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**THE USE OF COAL FLY ASH FOR SOIL STABILIZATION
Topical Report**

**By
T. H. Brown
M. A. Brown
S. S. Sorini
G. Huntington**

December 1991

Work Performed Under Contract No. FC21-86MC11076

**For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia**

**By
University of Wyoming Research Corporation
Western Research Institute
Laramie, Wyoming**

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Morgantown Energy Technology Center
P.O. Box 880
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P.O. Box 3395
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SUMMARY

The use of solid fossil fuels results in significant volumes of waste materials. Costs associated with the disposal of such material is becoming a heavy burden for power plant operations. Alternative uses for fly ash that reduce the volumes of material requiring disposal and that provide economic benefit to the power plant operation must be developed. This circumstance provides the incentive for the research conducted under Task 3.4.2 - Use of Solid Waste for Physical Stabilization of the 1991 Cooperative Agreement Plan, and described in this report. The work described herein constitutes the initial phase of a research effort to assess the ability of various fly ash materials to stabilize soils, wastes, and other materials. A major effort will be made to understand the reactions and mineral formations responsible for stabilization and/or cementation. This research effort is anticipated to be ongoing. The achievement of ultimate goals will require several years of intense work.

The objective of this work was to examine the potential use of Wyoming subbituminous coal fly ash materials for cementation of soil materials. The emphasis of the work was to determine strength relationships of soil/fly ash mixtures and to determine the mineral formation characteristics responsible for strength. Also, the potential for toxic element release from fly ash use sites was investigated through experiments using selective dissolution and colloidal material extraction.

The work was done under four subtasks designed to meet the objectives described above. The subtasks were: (1) characterization of fly ash and the reaction products of the soil/fly ash mixtures, (2) unconfined compression strength of soil/fly ash mixtures, (3) selective dissolution of soil/fly ash mixtures, and (4) investigation of the formation of mobile colloids in soil/fly ash mixtures. Subtasks 1 and 2 were designed to provide information relative to the amount of strength developed from the soil/fly ash reactions and to the minerals responsible for the formation of strength. The selective dissolution task (subtask 3) was done to provide solution data that could be used to assess the nature of the solid phases controlling element concentrations in solution and to determine if toxic elements are released from the soil/fly ash mixtures by selective dissolution methods that simulate weathering. Subtask 4 was planned to determine if mobile colloids formed as a result of soil/fly ash interactions. Mobile colloids could provide a mechanism for the transport of toxic elements or compound into water sources.

Specimens made from Laramie River (LR) fly ash had higher unconfined compression strength and more brittleness than the specimens made with Dave Johnston (DJ) fly ash. However, soil/DJ fly ash mixtures that were cured for 28 days had relatively good strengths without the brittleness that the LR specimens developed. These characteristics of the DJ fly ash may be important attributes for road stabilization applications. The detailed mineralogical evaluation provides some insight into which minerals may enhance development of strength in these materials. In general, selective dissolution of the soil/fly ash mixtures shows that many of the potentially toxic elements (e.g., B, Cr, Fe, Mn, Ni, Pb) are associated with the sulfide phase (HNO_3 extractable) and with the residual material. In this study, the dynamics of elemental release from the element pools did not result in toxic conditions. The formation of colloidal material capable of mobilizing potentially toxic elements was not found in the soil/fly ash mixtures. Apparently, the high pH of the materials enhanced immobilization of the high molecular weight material.

SECTION I.
CHARACTERIZATION OF FLY ASH AND REACTION PRODUCTS
OF THE SOIL/FLY ASH MIXTURES

Introduction

Composition of Fly Ash

Fly ash materials generated at coal-fired power plants are typically composed of unburned carbon, detrital quartz, amorphous material, and many trace minerals. The heterogeneity of fly ash results from variations in coal rank (e.g., bituminous, subbituminous, lignite), seam-to-seam changes within the same mine area, and conditions existing during the burning process (Hemmings and Berry 1988). These variations lead to different interbedded minerals that are differentiated largely on the basis of Ca and Fe and to a lesser extent on Na/K and Al/Si ratios. Heterogeneity in fly ash extends to the microstructural level as there appears to be phase separation of glasses in some individual particles (Hemmings and Berry 1988).

McCarthy (1988) determined that five major mineral groups exist in most coal fly ash: silica, aluminosilicates, carbonates, sulfides, and chlorides. It was also found that most fly ash consists of at least 50% amorphous material or glass. Fly ash is principally categorized as either Class C or Class F. Class C fly ash is characterized by its high-Ca concentration and typically contains the minerals listed in Table 1. Manz and Manz (1985) noted that the Class C fly ash contains less glass than Class F fly ash. Also, the Class C fly ash was found to have cementing characteristics when hydrated because of the presence of lime in the ash.

Table 1. Common Mineral Phases Identified in Class C Fly Ash (Roy et al. 1985)

Lime	CaO
Tricalcium aluminate	$\text{Ca}_3\text{Al}_2\text{O}_6$
Anhydrite	CaSO_4
Tricalcium silicate	Ca_3SiO_5
Melilite	$\text{Ca,Mg,Al}(\text{Si}_2\text{O}_7)$
Bredigite, larnite	Ca_2SiO_4
Ferrite spinel	$(\text{Mg,Fe})(\text{Fe,Mg})_2\text{O}_4$
Merwinite	$\text{Ca}_3\text{Mg}(\text{SiO}_4)_2$
Nepheline	NaAlSiO_4
Quartz, cristobalite	SiO_2
Al-huayne	$\text{C}_4\text{A}_3\text{S}$
Periclase	MgO
Feldspar	(Na,Ca,Al silicate)
Pyroxene	(Mg,Fe,Ca,Al silicate)
C_{12}A_7	$12\text{CaO} \cdot 7\text{Al}_2\text{O}_3$

Class F fly ash (silico aluminous) commonly contains the crystalline phases listed in Table 2. Quartz is a major crystalline phase in both fly ash classes. Silica is predominantly unaltered by the combustion process and commonly is in the form of detrital quartz of sand-or-silt size particles (Hemmings et al. 1987). McCarthy (1988) described silica as ubiquitous in fly ash.

Table 2. Crystalline Phases Identified in Class F Fly Ash (Roy et al. 1985)

Mullite	$\text{Al}_6\text{Si}_2\text{O}_{13}$
Quartz	SiO_2
Magnetite-ferrite	$\text{Fe}_3\text{O}_4-(\text{Mg},\text{Fe})(\text{Fe},\text{Mg})_2\text{O}_4$
Hematite	Fe_2O_3
Anhydrite	CaSO_4

Oxides such as lime (CaO) and periclase (MgO) are very common in coal fly ash. Roy et al. (1985) stated that crystalline CaO typically accounts for 2 to 5% of the total CaO present. This mineral is thought to be deposited as a film instead of individual crystals. The formation of these oxides is attributed to a variety of causes including the release of CO_2 upon carbonate decomposition (Hemmings and Berry 1988), the oxidation of Ca associated with the organic fraction in subbituminous coals (McCarthy 1988), the oxidation of organic magnesium in lower rank coals, and the oxidation of dolomite ($\text{CaMg}(\text{CO}_3)_2$) in the original coal.

Pyrite (FeS_2) and organics in coal oxidize to form SO_2 and metallic oxides such as magnetic spinel, hematite, magnetite, and ferrite spinel (McCarthy 1988). Norton et al. (1986) reported that typical combusted coal fly ash contains approximately 20% magnetic oxides. Magnetite is the principal magnetic phase present in most fly ash. This phase is often referred to as spinel or ferrite spinel because varying amounts of Mg and Al replace Fe^{2+} and Fe^{3+} during mineral formation (McCarthy 1988). Hemmings and Berry (1988) noted that the ferrite spinel phase precipitates in either dendritic masses intergrown with glass or as octahedra.

The volatilization and sulfation of chlorides generate HCl and sulfates, namely CaSO_4 . CaSO_4 is often detected in high-Ca fly ash because excess CaO is used to scrub SO_2 from the off-gases (McCarthy 1988). According to Manz and Manz (1985), the form of the Ca in a fly ash is very important because it influences the reactivity of the material.

Aluminosilicate clays melt at high temperatures, forming glasses which subsequently dissociate to form various amorphous and crystalline aluminosilicate phases (Hemmings and Berry 1988). Mullite forms through an exothermic reaction (Wada 1989) as the original coal is burned. McCarthy (1988) noted the presence of mullite in nearly all subbituminous and bituminous coal fly ash.

Other minerals common in coal fly ash are: tricalcium aluminate ($\text{Ca}_3\text{Al}_2\text{O}_6$), merwinite ($\text{Ca}_3\text{Mg}(\text{SiO}_4)_2$), and melilite ($(\text{Ca},\text{Na})_2(\text{Al},\text{Mg})(\text{Si},\text{Al})_2\text{O}_7$). Tricalcium aluminate dominates in fly ash with high-Ca concentrations, and merwinite will dominate if the MgO concentration is high. The term melilite includes the minerals akermanite ($\text{Ca}_2\text{MgSi}_2\text{O}_7$), gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$), and Na-melilite ($\text{NaCaAlSi}_2\text{O}_7$). Significant amounts of MgO and CaO are needed for both melilite and merwinite to occur (McCarthy 1988).

Minerals such as hercynite (FeAl_2O_4), wustite (Fe_{1-x}O), fayalite (Fe_2SiO_4), and iron sulfides (FeS) are commonly detected in ash formed in reducing environments (Hemmings and Berry 1988).

Hemmings and Berry (1988) did a study on the speciation of size and density fractionated fly ash. They found that size separating fly ash samples increases the amount of glass or amorphous material in proportion to the amount of crystalline material (McCarthy 1988; Hemmings and Berry 1988). The use of density fractionations was found to be more successful (Hemmings and Berry 1988). In this study, before analysis, the samples were separated into three density ranges: $<0.79 \text{ g/cm}^3$, $1.5\text{--}2.0 \text{ g/cm}^3$, and $2.5\text{--}2.75 \text{ g/cm}^3$. The lightest fraction consisted of primarily well-formed, smooth-surfaced cenospheres that ranged in size from 15 to 60 μm . The middle density fraction contained irregular-shaped, vesicular particles of partially combusted carbon. This fraction also contained some well-formed spheres. The most dense fraction was made up of irregular, partly fused, fire-polished, detrital quartz. In another analysis of size-fractionated fly ash, Joshi et al. (1985) discovered that partially combusted carbon particles increased in abundance in the coarser fractions.

Grutzeck et al. (1985) reported that glass is present in all coal fly ash. The glassy particles are formed in the high temperatures reached during the combustion of coal. Spheroidization of the fly ash particles results from surface tension forces acting on the melt (McCarthy 1988). Fly ash exits the furnace via the gas stream and is quenched into a glassy solid state as it moves to a lower temperature. Fly ash particles come in a variety of shapes that include spheres and incomplete spheres, vacuoles with glassy agglomerates, and unburned or partially burned carbon. The surfaces of the spheres can be glassy and smooth, rough and pebbly, and partially powder covered. In addition, they can be hollow (cenospheres), hollow filled with smaller spheres (plerospheres), or solid. Hemmings and Berry (1988) found that most of the glassy fly ash particles are hollow spheres with varying wall thickness while other particles form droplets. These variations lead to mineralogical and chemical differences between fly ash particles. In general, the coarser particles were more crystalline and less glassy than the fine particles because they cooled at a slower rate. In addition, the smaller particles are very reactive. Distinctions in the structure of glass can be made between high-Ca and low-Ca fly ash. Calcium aluminate (melilite) or pyrosilicate (Si_2O_7 or SiAlO_7) controls the amorphous structure of high-Ca fly ash, and the glass in low-Ca fly ash is siliceous (Hemmings and Berry 1988). In addition to these distinctions, Berry et al. (1986) report that the glass in fly ash can be classified into three general types by density separation. The

lowest density glass has low-Fe and low-CaCO₃ concentrations and consists of hollow spheres of aluminosilicate, mullite, and glass ceramics. The intermediate density glass is high in Fe and is therefore a ferroaluminosilicate that also contains mullite and glass ceramics. The highest density glass is made up of spinel/hematite crystals embedded in an Fe-substituted glass. An evaluation of the molecular structure of the fly ash using a microprobe, Hemmings and Berry (1988) found different aluminosilicate glasses coexist over a wide range of compositions. The materials ranged in composition from highly polymerized tectosilicates to highly modified, depolymerized structures with many nonbridging oxygens. The aluminosilicate glasses were found to be modified by the inclusion of oxides (e.g., Na, K, Mg, Ca, and Fe).

Reactions of the Fly Ash Materials

It is believed that the reactive phases contributing to the pozzolanic and cementitious properties of the Class C fly ash are CaO, Ca₃Al₂O₆, CaSO₄, Ca₃SiO₅, Ca₂SiO₄, and glass (Manz and Manz 1985). McCarthy et al. (1984) reported that the main crystalline phases present in Class F fly ash (mullite, quartz, magnetite-ferrite, and hematite) do not react during hydration. Manz and Manz (1985) stated that the glass in Class F fly ash hydrates slowly in the presence of lime or alkalis. Carles-Gibergues and Aitcin (1986) found that the sulfates from Class F fly ash react to form ettringite (Ca₆Al₂(SO₄,SiO₄,CO₃)₃(OH)₁₂·26 H₂O), and that increases in long-term compression strength resulted from the formation of a secondary calcium-silicate-hydroxide gel.

Grutzeck et al. (1985) determined that fly ash is a nucleation and growth site for ettringite in addition to being a source of soluble alumina. The copious amounts of Al in fly ash remove soluble Ca and SO₄²⁻ leaving a favorable environment for the formation of ettringite. Also, they observed the early formation of ettringite in high-Ca fly ash. Grutzeck et al. (1985) suggested that during the early stages of hydration, Ca²⁺ is adsorbed on the surface of the fly ash causing the concentration of Ca ions in solution to decrease. The decrease in soluble Ca levels stagnated the precipitation of Ca(OH)₂, prolonging the hydration reactions.

Grutzeck et al. (1985) reported that both high- and low-Ca fly ash act as a retardant to the hydration processes in cements. However, the high-Ca fly ash is more efficient. A possible reason for the cause of hydration retardation was suggested earlier in the discussion about ettringite. Manz and Manz (1985) also concluded that the hydration retardation might be partially due to the chemisorption of Ca²⁺ ions to the fly ash surface. They assumed that this process action caused a delay in the nucleation and crystallization of calcium-silicate-hydroxide and Ca(OH)₂ and the subsequent delay of cement hydration. Ghose and Pratt (1981) showed that the addition of fly ash to cement caused an increase in the period of induction, and the time to reach the peak of the hydration exotherm. They also noted that the hydration of both silica and alumina was retarded by the fly ash and that the reactions between fly ash particles was slow and inconsistent.

In addition to slowing down the hydration reactions in cement pastes, fly ash is used for its pozzolanic capabilities. Fly ash with pozzolanic characteristics can react with lime to form cement-like products. The better the pozzolanic properties, the better the strength will be of a mix with either soil or cement. Baker and Laguros (1985) showed that calcium-silicate-hydroxide gel formation enhances the strength of fly ash mixtures. A deterrent to strength development in fly ash mixtures is substantial amounts of unburned carbon particles which interfere with hydration reactions (Joshi et al. 1985).

A considerable amount of work has been done on the use of fly ash as pozzolanic material for soil-cements. However, the chemistry associated with its use in these applications has not been completely developed. The objective of this research was to investigate fly ash chemistry when used in combination with soil materials in an attempt to further understand fly ash cementation.

Materials and Methods

Three fly ash materials were sampled for analysis. Two of the fly ash materials were collected from the Dave Johnston power plant (DJ) located near Glenrock, Wyoming. The plant is a conventional, coal-fired plant. The first material is comprised of fly ash generated in DJ Units 1 and 2 and the second material was collected from Unit 3. The third material was generated at the Laramie River (LR) power plant located near Wheatland, Wyoming. The LR plant is a coal-fired system that uses lime injection to capture SO_2 from the air stream. Samples collected from the DJ plant consisted of eight samples from Units 1 and 2 and one sample from Unit 3. Four samples were collected from the LR plant. Samples collected from DJ Units 1 and 2 and LR were separated by screening into four size fractions: +120, +240, +400, and -400. The DJ Unit 3 sample was not screened because the process did not delineate separations between mineral groups. Total elemental analysis was done on the fly ash materials. Three digestion methods were used, depending on the element being evaluated. These methods consisted of acid digestion (EPA Method 3050), HNO_3 -HCl-HF digestion (WRI-024), or Na_2O_2 . Elemental analysis was done using a Thermal Jarrell-Ash Auto Comp 1100 inductively coupled plasma (ICP) spectrophotometer. The elements determined were Al, Sb, As, Ba, Be, Bi, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Hg, Mo, Ni, P, K, Se, Si, Ag, Na, Sr, Th, V, and Zn. The oxide forms of Si, Al, Fe, Ca, Mg, Na, K, P, Mn, S, and Ti were calculated from measured concentrations.

The soil materials used for this research were collected from the county road located south of the DJ power plant. The soils used were identified as sample numbers RS-1, RS-3, and RS-4. Soil RS-1 was classified as a sand. The RS-3 and RS-4 soils were loamy sands. To provide some indication of the surface reactivity for cation adsorption of the soil materials, the cation-exchange capacity (CEC) of RS-4 was determined using the Rhoades (1982) methods. A CEC value of about 8 meq/100 g of material was determined.

Chemical Evaluations of Fly Ash Leachates

Fly ash extracts were prepared from eight samples collected from the DJ power plant Units 1 and 2, and from four samples collected from the LR power plant. Solution extracts were collected from each sample to determine if fly ash sources were different. Chemical evaluations were not done on extracts from the fly ash sample collected from DJ Unit 3. Duplicate subsamples were collected from each fly ash sample to be used to generate 1:1 extracts. The DJ fly ash was extracted using 100 g of fly ash and 100 mL of Milli-Q water. After the water was mixed with the fly ash, each sample was stirred, and the beaker was covered with Parafilm. Two hours later, each sample was stirred. The solutions were left overnight and were filtered through Whatman #42 using Buchner funnels under suction.

The same procedure was followed for the LR fly ash samples, except that 400 g of ash was mixed with 400 mL of Milli-Q water. This was done so that more extract could be obtained for analysis. After the water had percolated into the LR fly ash samples, the fly ash had hardened to the point that it could not be stirred. After sitting overnight, the samples had solidified in the beakers. The hard material was scraped and crushed, stirred for 15 minutes, and filtered. The solution extract was refrigerated until analyzed. For comparison purposes, another 1:1 extract was mixed using 500 g of LR fly ash and 500 mL of Milli-Q water. The samples were stirred constantly for 3 minutes and filtered. The samples did not solidify during this process. The solution extract was held in cold storage until analyzed.

Each extract was tested for pH using a Sargent-Welch Meter Model MPT, and for conductivity using a YSI Model 34 conductance-resistance meter. The fly ash extracts were analyzed for Al, Sb, As, Ba, Be, Bi, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Hg, Mo, Ni, P, K, Se, Si, Ag, Na, Sr, Th, V, Zn, F⁻, Cl⁻, Br⁻, NO₃⁻ and SO₄²⁻. Analysis for elements was done using a Thermal Jarrell-Ash Auto Comp 1100 inductively coupled plasma (ICP) spectrophotometer. Anions were analyzed using a Dionex 400i ion chromatograph.

X-ray Diffraction Analysis

X-ray diffraction (XRD) analysis was done on soil/fly ash samples that were crushed with a mortar and pestle in an ethanol slurry. The samples were then mounted on glass slides and sample holders. Bulk mineralogy of the fly ash and fly ash mixtures was determined by XRD using an automated, computer-driven Scintag PAD V diffractometer system. The diffractometer is equipped with an intrinsic germanium, solid-state detection system and computer-assisted, user-interactive software supplied by the manufacturer. The system was operated at 45 kV and 40 aA using Ni-filtered CuK α radiation. Spectra were acquired from 2 to approximately 70° 2 θ at 2°/min. The diffractograms were compared to the Joint Committee on Powder Diffraction Standards files for phase identification.

The fly ash minerals that were expected to be present in most samples are listed in Table 3.

Table 3. Minerals Commonly Detected in Fly Ash

Quartz	Mullite
Hematite	Anhydrite
Lime	Tricalcium aluminate
Melilite	Tricalcium silicate
Bredigite/larnite	Ferrite spinel
Merwinite	Nepheline
Al-huayne	Periclase
Feldspar	Pyroxene
Brownmillerite	Thenardite

Density Separation for XRD Analysis

Four columns were prepared using RS-4 soil and 15% LR fly ash. The columns were 3 inches in diameter and 6 inches in length. Columns 1 and 2 were prepared under a reduced environment and saturated water conditions. Columns 3 and 4 were prepared in an ambient-no gas/nonsaturated environment. A density separation was performed using bromoform (specific gravity 2.85) on columns 2 and 4. Two mixed soil/fly ash columns were air dried and ground with a mortar and pestle. Each sample was then added to a separatory funnel that was filled with pure bromoform. The samples were stirred in the funnel, and the heavy minerals were allowed to settle. The heavy particles were removed, cleaned of bromoform using acetone, and set aside to dry. The remaining sample was drained from the funnel, cleaned of bromoform with acetone, and returned to the separatory funnel with a bromoform-acetone mixture. To make a solution with a specific gravity of 2.51 g/cm³, 120 mL of bromoform was added to 28 mL of acetone resulting in a 30:7 ratio. The same procedure was followed as with the first separation and the heavy portion of this solution was the medium density. The fraction that floated on the surface of the 2.51 g/cm³ solution was the low density material.

Scanning Electron Microscopy/Energy Dispersive X-ray Analysis

The samples were mounted to either an aluminum or carbon stub with colloidal graphite and then coated with either gold or carbon using an Edwards Sputter Coater S105B. All of the samples were very friable and difficult to mount onto sample stubs. The irregular surfaces made it difficult to achieve an even coat.

Samples were analyzed using an Amray Model 1820 computer-enhanced, digital-imaging scanning electron microscope (SEM) (100-s count, 10.00 kV, 35° take-off angle). The SEM is equipped with a Noran Model 5500 Series II fully computer-operated energy dispersive X-ray analyzer (EDXA), which was used for semiquantitative elemental analysis.

Results and Discussion

Chemical Characterization of Fly Ash

The chemical composition of the fly ash generated in the DJ Units 1 and 2, DJ Unit 3, and the LR plant are listed in Tables 4 and 5. The composition of fly ash collected from the DJ Units 1 and 2 and DJ Unit 3 were similar with respect to the oxides (Table 4). However, variation in elemental composition is apparent (Table 5). This variation is probably due to differences in combustion environment and coal composition. The largest variations were found for Ba, Be, Pb, Se, Ag, and Sr. The Ba levels found in the DJ Unit 3 fly ash were 890 mg/L compared to 0.2 mg/L found in the DJ Units 1 and 2 fly ash. Be and Ag concentrations were also much higher in the DJ Unit 3 fly ash as compared with the DJ Units 1 and 2 fly ash (Table 5). Elements such as Pb and Sr were much lower in the DJ Unit 3 fly ash as compared to the DJ Units 1 and 2 fly ash. The reasons for these apparent differences have not been determined. Both were classified by ASTM procedures as marginal Class C fly ash (Table 4). The pozzolanic activity values ($\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$) for DJ Unit 3 fly ash and DJ Units 1 and 2 fly ash are 68.2% and 69.4%, respectively. The minimum value for a Class F fly ash is 70%. Differences in particle size distribution between the DJ Unit 3 fly ash and DJ Units 1 and 2 fly ash also exist. The fly ash collected from DJ Unit 3 contained 38.3% of +325 mesh material while the DJ Units 1 and 2 fly ash contained 29.2% of +325 mesh material. This characteristic undoubtedly would influence reaction kinetics. However, these relationships were not investigated.

Table 4. Chemical Characterization of the Dave Johnston Fly Ash, Units 1 and 2 and Unit 3, and the Laramie River Fly Ash, %

Chemical Characteristics	Dave Johnston Units 1 and 2	Dave Johnston Unit 3	Laramie River
SiO_2	42.7	41.5	31.0
Al_2O_3	22.6	22.2	16.8
Fe_2O_3	4.1	4.5	6.2
SO_3	0.7	0.8	2.7
CaO	16.2		28.4
MgO	3.0	3.3	6.2
Na_2O	0.2	0.2	1.1
K_2O	1.2	1.1	0.3
P_2O_5	0.3	0.4	0.7
MnO	0.0	0.0	0.0
TiO	0.0	1.0	1.1

Table 5. Elemental Composition of the Dave Johnston Fly Ash, Units 1 and 2 and Unit 3, and the Laramie River Fly Ash^a, mg/L

Element	Dave Johnston Units 1 and 2	Dave Johnston Unit 3	Laramie River
Sb	nd ^b	31	nd
As	nd	nd	nd
Ba	0.2	890	nd
Be	nd	4.7	nd
Bi	nd	nd	nd
B	404	410	732
Cd	nd	1.5	nd
Co	24.9	15	34.6
Cr	98.3	75	53
Cu	128	110	175
Hg	0.8	nd	0.3
Pb	103	33	151
Li	35.7	33	28.9
Mo	3.0	6.0	3.2
Ni	42.2	36	48.8
Se	10.3	3.0	23.3
Ag	1.8	5.4	2.0
Sr	1230	820	3870
Th	25.9	22	33.3
V	222	190	163
Zn	123	92	91.6

^a Elements analyzed in addition to those shown in Table 4

^b nd = not detectable using available equipment

The LR fly ash is also classified as Class C by ASTM procedures (Table 4). This was expected because limestone injection is used at the plant to reduce SO_2 from the gaseous emissions. As a result, the LR fly ash has very good cementitious qualities. Removal of extracts from the fly ash was difficult because the material quickly solidified during experimentation. The LR fly ash has a pozzolanic activity value of 54% (Table 4) and a CaO content of 28.4%. The elemental concentrations in the LR fly ash varied somewhat from the levels found in the DJ fly ash (Table 5). For example, in the LR fly ash concentrations of B, Pb, Se, Co, Sr, and Cu were relatively high, and Cr, Li, and V were relatively low. Major differences in composition resulted from differences in coal composition and preparation, combustion conditions, and fly ash handling procedures. Minor differences may be caused by sample preparation and analysis errors.

Analysis of Fly Ash Extracts

The analysis of the extracts collected from the DJ Units 1 and 2 fly ash and the LR fly ash are listed in Tables 6 and 7, respectively. Data show that subsamples collected from the DJ power plant were very similar. The most notable differences were with soluble Al, Ba, K, Sr, F^- , and Cl^- . However, such differences maybe attributed to sample variation, error in sample preparation, and to experimental error. Extracts collected from the DJ fly ash samples did not contain any elements at concentrations that might be considered toxic. However, the extracts did have pH values of about 12.3 and can be considered saline with EC values of about 6.2 dS/cm. Extracts from the LR fly ash samples show considerable differences between sample values. However, this variation may be expected, since the samples cemented in a few minutes, and considerable problems were experienced attempting to dissolve the material for extraction. Sample LR-5 was extracted from a fly ash/water slurry following 3 minutes of continuous mixing prior to cementation of the material. The analysis of these extracts provides some interesting comparisons for the extracts collected from the dissolved material (Table 7). The LR-5 sample had very high concentrations of Al in solution compared with the other samples. In addition, the concentrations of SO_4^{2-} in solution were also relatively high. Based on a geochemical model run using MINTEQA2, Al and SO_4^{2-} are expected to form minerals such as AlOHSO_4 , $\text{Al}_4(\text{OH})_{10}\text{SO}_4$, alinite, and possibly diaspore. With expected decreases in pH due to dilution and to the influence of surrounding materials, a large group of minerals would be expected to form including $\text{Al}(\text{OH})_3$, aragonite, barite, calcite, anacime, and various clay minerals.

Mineralogy

The major crystalline phases identified in of the DJ fly ash samples are listed in Table 8. Mineralogically, the DJ fly ash resembles a Class F fly ash.

Table 6. Analysis of Extracts Collected from the Dave Johnston Units 1 and 2 Fly Ash, Eight Subsamples, mg/L

Elements	1	2	3	4
Al	2.705	2.715	3.01	0.6815
Sb	<0.10	<0.10	<0.10	<0.10
As	<0.10	<0.10	<0.10	<0.10
Ba	38.7	37.7	36.05	13.5
Be	<0.002	<0.002	<0.002	<0.002
Bi	<0.10	<0.10	<0.10	<0.10
B	0.9015	0.925	0.9825	0.0155
Cd	<0.010	<0.010	<0.010	<0.010
Ca	246	251.5	253.5	277
Cr	<0.008	<0.008	<0.008	<0.008
Co	0.0255	0.0275	0.0265	0.012
Cu	<0.006	<0.006	0.008	<0.006
Fe	<0.006	<0.006	0.009	<0.006
Pb	<0.050	<0.050	<0.050	<0.050
Li	0.148	0.125	0.157	0.163
Mg	<0.010	<0.010	<0.010	<0.010
Mn	<0.003	<0.003	<0.003	<0.003
Hg	<0.50	<0.50	<0.40	<0.50
Mo	<0.010	<0.010	<0.010	<0.010
Ni	<0.020	<0.020	<0.020	<0.020
P	<1.0	<1.0	<1.0	<1.0
K	7.535	5.85	9.11	5.85
Se	<0.10	<0.10	<0.10	<0.10
Si	0.232	0.222	0.2395	0.2775
Ag	<0.007	<0.007	<0.007	<0.007
Na	21.6	19.4	23.35	12.95
Sr	52.25	51.65	50.1	31.3
Th	<0.050	<0.050	<0.050	<0.050
V	<0.008	<0.008	<0.008	<0.008
Zn	0.021	0.022	0.0235	0.0195
SO ₄ ²⁻	0.45	0.22	0.22	0.33
F ⁻	2.04	1.70	0.99	1.45
Cl ⁻	0.85	0.85	0.85	1.50
Br ⁻	0.29	0.30	0.28	0.81
NO ₃ ⁻	0.41	0.38	0.34	0.46
pH	12.28	12.30	12.27	12.29
EC	6.02	6.15	6.22	6.11

Table 6. Analysis of Extracts Collected from the Dave Johnston Units 1 and 2 Fly Ash, Eight Subsamples, mg/L (continued)

Elements	5	6	7	8
Al				
Sb	<0.10	<0.10	<0.10	<0.10
As	<0.10	<0.10	<0.10	<0.10
Ba	26.4	14	15.3	26.05
Be	<0.002	<0.002	<0.002	<0.002
Bi	<0.10	<0.10	<0.10	<0.10
B	0.696	0.0915	0.1275	0.4625
Cd	<0.010	<0.010	<0.010	<0.010
Ca	254.5	264.5	256.5	236.5
Cr	<0.008	<0.008	<0.008	<0.008
Co	0.0185	<0.007	0.012	0.018
Cu	<0.006	<0.006	<0.006	0.008
Fe	0.008	<0.006	0.008	0.011
Pb	<0.050	<0.050	<0.050	<0.050
Li	0.172	0.156	0.156	0.15
Mg	<0.010	<0.010	<0.010	<0.010
Mn	<0.003	<0.003	<0.003	<0.003
Hg	<0.50	<0.50	<0.50	<0.50
Mo	<0.010	<0.010	<0.010	<0.010
Ni	<0.020	<0.020	<0.020	<0.029
P	<1.0	<1.0	<1.0	<1.0
K	9.22	8.77	8.55	5.545
Se	<0.10	<0.10	<0.10	<0.10
Si	0.2945	0.322	0.332	0.2925
Ag	<0.007	<0.007	<0.007	<0.007
Na	19.1	13.45	14.35	21.95
Sr	42.2	32.8	33.2	43.8
Th	<0.050	<0.050	<0.050	<0.050
V	<0.008	<0.008	<0.008	<0.008
Zn	0.0175	0.021	0.016	0.015
SO ₄ ²⁻	nd	0.27	0.27	0.26
F ⁻	1.61	2.36	1.48	2.09
Cl ⁻	1.16	1.78	1.89	2.29
Br ⁻	0.56	0.93	0.89	0.73
NO ₃ ⁻	0.36	0.54	0.50	0.56
pH	12.28	12.28	12.31	12.28
EC	5.91	5.93	5.85	5.69

Table 7. Analysis of Extracts Collected from the Laramie River Fly Ash, Five Subsamples, mg/L

Elements	LR-1	LR-2	LR-3	LR-4	LR-5
Al	2.03	5.83	0.325	0.7345	245
Sb	nd	<0.10	<0.10	<0.10	<0.10
As	<0.10	<0.10	<0.10	<0.10	<0.10
Ba	36.7	41.5	82.4	86.3	0.602
Be	<0.002	<0.002	<0.002	<0.002	<0.002
Bi	<0.10	<0.10	<0.10	<0.10	<0.10
B	<0.020	0.165	0.0145	0.285	0.049
Cd	<0.010	<0.010	<0.010	<0.010	<0.010
Ca	173	208	229.5	184	23.2
Cr	<0.008	<0.008	<0.008	<0.008	0.129
Co	0.024	0.028	0.053	0.0585	<0.007
Cu	0.007	<0.006	<0.006	0.007	<0.006
Fe	<0.006	<0.006	<0.006	0.026	<0.006
Pb	<0.050	<0.050	<0.050	<0.050	0.443
Li	0.4825	0.47	0.736	0.056	0.664
Mg	<0.010	<0.010	<0.010	<0.010	<0.010
Mn	<0.003	<0.003	<0.003	<0.003	0.006
Hg	<0.50	<0.50	<0.50	<0.50	<0.50
Mo	<0.010	<0.010	<0.010	<0.010	0.408
Ni	<0.020	<0.020	<0.020	<0.020	<0.020
P	<1.0	<1.0	<1.0	<1.0	<1.0
K	38.85	38.2	56.15	76	96.9
Se	<0.10	<0.10	<0.10	<0.10	0.176
Si	1.001	0.891	0.771	1.0415	0.730
Ag	<0.007	<0.007	<0.007	<0.007	<0.007
Na	265	255.5	374	516.5	676
Sr	66.35	64.6	101.5	111.1	10.8
Th	<0.050	<0.050	<0.050	<0.050	<0.050
V	<0.008	0.009	<0.008	<0.008	0.018
Zn	0.0195	0.0185	0.0225	0.0345	0.023
SO ₄ ⁻²	2.45	0.36	0.30	0.57	310
F ⁻	5.59	5.90	6.22	5.29	9.47
Cl ⁻	3.51	3.85	4.60	5.61	16.30
Br ⁻	0.51	0.47	0.59	0.85	4.00
NO ₃ ⁻	4.69	6.10	7.50	6.79	14.60
pH	12.02	12.12	12.30	12.37	11.92
EC	5.30	5.55	7.39	8.69	5.68

Table 8. Crystalline Phases Identified in the Dave Johnston Units 1 and 2 Fly Ash

Quartz	SiO_2
Mullite	$\text{Al}_6\text{Si}_2\text{O}_{13}$
Periclase	MgO
Spinel	MgAl_2O_4
Lime	CaO
Merwinite	$\text{Ca}_3\text{Mg}(\text{SiO}_4)_2$
Tricalcium aluminate	$\text{Ca}_3\text{Al}_2\text{O}_6$
Brownmillerite	$\text{Ca}_2(\text{Al,Fe})_2\text{O}_5$

The fly ash material appears to be somewhat variable in mineralogical composition. Variations were detected in the DJ fly ash among the eight subsamples and the different size fractions that were evaluated. However, quartz and mullite were detected in almost all of the samples (Table 9). This can be attributed to the method of size separation. Hemmings and Berry (1988) reported that the speciation was dependent on the size and density separation techniques used. Hemmings and Berry (1988) and McCarthy (1988) noted that size fractionation leads to an increase in the amount of amorphous material and quartz detected by X-ray diffraction. The mineral and amorphous phases of a typical X-ray diffractogram of the DJ Units 1 and 2 fly ash are shown in Figure 1. (Figures are located in the Appendix.) These two phases possibly mask the other common fly ash phases. The DJ Unit 3 fly ash was not separated by size, and the only crystalline phases that were positively identified were quartz and mullite (Figure 2).

The LR fly ash is a Class C fly ash (Table 10). A representative X-ray diffractogram of the LR fly ash (Figure 3) exhibits notable differences from the DJ fly ash. The LR fly ash contains minerals such as anhydrite, spinel, lime, and merwinite which are not apparent in the DJ fly ash.

There was less variation among the size fractions of the LR fly ash than found with the DJ fly ash. Five mineral phases (quartz, lime, periclase, merwinite, and anhydrite) were positively identified in most of the samples (Table 11).

Select samples of fly ash were analyzed using the scanning electron microscopy-energy dispersive X-ray analysis (SEM/EDXA). The particle size classifications of all the DJ and LR fly ash samples revealed limited information about the morphology of the fly ash samples. The largest size fraction (+120 mesh) contained particles that were vesicular and irregular in form (Figure 4). Primarily, the particles consisted of partially fused ash and partially combusted carbon. Well-formed individual spheres were not widely detected in this size fraction. A small number of irregular-shaped particles and a larger number of spheres were identified in the +270 fraction. The other two size fractions (+400 and -400) consisted mainly of fly ash spheres.

Table 9. Mineral Phases in the Dave Johnston Fly Ash, Units 1 and 2 and Unit 3, Identified by X-ray Diffraction Analysis

Unit	Sample	Size Fraction (mesh)	Qtz	Mu	Sp	Per	Lime	Mw	C ₃ A	Bm	Hm
1 & 2	1	+120	*	*	✓	✓	✓				
1 & 2	1	+270	*	*		✓					*
1 & 2	1	+400	*	*		*	*				
1 & 2	1	-400	*	*							
1 & 2	2	+120	*	*							
1 & 2	2	+270	*	*							
1 & 2	2	+400	*			✓				✓	
1 & 2	2	-400	*	*		✓	*				*
1 & 2	3	+120	*	*	✓						
1 & 2	3	+270	*	*							
1 & 2	3	+400	*	*	✓						*
1 & 2	3	-400	*	*		✓	*	*	✓		
1 & 2	4	+120	*	*	✓						
1 & 2	4	+270	*	*		✓		✓			✓
1 & 2	4	+400	*	*	✓		*		✓		
1 & 2	4	-400	*	*		✓	*	✓			
1 & 2	5	+120	*	*							
1 & 2	5	+270	*	*		✓					
1 & 2	5	+400	*	*			✓	✓			
1 & 2	5	-400	*	*		*	*				
1 & 2	6	+120	*	*					✓		
1 & 2	6	+270	*	*							
1 & 2	6	+400	*	*		✓	✓				
1 & 2	6	-400	*	*			✓	✓			
1 & 2	7	+120	*	*							
1 & 2	7	+270	*	*							
1 & 2	7	+400	*	*						✓	
1 & 2	7	-400	*	*		✓	✓	✓			✓
1 & 2	8	+120	*	*							
1 & 2	8	+270	*	*	✓						
1 & 2	8	+400	*	*			*	✓			
1 & 2	8	-400	*	*		✓	*	✓	✓		
3			*	*							

* = major phase

✓ = minor phase

Qtz = quartz, Mu = mullite, Sp = spinel, Per = periclase, Mw = merwinite, C₃A = tricalcium aluminate, Bm = brownmillerite, Hm = hematite

Table 10. Crystalline Phases Identified in the Laramie River Fly Ash

Quartz	SiO_2
Periclase	MgO
Lime	CaO
Merwinite	$\text{Ca}_3\text{Mg}(\text{SiO}_4)_2$
Melilite	$(\text{Ca}, \text{Na})_2(\text{Mg}, \text{Al}, \text{Fe})(\text{Si}, \text{Al})_2\text{O}_7$
Mullite	$\text{Al}_6\text{Si}_2\text{O}_{13}$
Brownmillerite	$\text{Ca}_2(\text{Al}, \text{Fe})_2\text{O}_5$
Anhydrite	CaSO_4
Hematite	Fe_2O_3
Spinel	MgAl_2O_4

Table 11. Mineral Phases in the Laramie River Fly Ash Identified by X-ray Diffraction Analysis

Sample	Size Fraction (mesh)	Qtz	Per	Lime	Mw	Ml	Mu	Bm	An	Hm	Sp
1	+120	*		*	✓	✓	*		*	*	✓
1	+270	*	✓	✓	*				*		✓
1	+400	*	*	*	*		*		*	*	
2	+270	*	*	*	*				✓	✓	
2	+400	*	*	✓	*			*	✓		
2	-400	*	*		*			*	*		
3	+120	*	✓	✓	✓	✓	✓		*		
3	+270	*	*	✓	*				✓		
3	+400	*	*	✓	✓				✓		
3	-400	*	*	✓	*						
4	+120	*	✓	*	✓				*		
4	+270	*	*	✓	✓			✓	✓		
4	+400	*	*	✓	*				✓		
4	-400	*	*	✓	*	✓			✓		

* = major phase

✓ = minor phase

Qtz = quartz, Per = periclase, Mw = merwinite, Ml = melilite, Mu = mullite, Bm = brownmillerite, An = anhydrite, Hm = hematite, Sp = spinel

The major elements detected in all samples by SEM/EDXA were Si, Ca, and Al. The DJ fly ash was Si rich, with Ca and Al following in relative abundance. Minor elements identified in these samples included K, Fe, S, and Ti. The LR fly ash was predominantly Ca rich. However, the remaining major (Si, Al, S, Fe) and minor (K, Ti, Ba, Mg) elements present are nearly identical to those in DJ fly ash.

Two samples of the LR fly ash were hydrated and analyzed on the X-ray diffractometer. The only crystalline phases that were positively identified were quartz and feldspar. Orthoclase $(K,Ba,Na)(Si,Al)_4O_8$ and anorthite $(CaAl_2Si_2O_8)$ are most likely candidates for the feldspar phase. SEM analysis shows that these samples are physically different than the unhydrated ash. The photomicrographs show fly ash spheres, detrital quartz, and a platy mineral phase that is covered by thin rod-like fibers (Figure 5). The rod-shaped minerals appear in clusters (Figure 6) and originate from the fly ash spheres (Figure 7). EDXA analysis of the 7-day hydrated LR fly ash showed little elemental variation from the size-separated ash. The elements detected in the samples included Si, Ca, Al, Fe, and S with minor levels of K, Mg, and Ti. Energy dispersive X-ray analysis of the fly ash spheres, platy minerals, and rod-shaped minerals showed no discernible differences in elemental content.

Samples of DJ Units 1 and 2 fly ash were mixed with RS-1 (sand) and RS-3 (loamy sand) soils; samples of DJ Unit 3 fly ash were mixed with RS-4 (loamy sand) soil; and the mixtures were compressed into individual columns. These samples were neither size nor density separated. Representative samples were analyzed using XRD. All of the specimens contained quartz and feldspar; however the specific feldspar phase was difficult to identify. During sample preparation, pink potassium feldspar grains were noticeable. In the RS-1/fly ash samples (Table 12), quartz and feldspar were found in all the samples. Clinoferrrosilite ($FeSiO_3$) was detected in most of the samples and spinel was established in two samples. Calcite ($CaCO_3$) and ettringite ($Ca_6Al_2(SO_4, SiO_4, CO_3)_3(OH)_{12} \cdot 26H_2O$) were identified in a sample that contained 5% lime and 25% fly ash (Figure 8).

Hedenbergite ($Ca(Fe,Mg)(SiO_3)_2$) was identified in a few of the RS-3/fly ash samples and clinoferrrosilite was detected in one sample (Table 13). The RS-4/fly ash samples were very similar with various amounts of clinoferrrosilite, hedenbergite, and clay material identified (Table 14).

A selection of the samples containing DJ Units 1 and 2 fly ash and the RS-1 (sand) soil were analyzed using SEM/EDXA. The 10% fly ash mixture with 13.9% water contained a few poorly developed rod-like minerals (Figure 9). EDXA showed that the sample contained Ca, Si, and Al, with minor amounts of Fe, S, Mg, K, and Na. The 15% fly ash mixture had a greater abundance of the rod-like minerals (Figure 10). EDXA of the 15% fly ash mixture with 6.9% water showed that Ca was the most abundant element, followed by Si and Al. Minor phases included K, Mg, S, and Fe. The RS-1 (sand)/fly ash sample with a 20% fly ash mixture and a water content of 11.6% contained mineral formations that were

Table 12. Mineral Phases Identified in the Samples Containing RS-1
(Sand) Soil and Dave Johnston Units 1 and 2 Fly Ash

	Quartz	Feldspar	Cfs ^a	Spinel	Ettringite	Calcite
5% Fly Ash						
W ^b = 6.7%	*	*				
W = 8.2%	*	*	*			
W = 9.4%	*	*	*			
W = 11.9%	*	*	*			
10% Fly Ash						
W = 7.5%	*	*				
W = 9.4%	*	*				
W = 11.1%	*	*	*	*		
W = 13.9%	*	*	*			
15% Fly Ash						
W = 6.9%	*	*	*			
W = 9.1%	*	*				
W = 10.8%	*	*	*			
W = 12.4%	*	*	*			
20% Fly Ash						
W = 6.3%	*	*	*			
W = 7.9%	*	*				
W = 10.0%	*	*	*			
W = 11.6%	*	*	*			
25% Fly Ash						
W = 6.3%	*	*	*			
W = 7.9%	*	*				
W = 9.5%	*	*				
W = 11.8%	*	*	*			
5% Lime and 25% Fly Ash						
W = 7.2%	*	*	*		*	*
W = 8.9%	*	*				
W = 11.3%	*	*	*		*	
W = 12.6%	*	*	*			*

^a Cfs = clinoferrosilite

^b W = water content

**Table 13. Mineral Phases Identified in the Columns Containing RS-3
(Loamy Sand) Soil and Dave Johnston Units 1 and 2 Fly Ash**

	Quartz	Feldspar	Hedenbergite	Clinoferrosilite
5% Fly Ash				
W = 8.0%	*	*	*	
W = 9.6%	*	*		
W = 12.0%	*	*	*	
W = 13.8%	*	*		
10% Fly Ash				
W = 8.2%	*	*	*	
W = 10.2%	*	*		
W = 12.5%	*	*		
W = 14.0%	*	*		
15% Fly Ash				
W = 8.3%	*	*		
W = 10.3%	*	*		
W = 12.7%	*	*		
W = 14.4%	*	*		
20% Fly Ash				
W = 8.4%	*	*	*	
W = 10.4%	*	*	*	*
W = 12.3%	*	*		
W = 14.1%	*	*		
25% Fly Ash				
W = 8.1%	*	*		
W = 10.1%	*	*		
W = 12.6%	*	*		
W = 14.0%	*	*	*	

Table 14. Mineral Phases Identified in the Columns Containing RS-4 (Loamy Sand) Soil and Dave Johnston Unit 3 Fly Ash

	Quartz	Feldspar	Cfs ^a	Spinel	Hedenbergite	Clay
20% Fly Ash						
W ^b = 10.0%	*	*		*		
W = 11.6%	*	*			*	*
W = 13.7%	*	*				
W = 15.2%	*	*				
25% Fly Ash						
W = 9.9%	*	*				
W = 11.7%	*	*				
W = 13.5%	*	*	*			
W = 15.5%	*	*				

^a Cfs = clinoferrosilite

^b W = water content

slightly different than the other samples (Figure 11). In addition to rod-like minerals, this sample contained small, platy, clay-like minerals adhering to the fly ash surfaces. The only elemental difference detected by EDXA was the lack of Mg in this sample. The 25% fly ash and 6.3% water content mixture shown in Figure 12 contains only the rod-like minerals, with no platy minerals. The calcite rhombohedron clusters identified in the RS-1 (sand)/fly ash sample with 5% lime and 25% fly ash are illustrated in Figure 13; the rod-like minerals were also present. The photomicrograph in Figure 14 shows there may be a relationship between the formation or presence of rods and the disintegration of the fly ash spheres. Calcium is the major phase identified in the EDXA of the rhombohedron-shaped minerals (Figure 15). EDXA of the rod-like phase in the lime/fly ash sample identified the same elements that were detected in the other RS-1 (sand)/fly ash samples (Figure 16). One RS-3 (loamy sand)/fly ash sample containing 15% fly ash with 12.7% water looks physically similar to the RS-1 (sand)/fly ash samples (Figure 17). EDXA detected the elements: Si, Al, Ca, S, and minor amounts of Mg.

XRD Analysis of Density-Separated Samples

Density separation was performed on two samples taken from columns 2 and 4. Column 2 was cured in a reducing environment, and column 4 was cured in an oxidizing environment.

It was evident that the density separation experiment was not entirely successful because quartz appeared in almost all density fractions (Table 15). XRD analysis was used to determine whether or not ettringite and other minerals were present. Ettringite was identified in sample 4 but not in sample 2. Sample 2 was allowed to cure in a reducing environment, which may have hindered the development of ettringite.

Table 15. Analysis of Density Separated Laramie River Soil/Fly Ash Mixtures

	Sample 2	Sample 4
<2.51 g/cm ³ :	Quartz, Feldspar,	Quartz, Feldspar, Ettringite
2.51-2.85 g/cm ³ :	Quartz, Feldspar	Quartz, Feldspar
>2.85 g/cm ³ :	No positive identification, possibly Quartz	Quartz, Hematite

Samples removed from the columns were also analyzed with the SEM/EDXA. Fly ash spheres were associated with rosettes of plate-like minerals (Figure 18) that were similar in form to the photomicrographs of chlorite ((Mg,Al,Fe)₁₂[(Si,Al)₈O₂₀](OH)₁₆) described by Welton (1984, Figure 19). All of the samples contained Si, Ca, Al, Mg, K, S, and Fe. Calcium was the major element in the rod-like minerals.

SECTION II.

UNCONFINED COMPRESSION STRENGTH TESTS OF SOIL/FLY ASH MIXTURES

Introduction

A considerable amount of research has been done investigating the use of fly ash for various applications, including soil stabilization, cement substitution, and waste stabilization. This work is published in hundreds of articles throughout the literature. A relatively small part of this research has been dedicated to the use of fly ash to stabilize the surfaces of unpaved (dirt) roads to prevent break-up and the development of washboards. Much of the work done using this technology has been done by Scholen and Coghlan (1989) of the U.S. Forest Service. The method they proposed consists of using low amounts of fly ash with the addition of appropriate amounts of aggregate to stabilize logging and recreation roads located on National Forest lands.

This fly ash stabilization technique resulted in substantial reduction in road maintenance costs (Scholen and Coghlan 1989). The primary mechanisms of strength development that are important to unpaved road stabilization using fly ash have not been investigated. For example, it may be more important for the fly ash stabilized material to have low strength and good ductility rather than to have high strength with a brittle character. Highly brittle road materials may be expected to snap under stress, resulting in a fractured surface that will readily degrade. The more ductile material is expected to bend without developing a fractured system that would subsequently break-up. This investigation should provide a broad indication of how unconfined compression strength (UCS) and Young's modulus (degree of brittleness) change with fly ash content, fly ash type, and soil type.

Materials and Methods

Unconfined compression strengths (UCS) of soil/fly ash mixtures were tested with various combinations of soil, fly ash, and water. Moisture content and moisture-density relationships were performed initially to determine the maximum dry density and the optimum moisture content of the soil/fly ash mixtures. A range of soil texture classes were used to provide information that could be interpreted and correlated to a broad number of road sites. The soils used were a loamy sand (sample RS-3), a sandy clay loam (sample HR-S1L) and a clay loam (sample LL-2). Soil samples were sifted with a #10 screen (2.00 mm openings) to remove the large material that can bias the results because of particle size/column size relationships. The fly ash materials used in this work represent a Class C material that has very good cementation properties (Laramie River power plant fly ash) and a marginal Class C material that has slow cementing character (Dave Johnston power plant fly ash). The fly ash materials used were from the Dave Johnston power plant Units 1 and 2 (DJ-4) and from the Laramie River power plant (LR-4). Specimens were prepared with 0, 5, 10, and 20% fly ash as a percentage of the dry weight of the soil. Specimens were prepared at optimum moisture content with a select number of samples prepared at field moisture for comparative purposes. The development of strength, determined by UCS strength analysis, was tested for samples cured for 7 and 28 days.

Moisture Content

Moisture content was measured using the Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures (ASTM D 2216-90). Moisture content tins were tared; 50 g of material was added to each tin; and the gross weight was recorded. The tins containing the material were then placed in an oven set at 105°C ($\pm 5^\circ\text{C}$) for 24 hours. The tins were reweighed after an additional 24 hours to ensure that dehydration was complete. The reported moisture content is a percentage of the weight of remaining dry material. All dry weights are based on total weight divided by 1 plus moisture content.

Moisture-Density Relationships

Moisture-density relationships were determined at 0, 5, 10, and 20% fly ash as a percentage of the dry weight of the soil for each soil/fly ash combination. A modified version of the Standard Test Method for Moisture-Density Relations of Soils and Soil-Aggregate Mixtures (ASTM D 698-78) was used to determine the optimum moisture content and maximum dry density of the soil/fly ash mixtures. The modification consists of using the Harvard Miniature system in lieu of the standard column size specified in ASTM D 698-78. Three specimens were prepared for each soil/fly ash mixture with moisture contents varying by about 2%. The soil/fly ash/water mixture was added to the Harvard Miniature Compaction Apparatus (constructed by Soiltest) in three lifts. Each lift was compacted by 20 compressions of the spring supplied with the Harvard device. The spring has an area of 0.200 sq in. on its compactive surface and was calibrated to 20 lb. The specimen was weighed immediately after preparation. Moisture content was determined as described above. Optimum moisture content and maximum dry density were determined following the calculations described in ASTM D 698-78.

Unconfined Compression Strength

Unconfined compression strength (UCS) was determined by a modified version of ASTM D 2166-85, Standard Method for Unconfined Compression Strength of Cohesive Soil.

Specimens were prepared by first weighing out the amount of soil and fly ash necessary for the preparation of ten specimens. The mixture was placed in a Ziploc bag. Tap water was added to bring the total moisture content up to the optimum moisture content (except for those specimens prepared at field moisture). The soil/fly ash/water mixtures were thoroughly mixed in the Ziploc bags. Special care was taken to prevent caking and sticking. The mixture was allowed to sit for 5 to 10 minutes before preparation of the first specimen. Samples were collected for moisture content determination during the initial curing period.

Individual specimens were prepared by placing the mixture in the Harvard miniature device in three lifts. Each lift was compacted with 20 blows by a spring compactor provided with the Harvard device. Each specimen was wrapped in cellophane freezer-type wrap. Moisture content was measured in the remaining soil/fly ash/water mixtures after the ten

specimens had been prepared. Moisture content of the specimens at preparation was calculated by interpolation between the initial and final moisture contents of the mixture, determined by this method. The specimens were then placed in a curing chamber at 100% relative humidity and about 20°C for the prescribed period of time.

Unconfined compression strength (UCS) tests were performed on duplicate specimens after 7 and 28 days of curing. UCS was determined with a Soiltest G-900 Versa-Loader equipped with a U-903M Low-Load Accessory Set. Axial stress was applied at a rate of 1% of the height of the specimen per minute. The moment of failure was determined by noting when the deformation of the proving ring reached a maximum. The stress of the specimen at failure, the time to failure, and the deformation of the proving ring at failure were recorded. UCS was determined using the calibration of the proving ring and the surface area of the specimen. Young's Modulus (E), which provides an indication of the brittleness of the material, was also determined. The higher the E value, the more brittle the material. The E value was calculated using the proportional change in the axial dimension upon compression. The UCS divided by the proportional change in the axial dimension is equal to E.

Results and Discussion

Strength of Materials

In general, the strength of the specimens increased with the amount of fly ash present in the soil/fly ash mixtures (Figure 20). The one exception where strength did not increase in response to an increase in fly ash percentage was with the DJ fly ash that had been cured for 7 days. However, the DJ fly ash specimens cured for 28 days were consistently stronger with an increased fly ash percentage. These data show that the DJ fly ash is slower to react, but given time, the increases in UCS are substantial. These data also show that longer curing times result in higher strengths. At the 5% fly ash concentration, there was little difference in the UCS values; they ranged from 30 psi to about 37 psi for the DJ fly ash and the LR fly ash, respectively. These values are comparable to the specimens that did not contain fly ash. Curing times of 28 days did not appear to significantly change UCS values at the 5% fly ash level. However, greater differences existed for the specimens containing 10% fly ash. The material that contained 10% LR fly ash cured for 28 days, had a UCS of about 61 psi, whereas the 20% fly ash mixture cured for 28 days had a UCS of 63 psi. Therefore, an increase in the amount of LR fly ash by a factor of two had a minimal influence on the strength characteristic of the soil/fly ash mixture.

The LR fly ash cured for 7 days showed a relatively constant increase in UCS with increased fly ash used in the mix. This material reached strengths of 45 psi for the 10% fly ash addition and strengths of about 56 psi for the 20% fly ash level. The soil/DJ fly ash material that was cured for 28 days demonstrated similar strength development as the soil/LR fly ash mixtures cured for 7 days. In fact, the soil/DJ fly ash materials cured for 28 days reached strengths of about 53 psi at 20%

fly ash, which is comparable to the strength of specimens made of LR fly ash cured for 7 days. The DJ fly ash specimens that were cured for 7 days did not show any strength gain with zero to 20% fly ash additions.

It is obvious that the soil/DJ fly ash mixture gains strength slowly compared with the soil/LR fly ash mixtures. However, following a 28-day curing period the specimens with 20% DJ fly ash have comparable strength to the LR fly ash specimens. Also, samples with no fly ash that were cured for 28 days have lower UCS values than specimens cured for 7 days. These data show that the curing process used may reduce the strength of compacted soils with time.

The relationships between the quantities of fly ash used, the UCS of the LR and DJ fly ash specimens, and the sandy clay loam soil are shown in Figure 21. Specimens made using LR fly ash with the 7-day and the 28-day curing periods show increasing strength development with curing time (Figure 22). The DJ fly ash also demonstrates increasing UCS with curing time for the 10 and 20% addition levels. However, the 5% addition of DJ fly ash does not show an increase in UCS with increased curing time. These data show that the DJ fly ash reacts slowly compared with the LR fly ash.

The addition of the fly ash to a clay loam soil demonstrated slightly different strength relationships as compared to the sandy clay loam soil/fly ash mixture (Figure 23). The specimens that contained LR fly ash cured for 7 days gained strength with increasing fly ash content, whereas the clay loam/DJ fly ash mixtures cured for 7 days did not gain strength with increased fly ash. As shown with the sandy clay loam soils, the LR fly ash cured for 28 days reached maximum UCS with 10% fly ash addition. However, the specimens containing 20% fly ash that were cured for 7 days reached a higher UCS with the clay loam soil than with the sandy clay loam soil. With the clay loam soil material the LR fly ash specimens cured for 7 and 28 days had very similar strengths of 61 psi and 59 psi, respectively. These data provide an indication that the clay loam soil tends to cement and gain UCS to a greater extent than the sandy clay loam soil. The relationships between cure time and axial strength for the clay soil/fly ash mixtures are illustrated in Figure 24. The DJ fly ash consistently gains strength with increasing cure time for each fly ash concentration. Also, as the amount of DJ fly ash increases the strength increases. The LR fly ash specimens, however, tended to gain strength fast, commonly within the first 7 days for the 5 and 20% fly ash concentrations. The specimens with 10% fly ash level continued to gain strength at 28 days as measured in the sandy clay loam soils.

Young's Modulus (E)

The brittleness of a material stabilized with fly ash is an important consideration for determining proper use. The best stabilizing agent for road materials is one that develops strength that bends and deforms rather than snaps in a sudden loading. The data shown in Figure 25 describes the character of specimens made with clay loam soils and fly ash materials that result in relatively high and low E values. The clay loam soil stabilized with LR fly ash that was cured

for 7 and 28 days had relatively low E values of about 1300 psi with 5% fly ash additions. However, larger LR fly ash additions increased the E values. In samples with 10% fly ash addition, E values increased to about 3500 psi for the 7-day cure time and to 5200 psi for the 28-day cure time. In samples with 20% fly ash additions, E values increased to about 7600 psi and 9000 psi for the 7-day and 28-day cure times, respectively. The specimens made with clay loam soils and DJ fly ash proved to be a much more ductile material that had much lower E values. The specimens containing DJ fly ash cured for 7 days showed no increase in E from no fly ash to 10% fly ash additions. In samples with 20% fly ash addition, E increased from 700 psi to about 1800 psi. The specimens that were cured for 28 days show a similar relationship. There are no apparent increases in E with 10% fly ash addition and an increase from about 800 psi to 3700 psi at the 20% fly ash level. As shown in Figure 25, the DJ fly ash specimens have much lower E values than the LR fly ash specimens. Therefore, the materials stabilized with the DJ fly ash have much greater ductility, and in some instances, specimens have similar UCS (Figures 21 and 23).

In the clay loam soil/LR fly ash specimens, brittleness increased with increased cure time (Figure 26). The E values of these specimens changed at a constant rate with increased fly ash addition. The clay loam soil/DJ fly ash specimens did not show any apparent increase in E with cure time.

The E values for the LR and DJ fly ash specimens in combination with clay loam textured soils were somewhat different from E values calculated for the sandy clay loam soils (Figure 27). The LR fly ash specimens cured for 28 days resulted in increased E values with each addition of fly ash. The specimens with no fly ash addition had an E value of about 300 psi while the 5, 10, and 20% fly ash specimens had E values of 2400, 5000, and 7500 psi, respectively. These values are similar to those determined for the sandy clay loam soil/LR fly ash, except that the 20% fly ash resulted in an E value of 7500 psi compared to about 9000 psi for the sandy clay loam soils. The 5% LR fly ash specimens cured for 7 days had an increase in E values over the baseline from about 300 to 2200 psi. Additions of fly ash to 20% only increased E to about 2800 psi. These data show that the LR fly ash tends to develop a more brittle material with a sandy clay loam soil as compared with a clay loam soil. The specimens made with 5% DJ fly ash and clay loam soil and cured for 7 days had E values of about 1000 psi. This increased to 1600 psi with 10% fly ash addition and to 1800 psi with 20% fly ash addition. These values are very similar to those determined for the sandy clay loam soil/DJ fly ash, except that with 10% fly ash the specimens were more brittle with the clay loam soil than the sandy clay loam soil. The DJ fly ash materials cured for 28 days appeared to have an anomaly in the data. The E values changed from 2200 psi at 5% fly ash addition to 4500 psi at 10% fly ash addition and then decreased to 2800 psi at 20% fly ash addition. This anomaly is not understood.

The relationships between cure time and E for both the LR and DJ fly ash specimens are illustrated in Figure 28. In general, the specimens containing LR fly ash showed an increase in E with increased cure time, while the DJ specimens showed very little increase in E with cure time.

X-ray Diffraction Analysis

Soil/fly ash mixtures were evaluated to determine if the mineral content of the reacting material changed with curing time. Quartz and feldspar were identified in all of the samples containing DJ fly ash and the loamy sand soil. One sample containing 20% fly ash at optimum moisture with 7-day cure time may contain hedenbergite. The quartz and feldspar peaks obviously overwhelmed the system eliminating the detectability of other minerals. Quartz and feldspar were also the only mineral phases in the loamy sand soil/LR fly ash specimens that were positively identified (Table 16). Differences in mineralogy were not found between fly ash types, concentrations, moisture contents, or curing times.

Table 16. Mineral Phases Identified in Loamy Sand Soil/Fly Ash Mixtures

Fly Ash	Fly Ash Concentration by weight	Curing Time	Water Content	Mineralogy
Dave Johnston	5%	28 days	optimum	Quartz, Feldspar
Dave Johnston	5%	28 days	optimum	Quartz, Feldspar
Dave Johnston	5%	28 days	field	Quartz, Feldspar
Dave Johnston	10%	7 days	optimum	Quartz, Feldspar
Dave Johnston	10%	7 days	optimum	Quartz, Feldspar
Dave Johnston	20%	7 days	optimum	Quartz, Feldspar, Hedenbergite
Dave Johnston	20%	7 days	optimum	Quartz, Feldspar
Dave Johnston	20%	28 days	optimum	Quartz, Feldspar
Dave Johnston	20%	28 days	optimum	Quartz, Feldspar
Laramie River	5%	7 days	optimum	Quartz, Feldspar

The sandy clay loam soil was more mineralogically reactive than the loamy sand soil (Table 17). Quartz and feldspar were identified in all 13 of the samples containing sandy clay loam soil. Calcite was detected in most of these samples. Other phases identified include: hedenbergite, ettringite, and tricalcium aluminate. Ettringite and tricalcium aluminate are minerals that have been found to enhance the strength of soil materials. However, these minerals were only detected in the 20% LR fly ash mixtures with optimum moisture content cured for 7 days.

Table 17. Mineral Phases Identified in Sandy Clay Loam Soil/Fly Ash Mixtures

Fly Ash	Fly Ash Concentration by weight	Curing Time	Water Content	Mineralogy
Dave Johnston	5%	28 days	optimum	Quartz, Feldspar, Calcite
Dave Johnston	5%	28 days	optimum	Quartz, Feldspar, Calcite
Dave Johnston	20%	7 days	optimum	Quartz, Feldspar, Hedenbergite
Dave Johnston	20%	7 days	optimum	Quartz, Feldspar, Hedenbergite
Dave Johnston	20%	28 days	optimum	Quartz, Feldspar, Calcite, Muscovite
Dave Johnston	20%	28 days	optimum	Quartz, Feldspar, Calcite
Dave Johnston	20%	28 days	field	Quartz, Feldspar, Calcite
Laramie River	5%	28 days	optimum	Quartz, Feldspar, Calcite
Laramie River	5%	28 days	optimum	Quartz, Feldspar, Calcite, Hedenbergite
Laramie River	20%	7 days	optimum	Quartz, Feldspar, Ettringite, Calcite
Laramie River	20%	7 days	optimum	Quartz, Feldspar, Ettringite, Calcite, Tricalcium Aluminate
Laramie River	20%	28 days	optimum	Quartz, Feldspar, Calcite, Illite
Laramie River	20%	28 days	optimum	Quartz, Feldspar, Calcite, Illite

Calcite and hedenbergite may also be important constituents that influence the development of strength. However, the presence of hedenbergite cannot be correlated to specific curing conditions. The specimen made with 5% DJ fly ash at optimum moisture that was cured for 28 days (Figure 29) lacked large- and medium-size fly ash spheres found in other samples. Si, Ca, and Al were the only elements detected in this sample. The remaining samples contain mineral forms more consistent with the loamy sand columns (Figure 30). The elements identified in two other samples at optimum moisture (one with 20% LR fly ash and one with 5% LR fly ash) include: Si, Al, Ca, Mg, S, K, and Fe.

A mixture of 20% DJ fly ash and clay loam soil was cured for 28 days under field moisture conditions. SEM photomicrographs of this sample show that less reaction occurred between the fly ash and soil (Figure 31). Although the rod-like minerals that are evident in many of the other samples are lacking, an apparently similar phase was found (Figure 32). Elemental analysis was identical to the samples compacted at optimum moisture.

Two samples of DJ fly ash and five samples of LR fly ash were mixed with a clay loam soil at optimum moisture and analyzed with XRD (Table 18). Quartz and feldspar were identified in the five clay loam soil/LR fly ash mixtures. Quartz and illite were the only phases identified in the two clay loam soil/DJ fly ash mixtures. The specimens made from clay loam soil and 20% DJ fly ash at optimum moisture and cured for 7 days did not contain the rod-like or rosette plate-like minerals seen in the loamy sand samples (Figure 33). However, an amorphous bridging material was found between two fly ash spheres (Figure 34). Elemental analysis of the column detected Si, Ca, Al, Fe, K, S, and Mg. EDXA of the bonding material showed the same elemental composition as the rest of the sample. No elemental differences were detected in the amorphous bridging material.

Table 18. Mineral Phases Identified in Clay Loam Soil/Fly Ash Mixtures

Fly Ash	Fly Ash Concentration by weight	Curing Time	Water Content	Mineralogy
Dave Johnston	20%	7 days	optimum	Quartz, Illite
Dave Johnston	20%	7 days	optimum	Quartz
Laramie River	5%	7 days	optimum	Quartz, Feldspar
Laramie River	5%	7 days	optimum	Quartz, Feldspar
Laramie River	10%	7 days	optimum	Quartz, Feldspar
Laramie River	20%	7 days	optimum	Quartz, Feldspar
Laramie River	20%	7 days	optimum	Quartz, Feldspar

Conclusions

Despite the variability in the collected data (standard deviation = 14% for the UCS data and 26% for the E values), some important conclusions can be made. In general, the LR fly ash resulted in specimens that had higher UCS and more brittleness than the specimens made with DJ fly ash. The soil/DJ fly ash mixtures that were cured for 28 days reached strengths that approached the UCS values of the LR fly ash specimens. However, the DJ fly ash specimens did not develop the degree of brittleness that the LR specimens developed. These characteristics may be an important attributes for the DJ fly ash in road stabilization applications. The DJ fly ash provides good strength and also is relatively ductile. This characteristic may enhance its lasting time and reduce the occurrences of road surface breakup and washboarding.

SECTION III. SELECTIVE DISSOLUTION OF SOIL/FLY ASH MIXTURES

Introduction

Elemental speciation in solid matrices determines the physical and chemical properties of the solids including the availability of elements for potential contamination of the environment (Lake et al. 1984). Elemental speciation in solid phases can be characterized by applying direct or indirect methods of analysis or by applying a combination of both. Direct methods allow direct observations of elemental associations with techniques such as X-ray diffraction (XRD) and scanning electron microscopy (SEM). Indirect methods of determining elemental speciation use a specific chemical reagent to selectively extract an element from a chemically distinct mineral pool. These mineral pools include water soluble, exchangeable, adsorbed, organic, carbonate, silicate, etc. Sequential selective dissolution procedures selectively extract elemental forms by using a series of reagents of increasing chemical reactivity applied in a sequential fashion.

Several sequential selective dissolution techniques have been used to provide information on elemental solid-phase residencies (McLaren and Crawford 1973; Stover et al. 1976; Silvera and Sommers 1977; Tessier et al. 1979; Lund et al. 1980; Soon and Bates 1982; Sposito et al. 1982). Such techniques are not exact but can result in useful qualitative estimates of the chemical forms of elements in a solid matrix (Emmerich et al. 1982). The elements extracted by specific reagents may or may not be representative of the designated chemical forms, but can be classified as chemically similar and extractable by the specific reagent.

The purpose of this task was to apply a sequential selective dissolution method to soil/fly ash mixtures to provide information on elemental speciation in the mixtures. This information was correlated with data from XRD analysis of the samples and the solids remaining after each selective dissolution step to provide information on the cementing properties of the soil/fly ash mixtures and possible elemental fate and behavior when the mixtures are exposed to the environment.

Sequential Selective Dissolution Method

The sequential selective dissolution method described by Sposito et al. (1982) was used in this research. This procedure was developed by Stover et al. (1976) and modified by Lund et al. (1980) to partition trace elements into exchangeable, adsorbed, organic, carbonate, and sulfide forms.

In a selective dissolution procedure, the reagents that are used can not be entirely selective, and as a result, there can be overlap of the chemical forms that are extracted (Emmerich et al. 1982). In selecting a selective dissolution technique, emphasis must be placed on the selectivity of each extractant for a chemical form. The reagent used must not only effectively solubilize a specific form of an element, but it must also be selective for that particular form. Several factors can influence the rate and efficiency of extracting a particular fraction.

These include sample type, particle size, extraction time and temperature, solid-to-liquid ratio, and the reagents used (Sterrit and Lester 1980; Forstner et al. 1981).

In the selective dissolution procedure developed by Stover et al. (1976), the exchangeable elemental fraction was first removed by extraction with potassium nitrate. This is based on the assumption that the elements in this fraction can be displaced by saturating the exchange complex with potassium. Next, adsorbed elements were removed through the formation of soluble metal-fluoride complexes formed by treating the sample with potassium fluoride buffered to a pH of 6.5. Sodium pyrophosphate was then used to extract elements associated with organic matter. Ethylenediaminetetraacetic acid (EDTA) was used next to extract metal carbonates, and sulfides were extracted using nitric acid. Reagents for removal of metal carbonates and sulfides were developed using mixtures of pure metal carbonates and sulfides. Stover et al. (1976) found EDTA to extract metal carbonates without removing significant quantities of metal sulfides.

Lund et al. (1980) modified the sequential selective dissolution procedure of Stover et al. (1976). They believed that deionized water would be more effective than potassium fluoride in removing the adsorbed fraction. This was based on a review of stability constants, which shows that soluble metal-hydroxide complexes are more stable than metal-fluoride complexes. In addition, previous studies have shown metal adsorption to be a function of ionic strength. The higher the concentration of salts in a solution surrounding a solid, the greater the concentration of adsorbed metals. The more dilute an extracting solution, the greater the recovery of adsorbed metals. Lund et al. (1980) evaluated the effectiveness of deionized water, in comparison with potassium fluoride, for extracting trace elements adsorbed to iron and aluminum hydrous oxides. This evaluation showed that deionized water to be much more effective in removing adsorbed trace elements.

Lund et al. (1980) further modified the selective dissolution procedure of Stover et al. (1976) by using sodium hydroxide to extract elements from the organic fraction. This was based on the standard procedure for extracting humic and fulvic acid fractions from soils and sediments. Lund et al. (1980) report that no appreciable quantities of metal carbonates or sulfides were extracted by sodium hydroxide. Generally, smaller amounts of metals were extracted from carbonates and sulfides by sodium hydroxide than were removed by the sodium pyrophosphate solution used to release organic-bound metals in the procedure used by Stover et al. (1976).

Lund et al. (1980) also found the use of a single 0.05 M Na_2EDTA treatment to be more specific to carbonate-bound metals than double extraction using 0.1M Na_2EDTA , which is used in the Stover procedure. Despite the lower concentration of EDTA, essentially, a total recovery of the metals was achieved during extraction of pure metal carbonates. Only very small amounts of metals were extracted from pure metal sulfides using this reagent.

Finally, for extraction of metal sulfides, Lund et al. (1980) increased the strength of the nitric acid solution to 4 N at 80°C. When tested on pure metal sulfides, this reagent gave complete recovery.

Based on the selectivity of the reagents described above for extracting their specific elemental fractions, the selective dissolution method described by Sposito et al. (1982) was selected for use in this study. Sposito et al. (1982) improved the procedure used by Lund et al. (1980) by adding steps to correct the elemental data for the amount of metal in the solution entrained in the solid sample and carried over to the next extraction step. This method was applicable to the fly ash samples, because carbonate and sulfide minerals in the samples may contain a significant portion of the elemental distribution.

Methods

The selective dissolution procedure was applied to duplicate 2-g samples of the loamy sand soils (RS-3 and RS-4) and the soil/DJ fly ash mixtures identified as: sample 1 (RS-3 + 25% DJ Units 1 and 2 fly ash + 12.6% H₂O), sample 2 (RS-4 + 20% DJ Unit 3 fly ash + 13.7% H₂O), sample 3 (RS-3 + 15% DJ Units 1 and 2 fly ash) + 12.7% H₂O), sample 4 (RS-3 + 15% DJ Units 1 and 2 fly ash; at optimum H₂O and oxidizing conditions), and sample 5 (RS-3 + 15% DJ Units 1 and 2 fly ash; at optimum H₂O and reducing conditions). The soil used in these samples was classified A-2-4 using the AASHTO system. Replicate 2-g samples of samples 1, 2, and 3 were taken through the procedure to provide solid material after each extraction step for XRD and SEM analyses. The particle size of the samples used in the extractions was less than 1.2 mm; greater than 50% of each sample passing a 420- μ m sieve. The extractions were performed in 50-mL Oak Ridge-type polypropylene centrifuge tubes. The samples were extracted sequentially using 25.0 g of the following chemical reagents to selectively remove the corresponding mineralogical phases:

Exchangeable:	0.05 M potassium nitrate (KNO ₃) for 16 hours
Adsorbed:	deionized, distilled water (H ₂ O) for 2 hours; repeated three times, and the data combined
Organic:	0.05 M sodium hydroxide (NaOH) for 16 hours
Carbonate:	0.05 M disodium ethylenediaminetetraacetic acid (Na ₂ EDTA) for 6 hours
Sulfide:	4 M nitric acid (HNO ₃) for 16 hours at 80°C

After each extraction step, the samples were centrifuged using a Beckman Model J2-21 centrifuge for 10 minutes at 10,000 rpm (maximum radius). After centrifuging, the liquid extracts were removed from the centrifuge tubes using disposable pipettes.

Analysis of the Selective Dissolution Solids and Extracts

The extracts were analyzed using inductively coupled plasma (ICP) spectrometry and ion chromatography. Samples of the original solid materials were also digested, and the resulting solutions were analyzed by ICP. The total amount of an element extracted through the application of the sequential extraction procedure was subtracted from the total content of that element in the sample. The resulting nonextractable elemental fraction was labeled the residual fraction. Also, replicate samples were extracted to provide solid material after each extraction step for analysis by XRD to evaluate any changes in the mineralogy of the solids during the extractions. The XRD analysis was conducted using the equipment previously described.

The elemental and anion data collected from selective dissolution of the duplicate samples were converted to microgram of analyte extracted per gram of solid on a dry-weight basis. These data were compensated for carry-over from one extraction step to the next, and the duplicate values from each sample were then averaged.

Although the elements in the exchangeable, adsorbed, organic, carbonate, and sulfide fractions correlate with selective dissolution by KNO_3 , H_2O , NaOH , EDTA , and HNO_3 , a selective dissolution reagent is not entirely selective. As a result, the elements extracted by specific reagents may or may not be representative of the designated chemical forms. However, they can be classified as chemically similar and extractable by the specific reagent. For these reasons, elemental fractions are identified by the particular reagent used in the extraction (e.g., KNO_3 -extractable).

Results and Discussion

pH Values of the Selective Dissolution Extracts

The pH values of the selective dissolution extracts are listed in Tables 19 and 20. The pH values for extracts from the soil/fly ash mixtures in the two sample sets vary little within each set. The pH values of the KNO_3 , H_2O , NaOH , and Na_2EDTA extracts from samples 4 and 5 (Table 19) are high, ranging from 8.8 to 12.1. The pH values of the KNO_3 , H_2O , and NaOH extracts from sample 1, sample 2, and sample 3 (Table 20) are also high (10.7 to 12.4). However, the Na_2EDTA extracts of the soil/fly ash mixtures listed in Table 20 have lower pH values (~ 6) than the Na_2EDTA extracts of the mixtures listed in Table 19 (pH ~ 9). The pH values of the KNO_3 and H_2O extracts of loamy sand soil (Table 20) are lower than the pH values of the KNO_3 and H_2O extracts from the soil/fly ash mixtures. This is most likely because the alkaline fly ash is present in the mixtures. However, the pH value of the loamy sand soil extract for the Na_2EDTA fraction is the same as those for the soil/fly ash mixtures. The pH values of the extracts are important because pH has an effect on elemental solubility.

Table 19. pH Values of the Selective Dissolution Extracts of Samples 4 and 5

Extract	Sample 4	Sample 5	Blank
KNO ₃ -Extractable	11.1	11.3	3.9
H ₂ O-Extractable	9.4	10.0	6.7
NaOH-Extractable	12.1	12.1	12.1
Na ₂ EDTA-Extractable	8.8	9.2	4.7
HNO ₃ -Extractable	<1	<1	<1

Table 20. pH Values of the Selective Dissolution Extracts of Loamy Sand Soil

Extract	Loamy Sand Soil	Sample 1 25% Fly Ash	Sample 2 20% Fly Ash	Sample 3 15% Fly Ash
KNO ₃ -Extractable	7.7	11.4	11.2	11.3
H ₂ O-Extractable	9.6	10.8	10.8	10.7
NaOH-Extractable	12.3	12.3	12.4	12.4
Na ₂ EDTA-Extractable	5.9	6.0	5.9	6.0
HNO ₃ -Extractable	<1	<1	<1	<1

Characteristics of the Solid Samples and the Selective Dissolution Extracts

The concentrations of trace and major elements in the solid samples tested in this study are listed in Tables 21 and 22. Of the elements listed, those with the highest concentrations were Al, Ca, Fe, Mg, and Si. The data in Table 22 show that mixing the fly ash with the loamy sand soil results in higher concentrations of all of the elements of interest except for Cd and Mo found in sample 3.

The values in Tables 21 and 22 were used to calculate the elemental concentrations in the residual solids remaining after the selective dissolution treatment. The percentages of the total elemental concentrations present in the selective dissolution fractions and the residual fractions are listed in Tables 23 and 24 and are also shown graphically in Figures 35 through 45.

Table 21. Total Elemental Concentrations in Samples 4 and 5

Element	$\mu\text{g/g}$	
	Sample 4	Sample 5
Al	46,600	40,800
B	152	152
Ba	1,400	1,330
Ca	35,000	32,600
Cd	<2	<2
Cr	23.4	21.6
Fe	15,600	14,800
Mg	7,880	7,190
Mn	234	218
Mo	<0.9	<0.9
Ni	13.7	12.4
Pb	35.8	43.4
Se	<9	<9
Si	144,000	105,000

Table 22. Total Elemental Concentrations in Loamy Sand Soil/Dave Johnston Fly Ash Mixtures

Element	$\mu\text{g/g}$			
	Loamy Sand Soil	Sample 1 25% Fly Ash	Sample 2 20% Fly Ash	Sample 3 15% Fly Ash
Al	35,600	57,600	62,400	47,700
B	<19	71.8	73.4	55.5
Ba	492	717	579	659
Ca	5,610	26,400	20,200	20,000
Cd	<0.8	<0.8	<0.8	<0.8
Cr	16.9	33.3	35.8	29.8
Fe	9,310	14,400	14,400	12,700
Mg	2,730	5,440	5,370	4,720
Mn	145	214	227	198
Mo	<1	1.56	1.79	<1
Ni	8.30	15.1	15.2	13.2
Pb	21.8	31.5	44.4	29.9
Se	<0.4	1.17	1.07	1.50
Si	191,000	204,000	210,000	319,000

The major residency of Ca, Fe, Mg, Mn, and Ni in all of the samples tested is in the HNO_3 -extractable fraction (sulfide fraction), and the major residency of Al and Si in the samples is in the residual fraction. In the loamy sand soil and samples 3, 4, and 5; approximately 70% of the Ba is in the residual fraction and approximately 20% is in the HNO_3 -extractable fraction. In samples 4 and 5, approximately 40% of the total Ba is in the HNO_3 -extractable fraction, 30% is in the residual fraction, and 20% is in the Na_2EDTA -extractable fraction (carbonate fraction). The concentration of B in the loamy sand soil is less than the analytical detection limit, and no B was detected in the selective dissolution extracts of the soil. In all of the soil/fly ash mixtures, the majority of B is in the HNO_3 -extractable fraction. However, in samples 4 and 5, the percentages of B in the HNO_3 extracts (35 and 36%) are only slightly higher than in the Na_2EDTA fractions (33 and 32%). The majority of Pb in samples 2, 3, 4, and 5 is in the residual fraction, while the major residency of Pb in sample 1 and the loamy sand soil is in the HNO_3 -extractable fraction. However, for samples 1, 2, and 3 the percentages of Pb in the HNO_3 -extractable fractions are similar to the Pb percentages in the residual fractions. The majority of Cr in all of the samples is in the HNO_3 -extractable fraction, except for sample 3 where it is in the residual fraction.

Trace Element Associations with Major Elements

Similarities in distribution of trace and major elements suggest a solid-phase association. By comparing the extractability of the major elements and the trace elements, possible solid-phase associations may be determined indirectly. The information obtained from mineralogical characterization of the solids can also be used to identify possible solid-phase associations. The information from direct and indirect examination of the samples can be correlated.

In samples 1, 2, and 3, Al and Ba have similar extractabilities in all of the selective dissolution fractions (Figure 46). This suggests a possible solid-phase association of these elements in the samples.

As shown in Figure 47, for samples 4 and 5, Fe and Mn have similar extractabilities in all of the selective dissolution fractions. This suggests a possible solid-phase association of these elements in the samples.

Elemental Extractabilities

The data listed in Table 23, and represented in Figures 35-38, 40, and 45 show that the differences in the extractabilities of Al, B, Ba, Ca, Fe, and Si from samples 4 and 5 are less than or equal to 5% for each of the selective dissolution fractions. This suggests similar solid-phase speciations of these elements in these samples.

Table 23. Percent of Total Elemental Concentration Present in the Selective Dissolution Fractions of Samples 4 and 5

Element	Sample	Selective Dissolution Fraction, %					
		KNO ₃	H ₂ O	NaOH	Na ₂ EDTA	HNO ₃	Residual
Al	4	1	1	1	6	30	61
	5	1	1	1	5	35	57
B	4	7	16	2	32	36	7
	5	1	1	1	5	35	57
Ba	4	6	1	1	22	37	33
	5	6	1	1	20	39	33
Ca	4	8	6	1	36	42	7
	5	6	5	1	38	44	6
Cr	4	3	0	1	11	47	38
	5	2	0	1	8	53	36
Fe	4	0	0	0	9	57	34
	5	0	0	0	7	60	33
Mg	4	0	0	0	38	49	13
	5	0	0	0	7	60	33
Mn	4	0	0	0	16	56	28
	5	0	0	0	11	63	26
Ni	4	0	0	0	27	57	16
	5	0	0	0	21	68	11
Pb	4	0	0	3	22	21	54
	5	0	0	2	13	16	69
Si	4	0	0	0	2	1	97
	5	0	0	0	2	1	97

The data listed in Table 23 and represented in Figures 39 and 41-43 show that a higher percentage of Cr (6%), Mg (11%), Mn (7%), and Ni (11%) is extracted in the HNO₃-extractable fraction (sulfide fraction) of sample 5 than in the HNO₃-extractable fraction of sample 4. Although these differences are relatively small, these data suggest a possible difference in the speciation of these elements between the two soil/fly ash mixtures. The HNO₃-extractable fraction contains the highest percent of these elements in both samples.

Table 24. Percent of Total Elemental Concentration Present in the Selective Dissolution Fractions of Loamy Sand Soil and Soil/Dave Johnston Fly Ash Mixtures

Element	Sample	Selective Dissolution Fraction, %					
		KNO ₃	H ₂ O	NaOH	Na ₂ EDTA	HNO ₃	Residual
Al	Loamy Sand Soil	0	5	0	0	38	57
	Sample 1 (25% FA ^a)	0	1	1	2	29	67
	Sample 2 (20% FA)	0	2	1	3	28	66
	Sample 3 (15% FA)	0	1	1	2	23	73
B	Loamy Sand Soil ^b	--	--	--	--	--	--
	Sample 1 (25% FA)	24	26	5	23	53	-31
	Sample 2 (20% FA)	29	16	3	11	35	6
	Sample 3 (15% FA)	29	20	4	14	36	-3
Ba	Loamy Sand Soil	6	1	0	2	17	74
	Sample 1 (25% FA)	4	0	0	7	28	61
	Sample 2 (20% FA)	4	0	0	8	20	68
	Sample 3 (15% FA)	3	0	0	6	19	72
Ca	Loamy Sand Soil	26	2	1	18	38	15
	Sample 1 (25% FA)	10	4	1	22	51	12
	Sample 2 (20% FA)	16	1	1	25	48	9
	Sample 3 (15% FA)	12	2	1	20	39	26
Cr	Loamy Sand Soil	0	0	0	0	82	18
	Sample 1 (25% FA)	5	0	1	12	52	30
	Sample 2 (20% FA)	4	0	0	9	48	39
	Sample 3 (15% FA)	4	0	1	9	39	47
Fe	Loamy Sand Soil	0	0	0	1	103	-4
	Sample 1 (25% FA)	0	0	0	7	68	25
	Sample 2 (20% FA)	0	0	0	9	67	24
	Sample 3 (15% FA)	0	0	0	7	61	32
Mg	Loamy Sand Soil	0	10	0	3	103	-16
	Sample 1 (25% FA)	0	0	0	22	62	16
	Sample 2 (20% FA)	0	2	0	20	62	16
	Sample 3 (15% FA)	0	1	0	19	54	26
Mn	Loamy Sand Soil	0	0	0	36	82	-18
	Sample 1 (25% FA)	0	0	0	32	55	13
	Sample 2 (20% FA)	0	0	0	36	48	16
	Sample 3 (15% FA)	0	0	0	34	44	22
Ni	Loamy Sand Soil	0	0	0	11	90	-1
	Sample 1 (25% FA)	0	0	0	14	59	27
	Sample 2 (20% FA)	0	0	0	17	57	26
	Sample 3 (15% FA)	0	0	0	15	44	41
Pb	Loamy Sand Soil	0	0	3	20	54	23
	Sample 1 (25% FA)	0	0	0	23	42	35
	Sample 2 (20% FA)	0	0	2	20	32	46
	Sample 3 (15% FA)	0	0	3	22	36	39
Si	Loamy Sand Soil	0	2	0	0	0	98
	Sample 1 (25% FA)	0	0	0	1	0	99
	Sample 2 (20% FA)	0	1	0	2	0	97
	Sample 3 (15% FA)	0	0	0	1	0	99

^a FA = Fly Ash

^b The total concentration of B in the loamy sand soil is <19 µg/g.

In addition, the data listed in Table 23 and represented in Figures 41, 43, and 44 show that a higher percentage of Mg (11%), Ni (6%), and Pb (9%) is extracted in the Na₂EDTA-extractable fraction (carbonate fraction) of sample 4 than in the Na₂EDTA-extractable fraction of sample 5. Also, 15% more Pb was extracted from sample 4 than from sample 5 in the selective dissolution treatment. Although these differences are relatively small, these data suggest that there may be a difference in the speciation of these elements between the two soil/fly ash mixtures.

A comparison of the elemental extractabilities from the three mixtures shows that for B, Ba, and Pb, a higher percent (~17, ~8, and ~8%, respectively) of these elements is extracted in the HNO₃ fraction for sample 1 (25% fly ash) (Table 24; Figures 36, 37, and 44). In addition, lower percents of Ca (~10%), Cr (~11%), Fe (~7%), Mg (8%), and Ni (~14%) are extracted in the HNO₃ fraction for sample 3 (15% fly ash) (Table 24; Figures 38-41 and 43). These data suggests that there are differences in the solid-phase speciations of these elements in the mixtures.

A comparison of the extractabilities of sample 1 (25% fly ash), sample 2 (20% fly ash), and sample 3 (15% fly ash) with the extractability of the loamy sand soil used in the mixtures (Table 24) shows major differences in the extractabilities of certain elements. Approximately 11% more Al is extracted in the HNO₃ fraction from the soil than from the three mixtures. Approximately 30 to 50% more Cr, Fe, Mg, and Ni is extracted in the HNO₃ fraction from the soil than from the three mixtures. Approximately 20% more Mn is extracted in the HNO₃ fraction from the soil than from the three mixtures; and approximately 17% more Pb is extracted in the HNO₃ fraction from the soil than from the three mixtures. The residual amounts of these elements remaining after the selective dissolution treatment are much lower in the loamy sand soil than in the soil/fly ash mixtures (Figures 35 and 39-44).

The data in Table 20 show that the pH values of all of the HNO₃ extracts are less than one, which suggests no pH effect on elemental solubility between the extracts. The elements in the soil/fly ash mixtures may be trapped in the solid matrices of the mixtures. Lower percents of Al, Cr, Fe, Mg, Mn, and Ni are extracted in the HNO₃ fraction of sample 3 (15% fly ash) than from the other two soil/fly ash mixtures and the loamy sand soil (Table 24; Figures 35 and 39-43). The data in Table 21 show that the soil/fly ash mixtures contain higher concentrations of Si than the loamy sand soil, and sample 3 (15% fly ash) contains the highest concentration of Si. This suggests that there may be a possible correlation between the Si content in the samples and the release of certain elements from the solid matrix.

Mineralogical Analysis

Selective dissolution residues were evaluated using X-ray diffraction analysis. Quartz and feldspar were detected in all of samples (Table 25).

Table 25. Crystalline Phases Positively Identified in Materials Following the Respective Selective Dissolution Extraction Procedure

Sample Identification	Extraction Method	Mineral Phases
Loamy sand soil	KNO ₃	Quartz, Feldspar, clays
Sample 1	KNO ₃	Quartz, Feldspar, Clinoferrosilite
Sample 2	KNO ₃	Quartz, Feldspar
Sample 3	KNO ₃	Quartz, Feldspar
Loamy sand soil	H ₂ O	Quartz, Feldspar, possible Gismondine
Loamy sand soil	H ₂ O	Quartz, Feldspar
Loamy sand soil	H ₂ O	Quartz, Feldspar
Loamy sand soil	H ₂ O	Quartz, Feldspar
Sample 1	H ₂ O	Quartz, Feldspar
Sample 1	H ₂ O	Quartz, Feldspar, Clinoferrosilite
Sample 1	H ₂ O	Quartz, Feldspar, Clinoferrosilite
Sample 2	H ₂ O	Quartz, Feldspar
Sample 2	H ₂ O	Quartz, Feldspar, clay minerals
Sample 2	H ₂ O	Quartz, Feldspar
Sample 3	H ₂ O	Quartz, Feldspar
Sample 3	H ₂ O	Quartz, Feldspar
Loamy sand soil	NaOH	Quartz, Feldspar, clay minerals
Sample 1	NaOH	Quartz, Feldspar
Sample 2	NaOH	Quartz, Feldspar
Sample 3	NaOH	Quartz, Feldspar
Loamy sand soil	Na ₂ EDTA	Quartz, Feldspar
Sample 1	Na ₂ EDTA	Quartz, Feldspar
Sample 2	Na ₂ EDTA	Quartz, Feldspar
Sample 3	Na ₂ EDTA	Quartz, Feldspar, Hedenbergite
Loamy sand soil	HNO ₃	Quartz, Feldspar
Loamy sand soil	HNO ₃	Quartz, Feldspar
Sample 1	HNO ₃	Quartz, Feldspar, Muscovite
Sample 1	HNO ₃	Quartz, Feldspar, Clinoferrosilite
Sample 2	HNO ₃	Quartz, Feldspar, Clinoferrosilite
Sample 2	HNO ₃	Quartz, Feldspar
Sample 3	HNO ₃	Quartz, Feldspar, Ankerite, clay minerals
Sample 3	HNO ₃	Quartz, Feldspar, Hedenbergite

SECTION IV.
INVESTIGATION OF MOBILE COLLOIDS FORMED IN SOIL/FLY ASH MIXTURES

Introduction

The formation, transport, and deposition of mobile colloids resulting from fly ash materials may enhance the movement of elements from fly ash disposal and fly ash use sites. A number of studies have indicated that colloids play a major role in transport of various materials from disposal environments. Gschwend et al. (1990) reported that a $\text{Fe}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ colloidal phase was formed from a combination of sewage-derived phosphates and ferrous iron from aquifer solids. In a recent review article, McCarthy and Zachara (1989) noted that colloids may play a significant role in the transport of materials from disposal sites. They also suggested that the removal or creation of colloids may occur during sampling. Oxygen content, carbon dioxide content, temperature, pH, Eh, and light were possible factors involved in the formation or creation of colloids. Gschwend et al. (1990) used sampling techniques designed to prevent the formation of colloidal material and determined that colloidal formation was associated with coal ash disposal. The type of colloidal material formed was primarily silicates. The formation of the colloids was attributed to the dissolution of carbonates involved in cementation and to the release of colloidal silicates. Some fly ash materials contain potentially toxic elements and mobile colloids may be formed as a result of the reactive nature of fly ash. The study reported herein was undertaken to evaluate the solution phases extracted from the soil/fly ash mixtures, to determine whether or not soluble colloids are present in such materials, and to develop an understanding of their reactivity.

Materials and Methods

Solutions extracted from specimens composed of fly ash and soil were analyzed using a gel permeation chromatography (GPC) technique. The fly ash used to make the specimens was collected from the Dave Johnston power plant, Units 1 and 2, located near Glenrock, Wyoming. The soils used in the mixtures had loamy sand textures and were classified A-4-2. The soil was collected from the county road located south of the Dave Johnston power plant and is identified as RS-3.

Specimens were taken from the soil/fly ash mixtures that had been cured under varying conditions for 7 days. Samples 1 and 2 were cured in oxidizing conditions, and sample 3 was cured in reducing conditions (N_2 atmosphere). Solution was extracted from the soil/fly ash mixtures using an immiscible displacement procedure (Mubarek and Olsen 1976; Kittrick 1980, 1983). A centrifuge was used to remove particulates from the extraction solution. The extracts were tested to determine the presence of colloidal materials.

Gel permeation columns (Model C26/100) were set up using Sephadex G-25 fine gel, which has a molecular fractionation range of 25 to 5,000 daltons. The columns were used with a constant head and gravity feed from an Erlenmeyer flask located about 8 inches above the top of the column. Blue Dextran 2000, which has a molecular weight of ~2,000,000

daltons, was used to determine V_0 . V_0 was found to be 175 mL, which corresponded to about 115 minutes of elution time. Two mL of the supernatant collected from each specimen were run through the GPC column. During a separation run, the first 100 minutes of effluent from the column was discarded. After the first 100 minutes, samples were collected in polypropylene test tubes in 2-minute aliquots (about 3.0 mL). Each sample was composited into a test tube with 0.5 mL from each of six adjacent test tubes. Thus, each of the composite samples represented 12 minutes of effluent. The composite samples were analyzed using ICP to determine the presence of Ag, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, Sr, V, and Zn. Si and, in some cases, Ca were used as labels to identify colloidal materials with high molecular weights.

Results and Discussion

The extracts contained considerable amounts of particulates. Following the removal of the particulates, the resulting supernatant appeared clear. The solutions were then run through the GPC column for size separation to determine whether or not colloidal materials were present. The initial screening showed that Si was present in the eluant from 233 to 279 minutes. Subsequent analysis of this region narrowed the Si peak to between 233 and 275 minutes of elution time (Figure 48). This Si peak was identified as $\text{Si}(\text{OH})_4$, and did not correspond to a colloidal material.

The data show that colloids were not present in solution under the conditions of this experiment. Colloids containing silica compounds may have polymerized and precipitated or sorbed to available sites on soil particles.

Additional experiments were done on an extract from sample 2 (pH 10.9) to determine whether or not the pH difference between the eluant and the soil/fly ash material was influencing the stability of colloids present in the solution. The water in the gel column and the supply water were brought to pH 11.0 with 1 N NaOH. The pH of the final effluent following 288 minutes of eluting was 10.8. These elution samples were analyzed for Ca; however, the fractionated solutions did not contain detectable concentrations of Ca.

These data suggest that colloids containing Si and Ca are not present in solution under the environment existing in the soil/fly ash matrix. However, colloids could possibly form at the contact zones between the soil/fly ash and adjacent materials where lower pH values may exist. This topic was not considered in this research.

CONCLUSIONS

1. Soil/fly ash specimens made from Laramie River fly ash had higher unconfined compression strength than specimens made with Dave Johnston fly ash.
2. Soil/fly ash specimens made with Dave Johnston fly ash were more ductile, or less brittle, than the specimens made from Laramie River fly ash. The questions associated with these findings should be investigated in future research.
3. The mineralogical evaluations did not provide definitive data showing the minerals responsible for strength development.
4. Selective dissolution of the soil/fly ash mixtures shows that most of the "potentially" toxic elements are associated with the sulfide phase and with the residual material.
5. Formation of colloidal material capable of mobilizing potentially toxic elements was not occurring in the soil/fly ash mixtures.

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DISCLAIMER

Mention of specific brand names or models of equipment is for information only and does not imply endorsement of any particular brand.

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APPENDIX

Figures

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