Strength and Leaching Patterns of Heavy Metals from Ash-Amended Flowable Fill Monoliths

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Abstract Solidified flowable fill comprising of Type I portland cement, Class F fly ash, fine sand and water, is a porous monolith. In the case of excavatable fill, material mix proportions in the ash must be such that adequate but inexcessive strength is developed. For non-excavatable fill, maximization of fill strength is the primary objective. Furthermore, being that heavy metals are typically present in fly ash, physico-chemical interactions among mix components must mitigate against leaching out of metals. Herein, flowable fill monoliths containing class F fly ash in weight fractions of 0, 5, 10, 15, and 20, were subjected to unconfined compressive strength (UCS) tests and the American Nuclear Society's ANSI 16.1 leaching test, using de-ionized (DI) water and acidified water ($pH = 5.5$) as leachants. The results show that comprehensive strength is directly proportional to ash content, reaching 834kN/m² for excavatable fill and 3,753 kN/m2 for non-excavatable fill. The diffusion coefficients of arsenic (As) and selenium (Se) from samples decrease sharply with increase in ash content from 5% to 10% and stay relatively low at higher ash content. The leachability indices which are inversely proportional to the quantity of material leached, indicate that the effects of reduction in monolith internal permeability exceed the effects of increasing As and Se content introduced by higher ash content in the monoliths.

Keywords Leaching · flowable fill · fly ash

1 Introduction

Ash-amended flowable fill typically comprises sand aggregate, Portland cement, water, coal ash and possibly, some chemical additives in proportions that are selected for optimal strength, flowability, and permeability. These parameters are

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important with respect to the use of flowable fill which is frequently called controlled low strength material (CLSM) for utility and pipeline trenches and in pavement repair. In many circumstances, coal ash produced by electric power generating plants, is available in large quantities and at reasonable costs. When the source of ash is close to construction sites, there is usually interest in maximizing its use to reduce the cost of flowable fill projects. Several investigators (Inyang 2003; Sorvari 2003; Reijnders 2005) have devised decision support systems and reviewed methodologies on the use of waste materials such as ash in construction.

A critical constraint to the maximization of ash content in flowable fill is the possibility of an increase in the leachability of contaminants, especially heavy metals, in excessive concentrations, from ash concrete that contains a high weight fraction of ash. When solidified into ash concrete, flowable fill is a porous monolith in which the aggregate particles (sand) are held together by the cementing action of portland cement, coal ash and their reaction products. Generally, fly ash is used in flowable fills because of its high specific surface area relative to that of bottom ash, the largergrained coal combustion product. This high specific surface area is beneficial in solidstate chemical reactions. Compositionally, fly ash is known to be mostly glass (about 65–70%) formed as spherical particles with surficially attached heavy metals that are condensation products during coal combustion. Some heavy metals are also distributed more uniformly in the matrix of particles as described by Inyang (1992). Coal that is mined at fields in the eastern United States produces class F fly ash which has pozzolanic characteristics. The implication is that in a flowable fill mixture with portland cement, the fly ash reacts with lime that is produced during portland cement hydration reactions, to form cementitious compounds that account for strength development in the flowable fill monolith. In concrete, fly ash can be introduced as a partial replacement for either portland cement or fine aggregate. Usually, the fine aggregate is sand. If fly ash is introduced as partial replacement for portland cement, a net loss in strength may result. If it is replaced partially by fly ash, it essentially amounts to the replacement of a relatively inert material by a reactive one. Thus, monolith strength should be expected to increase as fly ash replaces sand as realized in experiments performed by Sahu and Piyo (2002). In that experiment, the confined compressive strength of sand slurry was found to increase as sand was replaced by fly ash. That observation is consistent with those of Siddique (2003) in which the compressive strength, splitting strength, and modulus of elasticity of concrete increased when fine aggregate was replaced by fly ash at the level of 50% by weight. For a particular ash concrete, the strength achieved depends on the type of fly ash, type of cement, water content and mix proportions of the components (ACI Committee 226 1987; Hurley et al. 1998). Several investigators, including Janardhanam et al. (1992), Gabr and Bowders (2000) and Subramaniam et al. (2005) have analyzed mix designs to assess the effects of fly ash on flowable fill concrete strength.

For facility maintenance considerations, it is most desirable to use flowable fill that is not excessively strong in backfilling of trenches for utilities and communication lines. This is because of the need to excavate the material to gain access to conduits, cables and equipment for repair. At the same time, the flowable fill must be strong enough to carry external (surface) loads and resist the borrowing activities of rodents. Low permeability which often correlates with high strength, is also desirable. ACI Committee 229 (1994), classifies flowable fill that has an unconfined compressive strength below $2{,}607 \text{ kN/m}^2$ (300 psi) as excavatable fill. Those that have unconfined compressive strength above $2,067 \text{ kN/m}^2$ (300 psi) are classified as non-excavatable fill. Nevertheless, with respect to excavation strength, levels below 689 kN/m2 (100 psi) are desirable.

Advances in solidification/stabilization (S/S) of waste during the past 20 years have provided some insights to the relationship among strength development, porosity evolution and other transport parameters in cemented systems. A review of fundamental processes of cement solidification and stabilization has been published by Glasser (1997). Therein, there is an acknowledgement that the introduction of fly ash into cemented systems can reduce the connectivity among pores to the extent that permeabilities lower than 10^{-12} m/s can be achieved. However, the generation of cracks during hydration processes can enhance the transport of chemical species in such systems. As noted for cement-stabilized wastes (Gougar et al. 1996; Rosetti and Medici 1995; Park 2000; Asavapisit et al. 1997), heavy metals from fly ash can also enter the structure of evolving chemical phases such as ettringite, during the hydration of cement. Apart from structural inclusions, heavy metals can conceivably precipitate out of internal pore fluids of solidified concrete, chemisorb on other particles out of internal pore fluids of solidified concrete, and other particles in the concrete matrix, react with other constituents to form new compounds, or diffuse out of the concrete matrix under suitable hydraulic and physico-chemical conditions.

Heavy metal diffusion from monolithic systems is a critical phenomenon in the assessment of contaminant leachability and has been analyzed by many researchers, exemplified by Batchelor (1990, 1992, 1998), Batchelor and Park (1998), Van der Sloot (2002), Kim and Batchelor (2001), Inyang et al. (2003), Ogunro and Inyang (2004), Bai et al. (1996), and Poon and Chen (1999). The migration of contaminants through wastes is hindered by intergranular cementation (Reddi and Inyang 2000; Naik et al. 2001). Where portland cement is used, alkaline conditions that can produce the precipitation of some metal from pore fluid can be generated (Cote et al. 1986).

2 Experimentation

In order to evaluate the extent to which the incorporation of Class F fly ash affects both the unconfined compressive strength and contaminant leachability from monolithic samples of flowable fill, experiments were performed using fly ash contents of 0%, 5%, 10%,15%, and 20% by weight. The other components that were used in the flowable fill mixtures are portland cement (Type 1), fine sand, water and admixture (Darafill Dry). Increase in fly ash content in the mixes was made through reductions in fine aggregate content in the mixes without decrease in cement content.

3 Sources of Ash and Concrete Materials

During the preliminary stages of this research, the materials needed for the mix design of the fill were procured from various companies. A 55-gallon capacity drum of dry fly ash was supplied by Duke Energy (Charlotte, NC) from its Marshall Station. The results of the XRF analysis of the ash used in this research are presented in Table 1 for the elemental composition and Table 2 for the normalized oxide composition. The ash conforms to ASTM C 618 specifications of coal combustible by-products. Lafarge Type 1 cement was supplied by Concrete Supply Co. (Charlotte, NC). This cement conforms to ASTM C 150 specifications. This cement is used commonly in ready mix concrete, and has a specific gravity of 3.15. Hanson Sand that meets ASTM C-33 specifications was supplied by Concrete Supply Co. (Charlotte, NC). Darafill Dry is an additive used in controlled low strength concrete per ASTM C 494. It was supplied by Grace Materials of Charlotte, NC. The moisture content of the sand was determined using the Marshall test. In this test, $400 g$ of wet sand is subjected to electric drying for nearly 10 min. Once the sand is dry, it is weighed. Then, from the initial and final weights of the sand, the moisture content is determined.

4 Mix Design and Flowable Fill

The mix design of the flowable fill was based on South Carolina Department of Transportation Specifications. These specifications are commonly used in projects in North Carolina from where the ash samples were taken. The air content of the mixes was assumed to be about 15%, and the initial water content was 0.85. Water content was gradually increased beyond 0.85 as ash content was increased to maintain good workability. During initial testing, mixes were designed and tested for

Trace metal	Concentration (wt $\%$)	Trace metal	Concentration (wt $\%$)
Si	52.90	Cu	0.03
Al	15.40	Zn	0.02
Fe	3.90	Rb	0.02
K	2.95	Y	0.02
Ca	1.13	Mn	0.02
Ti	1.04	Co	0.01
Mg	0.54	Ga	0.01
S	0.40	Mo	ND
Na	0.20	Hg	ND
Sr	0.09	C _d	0.00
Cr	0.08	Se	0.00
P	0.08	As	0.00
Zr	0.07	Pb	0.01
V	0.04		

Table 1 XRF analysis on the ash used in the research

Metal oxide	Concentration (wt %)
SiO ₂	53.20
AI ₂ O ₃	25.50
Fe_2O_3	7.74
K_2 O	4.26
SO ₃	2.50
CaO	2.34
TiO ₂	2.25
MgO	0.75
Na ₂ O	0.26
P_2O_5	0.26
BaO	0.19
SrO	0.17
Cr_2O_3	0.15
V_2O_5	0.09
ZrO ₂	0.07
CuO	0.07
ZnO	0.04
Y_2O_3	0.03
Rb, O	0.03
MnO	0.03
CoO	0.02
Ga ₂ O ₃	0.02

Table 2 Normalized metal oxide composition of the ash used in the research

Table 3 Mix design of ash flowable fill for excavatable fill mix

Material content	Percent of fly ash				
	0%	5%	10%	15%	20%
Cement $(kg/m3)$	61	61	61	61	61
Fly ash (kg/m^3)	$_{0}$	93	186	340	463
Sand (kg/m^3)	1,699	1,580	1,418	1,337	1,319
Water $(kg/m3)$	216	247	279	370	341

strength and suitability for use in subsequent leachability tests. The trial-strength of fill selected was 248 kN/m² which conforms to the ACI 229-R94 excavatable flowable fill strength criteria. However, the mix was developed by increasing the cement content significantly to satisfy not only the field strength criterion but also monolith strength for leaching. Mix designs for both excavatable and non-excavatable fill are shown in Tables 3 and 4 respectively.

5 Strength Tests and their Protocols

The standard proctor test which is a compaction test, was used to determine the dry density and optimum moisture content of the ash. The test meets ASTM D 698 criteria. To perform this test, 2 kg of ash was put in a tray and water was added at the level of 5% by mass. The fly ash absorbed all the water, and after mixing, it still remained as dry powder. Then, 5% more water was added and mixed. The prepared sample was then paced in layers into the compaction mold and compacted 25 blows of the hammer per layer. The sample was then trimmed above the mold, and the weight of the sample with mold was recorded. A portion of compacted ash was removed from the mold and then the procedure was repeated with various moisture contents up to 40%. A plot was drawn to relate moisture content of ash and the dry density calculated for each moisture content. This enabled the maximum dry density and optimum moisture to be established. The maximum dry density of the Duke ash is $1,212\,\text{kg/m}^3$ (75 pcf), and its optimum optimum moisture content is 28%. Based on the grain size distribution curve (Fig. 1), the ash is classified as a silty material, however the result of the compaction test revealed that the maximum dry density of the ash is lower than most natural soils.

The comprehensive strength test was performed in consistence with ASTM D 4832 standards. The sample was mounted on the rubber pad which in turn, was mounted on the load pad. Then, another rubber pad was placed on the top of the sample and the set-up was joggled to be in contact with the load cell. An initial load of 68.9 kN/m² (10 psi) was applied. Then, gradually, load was increased at the constant strain rate of 4.3×10^{-3} m/s (0.02 in./min) until each sample crumbled and broke in compression.

Fig. 1 Combined grain size distribution of the ash tested using both the sieving method and hydrometer method (square shaped points represent sieve analysis and triangle shaped points represent hydrometer analysis)

6 Leaching Tests and Protocols

Monolith leaching tests were performed largely in accordance with American Nuclear Society's ANSI 16.1 test method. Two leachants: de-ionized water and pH5.5 solutions were used. The de-ionized water simulated a leachant of neutral pH while the pH 5.5 leachant simulated mildly aggressive acid rain. The leachant was prepared using concentrated HNO_3 and de-ionized water. Specifically, 3.16 \times 10⁻⁶ $M HNO₃$ was transferred into a 15 gallon tank filled with de-ionized water. The leachant volume required to perform the leaching was calculated to be ten times the surface area of the sample. So, for a 2- in. (0.05 m) cubic sample, the required leachant volume was 1,500 ml. The leaching vessel was filled with the estimated leachant and the sample was placed in the vessel to undergo the leaching process. The starting time was noted and after 2 h the leachate was emptied, and a small portion was collected in test tubes for further analyses. Then, the same amount of fresh leachant was transferred into the leaching vessel. The leachant was renewed at 7, 24, 48, 72, 96, 124, 168, and 336 h, and at these times, the leachate was also sampled in the earlier fashion (Fig. 2). The sampled leachate was used in the estimation of the concentrations of the trace elements: arsenic and selenium. Although these two metals are typically found in many ash samples, they were not targeted in this research for regulatory compliance assessment.

Each concentration was determined using the Atomic Absorption Spectrometer (AAS). The process includes standard solution preparation, calibration, and analysis. Based on the absorbance of target elements (As, Se) from the AAS cook book (user's guide), three standards were prepared. The standard tubes were mounted on the standard platform of the AAS. Then, 10 ml samples of leachate were transferred into AAS tubes and placed in trays. The lamps of target elements were mounted onto the lamp chamber and the respective element lamp was turned on in the calibration settings. Calibration was repeated until a smooth calibration curve was

 (A) = Leaching vessel; (B) = Leachant; (C) = Monolith Sample; (D) = Support system; (E) = Leachant drained

Fig. 2 Leachant renewal cycle of monolith leaching test

observed. After obtaining the calibration, the analysis button was pressed. The concentration of the target element was then observed. From, the initial observations, it was concluded that the concentration levels of the target elements were too low and undetectable at the sensitivity of the AAS used. Hence, the samples were than spiked with a known higher concentration of each target element and tested in the fashion described earlier. The metals were then detectable. To confirm the latter approach, the samples were tested using a highly sensitive graphite furnace which detects even parts per billion (ppb) levels. The results from both types of tests indicate that the difference was insignificant.

7 Analysis and Discussion of Results

Effects of ash content on monolith strength: The strength of the excavatable fill mixes are 184, 461, 517, 754, and 834 kN/m2 for 0%, 5%, 10%, 15% and 20% mixes, respectively (Fig. 3). In the case of non-excavatable fill, the compressive strengths of 10%, 15%, and 20% mixes are 2,172, 2,952, and 3,753 kN/m2 respectively (Fig. 4).

Fig. 3 Compressive strength of excavatable flowable fill

Fig. 4 Compressive strength of non-excavatable flowable fill

Compressive strength increases with increase in ash content. The primary reason for the observed increase in strength is the presence of pozzolanic materials such as silica (53.2%), alumina (25.5%) and calcium compounds (2.34%) in the ash. These materials react with the tricalcium-alumino silicates present in the cement, thereby increasing the strength of the monoliths.

The water content required is directly proportional to the ash content in the mix. The water content demand increases for excavatable fill as ash contents increase from 0% to 5%, 5% to 10%, and 10% to 15%. However, from 15% to 20% ash content, the water demand decreases. A similar trend of increase in water content with increase in ash content was observed in the case of non-excavatable fill. Also, flowability increases with increase in water content. The strength of each mix was found to increase with ash content when the cement content is kept constant. This is due to the fact that pozzolanic activity is enhanced with increase in ash content, resulting in increased strength.

8 Effects of Ash Substitution on Contaminant Leachability Index

The leaching/diffusion test results for arsenic with both water and pH5.5 leachants for a given percentage of ash in excavatable fill mix are shown in Fig. 5. The sample diffusion coefficient (D_{α}) for arsenic for 5% excavatable fill mix is higher than those of other mixes for both pH5.5 and de-ionized water leaching. The highest recorded D_{es} is 5.00 × 10⁻⁴ m²/s (pH5.5 leaching). As the ash content in the mix is increased, the diffusion coefficient decreases. The mass leached from the sample

Fig. 5 Test result showing the relationship among fly ash content, arsenic diffusion coefficient and compressive strength of excavatable fill material

Fly ash percent $(\%)$		Mass of arsenic leached Leachant pH from sample(mg)	Sample leachability index
5% excavatable fill	DI	0.01	13.4
	pH5.5	0.01	13.3
10% excavatable fill	DI	0.01	14.0
	pH5.5	0.01	14.0
15% excavatable fill	DI	0.01	14.7
	pH5.5	0.01	14.8
20% excavatable fill	DI	0.02	14.4
	pH5.5	0.01	15.0
10% Non-excavatable fill	DI	0.01	14.5
	pH5.5	0.01	14.5
15% Non-excavatable fill	DI	0.01	14.8
	pH5.5	0.01	14.9
20% Non-Excavatable Fill	DI	0.01	14.9
	pH5.5	0.01	15.2

Table 5 Mass of arsenic leached from the sample and its leachability index with respect to ash percent and leachant quality

and the sample leachability index of arsenic are given in Table 5. In the case of excavatable fill with de-ionized water leachant, the mass of arsenic released, and leachability index for 5%, 10%, 15%, and 20% mixes are 0.0121, 0.0132, 0.0111, 0.0194 mg; and 13.2, 14.04, 14.66, 14.43, respectively (Tables shows rounded values). In the case of excavatable fill, for pH5.5 leachant, the mass of arsenic released and the leachability index with 5%, 10%, 15% and 20% mixes are 0.0138, 0.0131, 0.010, 0.010 mg; and 13.3, 14.04, 14.75, 14.99 respectively (Tables shows rounded values). The mass released from the sample is greater with de-ionised water leaching than with pH5.5 leaching except for 5 percent excavatable mix. This may be due to temporal changes in leachant chemistry and structural properties of the fill material. Arsenic may have leached more in the alkaline pH range than in the acidic range. In the case of non-excavatable fill, the diffusion coefficient (D_{∞}) of arsenic is observed at 10% ash content with pH5.5 leaching and de-ionised water leaching. It decreases with increase in the ash content up to 20% ash content (Fig. 6).

A similar trend concerning the relationship between selenium diffusion coefficient and ash content is shown in Fig. 7. The sample diffusion coefficient (D) for selenium of 5% excavatable mix is higher than those of other mixes in the cases of both pH5.5 leaching and de-ionised water leaching. The highest recorded D_{n} is 1.4 \times 10−14 m2 /s (pH5.5 leaching). As the ash content mix was increases, the diffusion coefficient decreases. A similar trend is also observed in the case of selenium leaching from non-excavatable fill mix, but the diffusion coefficients of non-excavatable fill are low in comparison to those of excavatable fill (Fig. 8). The range of selenium diffusion coefficients for excavatable fill is 2.59×10^{-14} m²/s – 1.42×10^{-12} m²/s. The D_{es} values of pH5.5 leaching for 10%, 15%, and 20% mixes are 3.0 × 10⁻¹⁵ m²/s, 1.0 ×10⁻¹⁵ m²/s, and 6.9 ×10⁻¹⁹ m²/s respectively. The mass leached from each sample and the sample leachability index of selenium are given in Table 6.

Fig. 6 Test result showing the relationship among fly ash content, arsenic diffusion coefficient and compressive strength of non-excavatable fill material

Fig. 7 Test result showing the relationship among fly ash content, selenium diffusion coefficient and compressive strength of excavatable fill material

The mass of selenium leached form the mixes vary from 0.0321 to 0.08 mg. The leachability index range is 11.85–13.49. Except for an ash content of 15%, pH5.5 produces higher leached quantities of selenium than de-ionised water.

9 Conclusions

The results of this research lead to the following conclusions. Increase in fly ash content results in increase in the compressive strength of both excavatable and nonexcavatable flowable fill monoliths. For both de-ionised water and pH5.5 leaching,

Fig. 8 Test result showing the relationship among fly ash content, selenium diffusion coefficient and compressive strength of non-excavatable fill material

Fly ash percent $(\%)$	Leachant pH	Mass of selenium leached from sample (mg)	Sample leachability index
5% Excavatable fill	DI	0.04	12.2
	pH5.5	0.05	11.9
10% excavatable fill	DI	0.04	12.8
	pH5.5	0.05	12.5
15% excavatable fill	DI	0.04	13.2
	pH5.5	0.04	13.4
20% excavatable fill	DI	0.08	13.0
	pH5.5	0.08	12.9
10% Non-excavatable fill	DI	0.04	13.0
	pH5.5	0.05	12.8
15% Non-excavatable fill	DI	0.03	13.6
	pH5.5	0.04	13.5
20% Non-excavatable fill	DI	0.05	13.4
	pH5.5	0.05	13.4

Table 6 Mass of selenium leached from the sample and its leachability index with respect to ash percent and leachant quality

increase in fly ash content of monoliths tends to suppress the diffusion of arsenic and selenium dramatically until an ash content of about 10% by weight. Although leachant acidity enhances the leaching of both metals, the effects tend to become insignificant as the content of fly ash increases in the monoliths. Perhaps, the buffering effects of fly ash constituents neutralize leachant acidity at higher ash content.

Undoubtedly, increases in fly ash content in the monoliths introduce higher quantities of both As, Se into the monoliths such that higher concentrations of these metals are available for leaching. However, the intensification of pozzolanic

activity in the monolith, which should be proportional to fly ash content, may retard the transport of the metals, thereby reducing their leaching rate.

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