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Journal Article

**1994
WWRC-94-12**

In

Water Research

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REACTION OF CO₂ WITH ALKALINE SOLID WASTES TO REDUCE CONTAMINANT MOBILITY

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(First received April 1993; accepted in revised form September 1993)

Abstract—The objective of this study was to determine the effects of a CO₂ treatment on the pH and soluble concentrations of inorganic contaminants in alkaline fly ash and spent shale solid wastes. A two-level (low and high), three-variable (moisture, time and pressure) statistical experiment was used to determine optimum CO₂ treatment conditions. Treated and untreated samples were subjected to solubility and X-ray diffraction (XRD) studies. CO₂ treatment conditions of 40 psi of pressure, 20% moisture and 120 h effectively precipitated calcite, and thus lowered the pH and leachable concentrations of certain inorganic contaminants (e.g. Cd, Pb, Zn, Mn, As and Se) in alkaline fly ash and spent shale samples. For instance, ≈64–89% reduction in the leachable concentration of Mn was observed for CO₂ treated samples. Our results suggest that reaction with CO₂ under slightly elevated pressures is an effective means of reducing the soluble concentrations of certain inorganic contaminants in alkaline solid wastes, which should prevent their migration from disposal environments into groundwater.

Key words—alkaline solid wastes, contaminants, CO₂ reaction, immobilization of contaminants

INTRODUCTION

Many countries use fossil fuels, such as coal and oil shale to produce energy. However, the combustion of coal and oil shale results in large volumes of solid wastes such as fly ash and spent shale, respectively. For example, in 1988 electric utilities in the U.S.A. generated ≈90 million tons of coal ash. About 62% was fly ash, 23% was bottom ash and boiler slag and 15% was flue gas desulfurization sludge. Of the total amount of ash produced, 20–25% was used in cement products, road bases and asphalt. The remaining 75–80% was placed in ponds and landfills, creating a potential disposal problem due to the leaching of certain contaminants such as Cd, Pb and Se (Cherry and Guthrie, 1977; Adriano *et al.*, 1980; Kopsick and Angino, 1981; Humenick *et al.*, 1983; U.S. EPA, 1988; Carlson and Adriano, 1993).

Extraction of oil from raw shale results in production of large volume of spent shale solid waste. Stiffler *et al.* (1974) reported that the volume of spent shale will increase ≈ by 50% over the raw shale due to crushing and expansion upon heating. Similar to fly ash, spent shale also represents a potential for leaching of certain inorganic contaminants, particularly F and Mo (Stollenwerk and Runnells, 1981; Stark and Redente, 1986; Essington, 1989).

Calcium (Ca) is one of the most abundant elements in fly ash and spent shale, because it is not volatilized during the combustion process of coal and oil shale.

Additionally, during the combustion process, high temperatures drive off CO₂ from coal and oil shale, and as a result the pH of aqueous extracts of fly ash and spent shale increases; this affects the solubility and mobility of inorganic contaminants. Therefore, reactions involving Ca and CO₂ are expected to control the pH, solubility and mobility of inorganic contaminants in fly ash and spent shale (Schramke, 1992).

Despite the importance of these reactions, only a few studies have examined the effects of a CO₂ treatment on the pH, solubility and mobility of inorganic contaminants in fly ash and spent shale (Reddy *et al.*, 1986, 1991; Essington, 1989; Schramke, 1992). Most of these studies either bubbled CO₂ through fly ash and spent shale slurries or aqueous solutions. However, such techniques are not practical for field application and require a longer time (months and years) to minimize the mobility of inorganic contaminants. Thus, the objectives of this study were:

- To react alkaline fly ash and spent shale samples with minimum moisture under CO₂ gas pressure and
- To determine the effects of CO₂ pressure treatment on the leachability of inorganic contaminants.

MATERIALS AND METHODS

Fly ash samples were collected from the Laramie River Station, Wheatland and the Central Energy Plant, Laramie,

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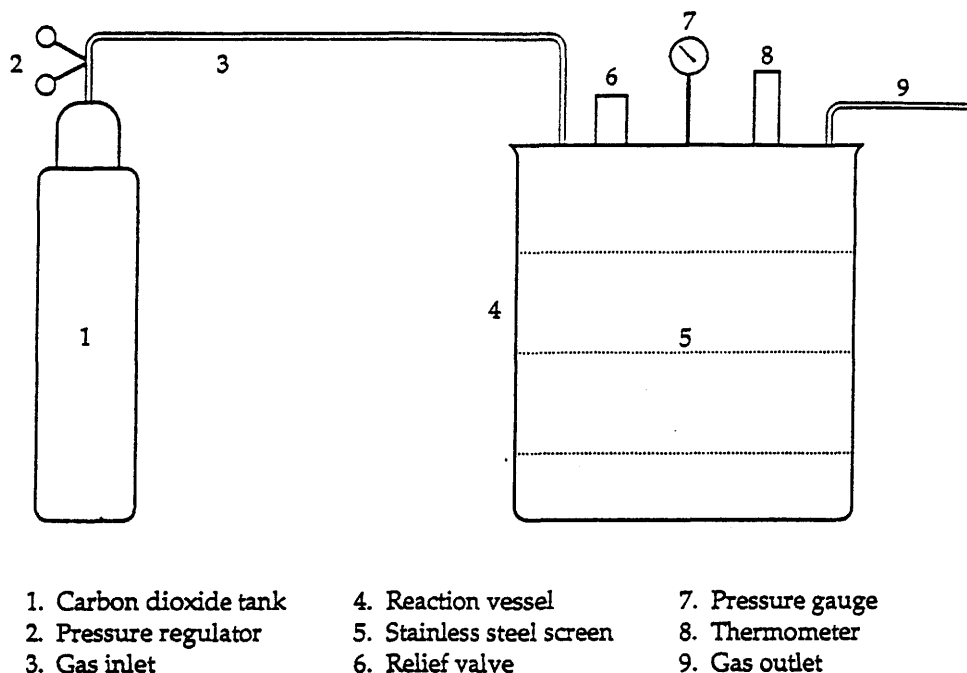


Fig. 1. Design of CO₂ treatment method for alkaline solid wastes.

Wyoming, U.S.A. These utilities burn coal from the Powder River Basin and the Hanna Basin, Wyo., respectively at ≈ 2273 K. Fly ash samples were collected either directly from the bag-house or electrostatic precipitators, placed immediately in plastic containers without head space and closed tightly.

Spent shale samples used in this study were Western Reference Green River Formation oil shale from the Piceance Creek Basin in Colorado, U.S.A., which had been processed at ≈ 1000 and 1295 K to produce PPP 6 (Paraho Pilot Process) and Lurgi, respectively. These samples were obtained from the Western Research Institute, Laramie, Wyo., U.S.A.

Samples were ground to pass a 0.25 mm sieve and used for subsequent experiments. For initial characterization, samples were subjected to the following studies: pH, total elemental composition, mineral composition, calcium carbonate content and leachability of inorganic contaminants.

The pH of the samples was measured in a saturated paste with an Orion combination electrode (Rhoades, 1982). Samples were digested in nitric acid (6 N) plus perchloric acid (60%) and determinations were made for the total concentration of Al, S, P, Ca, Fe, Mg, Mn, Cu, Cd, Zn, As, Se, Mo by inductively coupled plasma-optical emission spectrometry (ICP-OES). Sodium and K were determined by atomic absorption (AA). Complete details regarding these procedures are reported in Lim and Jackson (1982).

X-ray diffraction (XRD) analysis was performed on randomly oriented slurried samples with a Scintag PAD V powder diffractometer using CuK α radiation. XRD profiles obtained for samples were compared with Scintag software containing JCPDS (Joint Committee on Powder Diffraction Standards) files to determine major mineral phases.

Calcium carbonate content was measured using the method of Nelson (1982). In this procedure few grams of samples were dissolved in 3 N HCl and calcium carbonate content was calculated from the sample weight loss. The %CO₂ absorbed by fly ash and spent shale samples was calculated from calcium carbonate content.

AB-DTPA (ammonium bicarbonate diethylene triamine penta acetic acid), a multi-element extractant (Soltanpour

and Schwab, 1977) which removes both available and potentially soluble fractions, was used in predicting the leachability of Cd, Pb, Zn, Mn, Ni, Cu, As and Se from samples. The pH of AB-DTPA extracting solution is maintained at 7.6 using either NH₄OH or HCl, which allows for simultaneous determination of inorganic contaminants in slightly alkaline leachates. Several studies have tested this extraction procedure for predicting the availability of inorganic contaminants in alkaline materials (Folsom *et al.*, 1981; Schwab *et al.*, 1991).

A 30-cm dia. by 55 cm long vessel was designed (Fig. 1) for reacting samples under CO₂. The reaction chamber was connected to instrument grade CO₂ (99.9% pure). A pressure relief valve, pressure gauge and thermometer were inserted on top of the lid to measure and release pressure and monitor temperature, respectively. A stainless steel screen with filter paper was placed inside the vessel to hold samples. The gas outlet was vented into a fume hood.

A two-level, three-variable statistical experiment was (Table 1) conducted to test the effects that CO₂ reaction variables (moisture, pressure and time) had on the pH and solubility of inorganic contaminants. Information obtained from these experiments was used to form the basis for making selection of final CO₂ reaction conditions [20% moisture (by weight basis), 40 psi of CO₂ (99.9%) pressure and 120 h]. Approximately 1 kg of each sample was reacted under the final CO₂ reaction conditions and treated samples were analyzed as follows.

Samples were subjected to pH, XRD calcium carbonate content and AB-DTPA analyses, as explained earlier. Additionally, treated samples (1:4 solid-to-water ratio) were reacted on a mechanical shaker for determining the potential solid phases controlling the dissolved concentration of

Table 1. Three-variable, two-level statistical design for CO₂ treatment

Variable	Low level	Midpoint	High level
Pressure, X_1 (psi)	5	40	75
Moisture, X_2 (%)	5	15	25
Time, X_3 (h)	0.5	5.25	10

Table 2. Selected properties of the samples used in the study (units are mg/kg)

Property	Fly ash 1	Fly ash 2	PPP 6	Lurgi
Al	33,800	400	3300	2600
Na	59,300	10,000	58,200	6600
S	2400	16,600	9400	6200
P	2100	4700	2300	1100
Ca	53,049	51,730	41,116	42,806
Mg	1000	4662	9605	5240
K	23,269	20,319	20,461	26,165
Fe	39,217	65,398	37,272	27,950
Mn	2136	425	361	347
Cu	144	189	80	79
Pb	235	94	170	36
Zn	424	176	130	139
Cd	<10.0	<10.0	<10.0	<10.0
As	<20.0	<20.0	<20.0	<20.0
Se	<20.0	<20.0	<20.0	<20.0
Mo	38.0	13.0	23.0	43.0
Major mineral phases	Quartz Mullite Anhydrite	Quartz Calcium oxide Anhydrite	Quartz Periclase Diopside	Quartz K-feldspar Periclase
pH (saturated paste)	Amorphous phases 12.3	Amorphous phases 12.8	Calcite 12.4	Calcite 12.3

Ca in aqueous extracts. After 1, 3, 7 and 14 days reaction time, sample suspensions were filtered through 0.45 μm Millipore filters under an Ar atmosphere to minimize the uptake of atmospheric CO₂ by leachates.

Each filtered leachate was divided into two subsamples. One was acidified to pH 5–6 with HNO₃. The other subsample was left unacidified. Acidified subsamples were analyzed for Ca, Mg, Al, Fe, Mo, Si (ICP-OES), Na and K (AA). Unacidified subsamples were analyzed for pH, carbonate species (Reddy *et al.*, 1991), Cl⁻, F⁻ and SO₄²⁻ (ion chromatography).

The pH and total elemental concentrations of aqueous extracts were used as input to the GEOCHEM model (Sposito and Mattigod, 1980) to calculate chemical speciation (i.e. ion activities). This model can be obtained from the University of California, Berkeley, Calif., U.S.A. by contacting the senior author. Solution species that may be significant in alkaline environments were added to the GEOCHEM database. These details are reported in Reddy *et al.* (1993). Ion activity products (IAPs) were calculated from ion activities and compared with solubility products (K_{sp} s) to evaluate the potential solid phases controlling the dissolved Ca concentrations.

It was assumed that IAP within ± 0.50 log units of K_{sp} of solid phase represented near saturation, and that the solid phase was a probable control on the concentrations of the ions involved. The difference within that range is accounted for by the uncertainty of IAP estimations and K_{sp} measurements in the chemical literature (Stumm and Morgan, 1981).

RESULTS AND DISCUSSION

Table 2 shows data on selected properties of the samples used in the study. Fly ash and spent shale samples were different in their chemical composition. All samples are enriched in Al, Na, S, P, Ca, K and Fe. Fly ash 1 samples contained higher concentrations of Al, Na and Mn and lower concentrations of Mg and Fe when compared with fly ash 2 samples. Similar results were found for PPP 6 and Lurgi samples. Other elements such as Cu, Pb, Zn and Mo were present in minor concentrations. Cd, As and Se concentrations in all samples were below 20.0 mg/kg.

XRD analysis suggested that fly ash samples consisted largely of quartz, mullite, amorphous phases and to some extent anhydrite and calcium oxide. On

the other hand, spent shale samples consisted mainly of quartz, periclase, diopside and K-feldspar. Formation of these phases requires high temperatures (i.e. >1500 K). Such temperatures are usually attained during the combustion process of coal and oil shale.

Saturated paste pH of untreated fly ash and spent shale samples ranged from 12.3 to 12.8 (Table 2). During the combustion, high temperatures drive off CO₂ from coal and oil shale resulting in the formation of oxide (e.g. CaO) and silicate phases (e.g. CaSiO₃). These phases react rapidly with water, and as a result, the pH of aqueous extracts of fly ash and spent shale approach 12.0.

Since a two-level, three-variable statistical design was adopted, eight experiments were required. Four midpoint replications were also included in experiments to estimate the error variance. Calculated values of F were compared with the critical F -value [$F_{0.05}(1,3) = 10.10$] to decide which effects or interactions were statistically significant (Miller and Freund, 1985). Results from these experiments suggested that the variable time as well as the interaction between time and moisture were significant for the application of CO₂ to fly ash and spent shale samples. High moisture and longer reaction time played significant roles in the success of CO₂ reaction. However, higher pressure was not found necessary.

Chemical data from 1, 3, 7 and 14 day solubility studies suggested that 14 days provided the most consistent results. Therefore, solubility data from 14 days were used to determine the potential solid phases controlling the dissolved Ca concentration in aqueous extracts from CO₂ treated samples. A comparison between log IAPs and log K_{sp} s for calcium oxide, calcium hydroxide and calcium silicate indicated a high degree of undersaturation, suggesting that these phases were not controlling the dissolved concentrations of Ca in CO₂ treated samples.

A plot of log IAP of calcite versus pH is shown

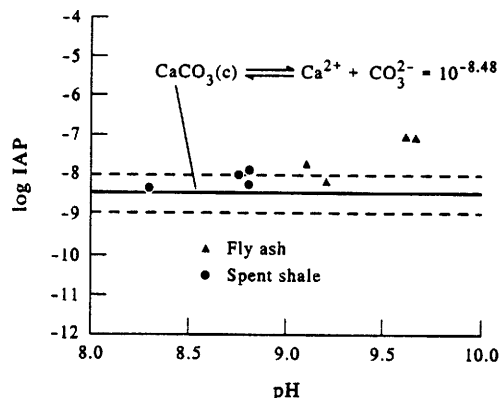


Fig. 2. The IAPs of calcite for CO₂-treated samples (pressure = 40 psi, moisture = 20%, reaction time = 120 h).

in Fig. 2 together with the solubility product (K_{sp}) of calcite. The K_{sp} value for calcite is taken from Plummer and Busenburg (1982). These results suggest that log IAPs for CO₂ treated samples are near saturation with respect to calcite (a mean value of -8.10 ± 0.21), except fly ash 1 samples. XRD analysis indicated the presence of or an increase in the intensity of calcite peaks for CO₂ treated samples when compared with untreated samples. These results suggest that reacting moist alkaline fly ash and spent shale samples under mild CO₂ pressures causes the precipitation of calcite and reduction in the pH.

Rai *et al.* (1987), on the basis of thermodynamic evaluations, suggested that dissolved Ca concentrations in fly ash samples are controlled by CaO and/or Ca(OH)₂. However, in CO₂ reacted samples the dissolved Ca concentrations would be controlled by calcite. Long term leaching experiments (Dudas, 1981) have suggested similar results. Reddy *et al.* (1986) and Essington (1989) have reported that continuous bubbling of CO₂ through aqueous extracts of spent shales, for 6–9 months, causes dissolution of silicates, precipitation of calcite and reduction in the pH from 12.0 to approx. 8.0. In our experiments, reacting moist fly ash and spent shale samples under slightly elevated CO₂ pressures for only 120 h accelerated precipitation of calcite and effectively lowered pH.

The effects of CO₂ treatment on CaCO₃ content and CO₂ uptake are summarized in Table 3. These

Table 3. Effects of CO₂ treatment (pressure = 40 psi, moisture = 20%, reaction time = 120 h) on the pH and calcium carbonate content

Sample	Treatment status	pH	%CaCO ₃ content	%CO ₂ absorbed
Fly ash 1	Untreated	12.3 ± 0.01	2.7 ± 0.03	
	Treated	9.7 ± 0.06	6.5 ± 0.9	3.8
Fly ash 2	Untreated	12.8 ± 0.01	6.0 ± 2.9	
	Treated	9.1 ± 0.1	20.0 ± 2.2	14.0
PPP 6	Untreated	12.4 ± 0.01	6.0 ± 1.8	
	Treated	8.8 ± 0.01	14.0 ± 0.2	8.0
Lurgi	Untreated	12.3 ± 0.01	19.0 ± 3.2	
	Treated	8.9 ± 0.02	23.0 ± 7.6	4.0

data suggest that fly ash and spent shale samples are effective in absorbing CO₂. Untreated fly ash samples were low in CaCO₃ content compared to spent shale samples because the combustion temperatures for coal are generally higher (>1500 K) than oil shale. High temperatures usually decompose carbonate phases rapidly during the combustion process. Both %CaCO₃ content and %CO₂ uptake increased upon the CO₂ reaction. However, the efficiency of a CO₂ treatment may depend on the type of coal and oil shale used in the combustion process as well as chemical composition of fly ash and spent shale samples.

The CO₂ treatment effects on the leachability of inorganic contaminants are summarized in Table 4. These results show that CO₂ treatment was effective in reducing the concentrations of certain contaminants in both materials. For example, Mn concentrations were decreased from 2.2 to 0.8 mg/l for fly ash 1 and from 15.5 to 2.7 mg/l for fly ash 2. For spent shale samples, CO₂ treatment decreased Mn concentrations from 12.0 to 1.3 mg/l for PPP 6 and from 20.0 to 2.2 mg/l for Lurgi. Similar effects were observed for Cd, Pb and Zn.

Results in this study suggested that reacting moist fly ash and spent shale samples under slightly elevated CO₂ pressures caused the precipitation of calcite. This effectively reduced the pH as well as leachable concentrations of certain inorganic contaminants. A possible reason for a decrease in the concentration of leachable metal contaminants (e.g. Cd, Pb, Zn and Mn) is probably due to the precipitation of metal carbonates. In fly ash and spent shale samples, these metals are present in the form of oxides and/or hydroxides [e.g. CdO, Pb(OH)₂] due to the combustion process. However, when moist alkaline fly ash and spent shale samples are reacted under CO₂ pressure, oxides and/or hydroxides were probably converted to carbonates (e.g. CdCO₃, PbCO₃), which are more insoluble than oxides and/or hydroxides (Rai *et al.*, 1987).

A reduction in leachable As and Se is attributable to an increased sorption of these elements by iron oxides at lower pH (Theis and Wirth, 1977). It is well established that by decreasing the soluble concentration of a contaminant, the mobility of that contaminant will also be reduced. The CO₂ treatment process described in this study involves treatment of materials in a solid form containing very small amounts of moisture rather than a slurry or an aqueous solution phase, and requires much shorter reaction times (under low pressure) than reported recently for aqueous slurries (Schramke, 1992; Essington, 1989; Reddy *et al.*, 1986).

The process described here has, under laboratory conditions, demonstrated its potential to reduce the leachability of certain harmful inorganic contaminants (e.g. Cd, As and Se) thereby minimizing the risk of these elements contaminating various aspects of the environment, including groundwaters. However,

Table 4. Effects of CO₂ treatment (pressure = 40 psi, moisture = 20%, reaction time = 120 h) on the leachability of certain inorganic contaminants (units are mg/l)

Sample	Cd	Pb	Zn	Mn	Ni	Cu	As	Se
Fly ash 1 BT*	0.1 ± 0.0	BD	0.8 ± 0.1	2.2 ± 0.05	0.2 ± 0.05	2.3 ± 0.05	BD*	BD
Fly ash 1 AT*	0.05 ± 0.0	BD	0.3 ± 0.1	0.8 ± 0.05	0.1 ± 0.05	0.6 ± 0.01	BD	BD
Fly ash 2 BT	1.8 ± 0.05	19.0 ± 0.1	40.0 ± 1.1	15.5 ± 0.3	5.5 ± 0.1	31.0 ± 0.15	BD	0.4 ± 0.04
Fly ash 2 AT	0.3 ± 0.0	2.9 ± 0.1	5.1 ± 0.1	2.7 ± 0.2	0.9 ± 0.1	9.0 ± 0.01	BD	0.10 ± 0.01
PPP 6 BT	BD	0.8 ± 0.1	1.9 ± 0.05	12.0 ± 0.3	0.3 ± 0.2	0.4 ± 0.0	BD	BD
PPP 6 AT	BD	0.2 ± 0.05	2.6 ± 0.05	1.3 ± 0.05	0.5 ± 0.05	2.0 ± 0.01	BD	BD
Lurgi BT	BD	BD	4.6 ± 0.05	20.0 ± 0.2	0.6 ± 0.0	1.4 ± 0.01	1.0 ± 0.1	BD
Lurgi AT	BD	BD	2.8 ± 0.15	2.2 ± 0.2	0.6 ± 0.0	1.5 ± 0.01	BD	BD
U.S. EPA DWSt	0.01	0.05	5.0	0.05	—	1.0	0.05	0.01

*BT = before treatment; AT = after treatment; BD = below detection limit of 0.05 mg/l.

†U.S. Environmental Protection Agency Drinking Water Standards.

further research is needed to determine its cost and economic viability for field conditions.

CONCLUSIONS

Fly ash and spent shale samples were reacted under CO₂ pressure to reduce the concentrations of leachable inorganic contaminants. Moisture and reaction time were found to be important variables for the success of the CO₂ treatment. However, high pressure was not found to be necessary to lower the concentrations of leachable contaminants. Reacting samples at 40 psi of CO₂ (99.9%) for 120 h with 20% moisture precipitated calcite and lowered the pH from 12.3–12.8 to 8.8–9.7. The IAPs for treated samples suggested a close approach to saturation with respect to calcite. Additionally, CO₂ treatment was found effective in absorbing CO₂ as well as decreasing the leachability of certain inorganic contaminants. The method described in this study uses CO₂, which could be obtained from the combustion process itself (i.e. flue gas).

Acknowledgements—The authors extend their appreciation to the Department of Interior, U.S. Geological Survey, for funding this research through the Wyoming Water Resources Center and to the Central Energy Plant, the Laramie River Station and the Western Research Institute, Wyoming for providing the samples. We also thank James I. Drever, Department of Geology and Geophysics, University of Wyoming for reviewing the initial manuscript and Satyendra Sarmah and Rita Clark for their help in various aspects of the research.

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