements were present in the FGD by-products (Table 2); thus, some of the increase in leachate As, B, and Se can be attributed to the increasing amounts of these elements added with the LIMB by-product. Because of its expansive and cementitious properties, the formation of ettringite would have limited the contact between leachate water and As, B, and Se present in the FGD by-products, thereby decreasing their concentrations in the leachate. There is evidence that oxyanions of As, B, and Se may substitute for SO_4^{2-} during the precipitation of ettringite that would also decrease their leachate concentrations (Hassett et al., 1990).

Boron is of concern because of the potential for the development of soil solution concentrations which are phytotoxic. Although there was some increase in leachate B with PFBC amendment, much larger increases were observed with LIMB amendment up to 160 g kg^{-1}. These increases were seen in both the AML and the active mine overburden. This was not surprising because total B in the LIMB by-product was much larger than in the PFBC by-product. The most phytotoxic B species are relatively water soluble and easily leached (Woodbury, 1992). In an actual mine reclamation site, leaching because of natural precipitation may rapidly move soluble B below the root zone, thereby decreasing the potential for B phytotoxicity.

Selenium concentrations were much larger in the active mine than in the AML spoil leachates. In the active mine overburden leachates, Se tended to decrease with LIMB amendment. Leachate Se concentrations, however, increased with PFBC amendment up to the 160 g kg^{-1} amendment. Because total Se concentration in the PFBC by-product was less than in the LIMB by-product, the reason for the increase in leachate Se was not readily apparent.

CONCLUSIONS

Both FGD by-products were effective in neutralizing acid conditions in the minespoil materials tested with amendments from 30 to 60 g kg^{-1} by weight. With amendments >30 to 60 g kg^{-1} high leachate pH and large leachate Ca and Mg concentrations indicated some potential for amelioration of phytotoxic conditions below the zone of incorporation when FGD by-products were applied in excess of that required to neutralize acidity in the spoil surface layer. Both FGD by-products also reduced leachate concentrations of Al, Fe, Mn, and Zn, metals that are frequently phytotoxic in acid soils. Decreased solubility and mobility of these metals would also improve water quality.

It appears that one of two factors will probably limit the amount of FGD by-product that can be applied to minespoils. In the case of Mg-containing by-products such as PFBC, large soluble salt concentrations could initially inhibit plant growth and decrease water quality. In the case of CaO and $Ca(OH)_2$ containing FGD by-products, such as the LIMB material, initially high pH levels following application, and the potential for ettringite formation, could both inhibit plant growth. The

Table 7. Trace element composition of final leachates from columns of abandoned mined land (AML) overburden and underclay amended with sewage sludge and lime injection multistage burners (LIMB) and pressurized fluidized bed combustion (PFBC) by-products.

Treatment	Flue gas desulfurization (FGD) amount	As	B	Cd	Cr	Cu	Mo	Ni	Se	Zn
	g kg^{-1}	mg L^{-1}								
	Overburden with 60 g kg^{-1} sewage sludge									
Overburden + sludge (0 FGD)		<0.05	0.41	<0.003	0.009	0.04	<0.02	0.24	<0.27	3.34
Overburden + sludge + LIMB	30	<0.05	0.84	0.003	0.005	0.02	<0.02	0.01	<0.27	0.10
	60	<0.05	1.02	<0.003	0.004	0.01	<0.02	<0.01	<0.27	0.03
	120	<0.05	3.72	<0.003	0.008	0.03	<0.02	0.01	<0.27	0.05
	240	0.14	0.61	<0.003	0.146	5.55	3.42	0.85	0.28	0.07
Overburden vs. Overburden + LIMB†		ns	**	ns	**	ns	**	ns	**	**
Overburden + LIMB: linear‡		ns	ns	ns	**	**	**	**	**	**
Overburden + LIMB: quadratic§		ns	**	ns	**	**	**	**	ns	**
Overburden + sludge + PFBC	30	<0.05	0.55	<0.003	0.014	0.16	<0.02	0.03	<0.27	0.20
	60	<0.05	0.57	<0.003	0.011	0.08	<0.02	0.02	<0.27	0.16
	120	<0.05	0.93	<0.003	0.021	0.06	0.09	0.01	<0.27	0.06
	240	0.07	0.97	0.004	0.043	0.21	0.39	0.11	0.54	0.12
Overburden vs. Overburden + PFBC†		ns	ns	ns	**	ns	**	ns	**	**
Overburden + PFBC: linear‡		**	**	ns	ns	**	**	ns	**	**
Overburden + PFBC: quadratic§		ns	**	ns	ns	ns	**	**	ns	**
Overburden + LIMB vs. Overburden + PFBC†		ns	**	ns	**	**	**	ns	**	ns
	Overburden without sewage sludge									
Overburden (0 sludge, 0 FGD)		<0.05	0.25	<0.003	0.008	0.07	0.01	0.11	<0.27	0.74
Overburden + LIMB	120	<0.05	1.93	<0.003	0.110	<0.01	0.81	<0.01	<0.27	0.13
Overburden + PFBC	120	<0.05	0.69	<0.003	0.013	<0.01	0.07	<0.01	<0.27	0.15
LSD(0.05)¶		ns	1.44	ns	0.036	0.03	0.18	0.09	ns	1.04
	Underclay with 60 g kg^{-1} sewage sludge									
Underclay + sludge (0 FGD)		<0.05	0.53	<0.003	0.010	0.25	0.02	0.96	<0.27	4.30
Underclay + sludge + LIMB	30	<0.05	1.42	<0.003	0.009	0.06	0.05	0.01	<0.27	0.17
	60	0.06	2.68	0.017	0.005	0.19	0.05	0.05	<0.27	0.14
	120	0.22	2.64	<0.003	0.069	11.1	1.94	1.04	0.38	0.08
	240	0.06	0.62	<0.003	0.022	26.1	1.22	3.96	0.27	0.06
Underclay vs. Underclay + LIMB†		**	**	ns	**	**	**	**	**	**
Underclay + LIMB: linear‡		ns	ns	ns	**	**	**	**	ns	ns
Underclay + LIMB: quadratic§		ns	**	ns	**	ns	**	**	ns	**
Underclay + sludge + PFBC	30	0.05	0.74	0.006	0.016	0.13	0.12	0.07	<0.27	0.16
	60	0.05	1.04	<0.003	0.025	0.15	0.34	0.06	<0.27	0.20
	120	0.06	1.60	<0.003	0.016	0.19	0.79	0.06	0.32	0.09
	240	0.22	1.30	<0.003	0.041	0.46	1.09	0.14	0.62	0.15
Underclay vs. Underclay + PFBC†		**	ns	ns	**	ns	**	**	**	**
Underclay + PFBC: linear‡		**	**	ns	**	**	**	ns	**	ns
Underclay + PFBC: quadratic§		**	**	ns	ns	**	**	**	ns	**
Underclay + LIMB vs. Underclay + PFBC†		ns	ns	ns	ns	**	**	**	**	ns
Overburden + FGD vs. Underclay + FGD†		**	ns	ns	**	**	**	**	**	ns

**, ns = Significance at the 0.05 probability level and nonsignificance, respectively.

† Single degree of freedom contrasts.

‡ Linear regression of the response variable against FGD amount.

§ Quadratic regression of the response variable against FGD amount and FGD amount squared.

¶ LSD (0.05) value for comparison of spoil treatments with and without sewage sludge with 0 and 120 g kg^{-1} FGD.

increased solution of organic matter resulting from high pH may also cause increased mobilization of trace metals, especially if organic amendments are applied. With the materials used in this study these undesirable effects were avoided when FGD amendments did not exceed 120 g kg^{-1}. Because of variability in the characteristics of minespoils and FGD by-products, this amendment limit will depend upon the particular FGD by-product and minespoil combination.

In these experiments, with FGD amendments of 160 g kg^{-1} or less, leachate concentrations of trace elements of environmental and regulatory concern remained very small. Most, in fact, were less than primary drinking water standards. It appears therefore, if FGD amendments are limited to amounts that will not cause excessively high pH or phytotoxic salt concentrations, there is little potential for adverse effects on water and soil quality from trace elements.

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Table 8. Trace element composition of final leachates from columns of active mine topsoil and overburden amended with lime injection multistage burners (LIMB) and pressurized fluidized bed combustion (PFBC) by-products.

Flue gas desulfurization (FGD) amount	As	B	Cd	Cr	Cu	Mo	Ni	Se	Zn
g kg^{-1}	mg L^{-1}								
	LIMB amended topsoil and overburden								
0	0.05	1.40	0.046	0.021	0.04	<0.02	2.45	0.94	2.67
40	<0.05	1.34	0.013	0.018	0.04	0.58	<0.01	1.17	0.02
80	<0.05	0.55	0.013	0.036	0.06	1.50	0.01	0.65	<0.01
160	<0.05	4.16	0.010	0.021	0.03	0.88	<0.01	<0.27	0.02
320	0.09	2.67	0.010	0.008	0.23	0.96	0.03	0.55	0.01
Overburden vs. Overburden + LIMB†	ns	ns	**	ns	ns	**	**	ns	**
Overburden + LIMB: linear‡	ns	**	**	ns	**	ns	ns	ns	ns
Overburden + LIMB: quadratic§	ns	**	**	ns	**	**	**	**	ns
	PFBC amended topsoil and overburden								
0	<0.05	1.53	0.032	0.020	0.02	<0.02	1.77	1.33	1.33
40	<0.05	1.00	0.007	0.024	0.01	0.09	0.02	2.17	0.04
80	<0.05	0.91	0.007	0.034	0.01	0.20	0.01	3.03	0.03
160	<0.05	0.79	0.005	0.036	<0.01	0.34	<0.01	2.83	<0.01
320	<0.05	1.35	<0.003	0.009	<0.01	0.25	<0.01	0.55	0.01
Overburden vs. Overburden + PFBC†	ns	ns	**	ns	ns	ns	**	**	ns
Overburden + PFBC: linear‡	ns	ns	ns	ns	ns	ns	ns	ns	ns
Overburden + PFBC: quadratic§	ns	ns	ns	**	**	**	**	**	**
LIMB vs. PFBC†	ns	**	ns	ns	**	**	ns	**	ns

**, ns = Significance at the 0.05 probability level and nonsignificance, respectively.

† Single degree of freedom contrasts.

‡ Linear regression of the response variable against FGD amount.

§ Quadratic regression of the response variable FGD amount and FGD amount squared.

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