



## Evaluation of leaching of trace metals from concrete amended with cement kiln baghouse filter dust



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### ABSTRACT

The production of Portland cement (PC) can result in the release of trace amounts of mercury (Hg) in stack emissions. As a means of controlling Hg stack emissions at Portland cement kiln operations, some facilities have instituted a process known as dust shuttling, where baghouse filter dust (BFD), a partially calcined byproduct, is collected and blended with PC as an inorganic process addition, instead of returning the material to the kiln pyro-process. In response to questions raised regarding environmental impacts of recycling BFD into concrete products, research was performed to investigate the chemical composition of BFD and to assess the potential for leaching of trace metals from concrete products amended with BFD. Leaching of monolithic concrete under submerged conditions was examined as a function of time; concentrations of detected elements were found to be below applicable risk thresholds. Despite elevated Hg in the BFD relative to the PC, no elevated Hg risk was observed. Leachates from raw BFD did exceed risk-based thresholds for Se and V. However, leaching of size-reduced BFD-amended concrete did not show concentrations of these elements above applicable risk based thresholds. The results of this study suggest that the use of BFD as an inorganic process addition, within the percentages currently allowed by US industry standards, does not pose any additional risk with respect to direct human hazard or leaching of trace metals to water supplies.

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### 1. Introduction

The production of Portland cement (PC) requires a number of air pollution control (APC) technologies to limit emissions and capture particulate matter (PM) during the manufacturing process. Baghouses, as well as other APC devices, are used at locations within the operation to collect dust particles generated during the production of PC. In some cases this material, frequently referred to as cement kiln dust (CKD), is extracted from the system as a waste product as the buildup of alkali salts can reduce final cement product quality (Shoaib et al., 2000). In other cases, the recovered PM is of sufficient quality to be recycled as a mineral additive in the PC manufacturing process or through direct incorporation into the PC (Sikkema et al., 2011); the latter practice is considered to be an inorganic process addition (IPA). Within the United States, the standard specifications for Type I PC (ASTM C150/AASHTO M85)

allow for the use of an IPA up to 5% by mass, as long as the final product specifications are met (ASTM, 2013a,b; AASHTO, 2011). Materials typically used as IPA include limestone, fly ash, bottom ash, slag, cement kiln feed, CKD, and other calcined materials (Taylor, 2008). The partially calcined byproducts collected in a cement kiln baghouse and subsequently recycled are referred to baghouse filter dust (BFD) throughout this study, and differ from CKD which is a more general term for a waste product purposely removed from the cement manufacturing process. BFD can be distinguished from CKD because of the lower content of alkali salts within the material, allowing for its use as an IPA; trace element content of BFD has not yet been reported in the literature, and producing a detailed data set is one objective of the study presented herein.

The promulgation of more stringent air emissions regulations for cement kilns within the US (US-EPA, 2010) has driven a number of kilns to implement modified operational strategies. As a means of controlling mercury (Hg) emissions from cement kiln operations, some facilities have proposed or instituted dust shuttling, a practice that involves the blending of a fixed percentage BFD into the PC for use as an IPA (Sikkema et al., 2011; Wang et al., 2014).

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Since Hg is known to partition into a cement plant's BFD (Mlakar et al., 2010), the removal of BFD through use as an IPA provides a mechanism to "bleed" off Hg out of the cement manufacturing operation and thus reduce air emissions. The potential for release of Hg into the environment as a result of products amended with BFD, however, must be addressed. The impacts of the vapor phase release of Hg from plants that have implemented dust shuttling were investigated by the authors in another study (Wang et al., 2014). Previous research has indicated that additional trace metals (As, Ba, Cd, Cr, Pb, Se, V, and Zn) are found in the PM generated by cement kilns, (Haynes and Kramer, 1982; PCA, 1992) and since BFD is produced in a similar manner to CKD, the evaluation of the effects of dust shuttling on the total and leachable concentrations of these trace metals is warranted.

While CKD characterization data are available, both on the raw material (Haynes and Kramer, 1982; PCA, 1992) and on CKD used in mortar and concrete (Siddique, 2006; Kunal et al., 2012), information on the chemical constituents found specifically in BFD used as an IPA are not common; the influences of dust shuttling on the concentrations of trace metals in PM from cement manufacturing are not well reported. The objective of this study is to address the current lack of data with regard to the impact of dust shuttling on the total and leachable concentrations of trace metals in BFD, as well as in Portland cement concrete (PCC) where BFD is used as an IPA. Two previously conducted nationwide (US) surveys characterizing the concentrations of trace elements in CKD and PC (Haynes and Kramer, 1982; PCA, 1992) are used as a benchmark for comparison to the data generated in this study. Bulk and trace elemental concentrations of BFD from a full-scale facility practicing dust shuttling were measured, and a suite of leaching tests were used to assess trace elemental release of the raw material, PC-BFD blends, and PCC products amended with BFD. These results were then compared to risk-based thresholds to determine if measured concentrations might pose a potential risk to human or environmental health under normal use scenarios (the concrete product itself and the crushed concrete recycled as a construction material).

## 2. Materials and methods

### 2.1. Sample collection, processing, and laboratory specimen creation

BFD and PC samples were collected from a cement production facility located in Florida, USA. The kiln incorporated a precalciner dry-process system. Samples were collected from the cement kiln over an 11-month period. Samples of BFD and PC from the facility were collected in five-gallon High Density Polyethylene (HDPE) buckets. Four grab samples of BFD and one PC sample were collected: BFD Sample A (collected Oct. 28, 2011), BFD Sample B (collected Dec. 15, 2011), BFD Sample C (collected Feb. 28, 2012), BFD Sample D (collected Aug. 24, 2012), and Cement Sample A (collected Aug. 24, 2012). Prior to use in any experimentation, the materials were homogenized by rotating the storage vessels in an end-over-end fashion for approximately 2 h (Analytical Testing Corporation, DC-20B rotator).

Recent harmonization of cement specifications (AASHTO M85 and ASTM C150) has led to a maximum allowable addition of IPAs (5% by mass) in Type I PC (ASTM, 2013a, 2013b; AASHTO, 2011). A National Cooperative Highway Research Program (NCHRP) study has precluded the use of more than an 8% by mass addition of CKD, due to Loss-On-Ignition (LOI) and insoluble residue limitations (Taylor, 2008). Taking this into account, two PC and BFD blends were created; a blend at 5% BFD and 95% PC by mass and a blend at 10% BFD and 90% PC by mass. These blends represented the maximum allowable concentration of BFD as an IPA in Type I PC, as well as a blend that would serve as a conservative overestimate.

The BDF-PC blends were mixed in a similar fashion to the collected samples prior to use.

Coarse and fine aggregate used to create the PCC specimens were purchased from local suppliers and conformed to ASTM C33 in all concrete mixes. The fine aggregate utilized consisted of silica sand and the coarse aggregate was limestone. All aggregate and cement were characterized for trace metals before use. After batching, PCC specimens were placed in rectangular molds (10.16 cm × 10.16 cm × 25.4 cm), wrapped in plastic sheeting to prevent moisture loss and cured at a temperature of  $23 \pm 2^\circ\text{C}$  per ASTM C192; PCC samples were cured for a period of 24 h. Although samples were cured for 24 h, it should be noted that concrete has been demonstrated to become less permeable as the hydration process continues: because this experiment was designed to assess the leachability of trace metals from BFD, a 24 h curing period was used as a conservative estimate of a real world scenario. Three PCC specimens of each of the following groups were created; Set A (10% BFD-90% PCC), Set B (5% BFD-95% PCC), and a control set which contained no BFD. All mix design parameters were maintained constant with the exception of the BFD additions and the PC it replaced. The cement content, water to cement ratio, and coarse and fine aggregate additions were  $467\text{ kg/m}^3$ , 0.53,  $827\text{ kg/m}^3$ , and  $661\text{ kg/m}^3$ , respectively. Following the initial 24-h curing period, a portion of the PCC was then size reduced with a jaw crusher to pass a 9.5 mm sieve, in accordance with the maximum particle size requirements outlined in EPA method 1312 (US-EPA, 2007). The size-reduced sample then was used in all batch tests with no further particle size modification. The remaining PCC samples were kept intact for use in monolithic leaching experiments.

### 2.2. Elemental analysis

Bulk elemental analysis was performed using Scanning Electron Microscopy – Energy Dispersive X-Ray Spectroscopy (SEM-EDS) (JEOL SEM-6400) and X-Ray Diffraction Spectroscopy (XRD) (Philips XPert MRD); the detection limit for the SEM-EDS was 0.1% mass for all elements. Bulk elemental analysis was conducted to verify that the total elemental concentration and mineral forms contained in the BFD were similar to CKD, however it should be noted that this analysis serves only as a semi-quantitative tool for elemental and mineralogical characterization. Total environmentally available (TEA) concentrations of trace metals were determined in accordance with EPA Method 3050b (US-EPA, 2007). To obtain the powdered sample required for analysis (Method 3050b) the material was first size reduced using a jaw crusher, and then ground to powder in a ball mill. Following this process the entirety of this material was able to pass a US #10 sieve (2 mm). The sample was then mixed and three aliquots were digested for each of the samples tested. The samples were exposed to multiple additions of nitric acid (15 mL  $\text{HNO}_3$ ), hydrogen peroxide (5 mL of 30%  $\text{H}_2\text{O}_2$ ), and hydrochloric acid (10 mL HCL) while being heated at a temperature of  $95 \pm 5^\circ\text{C}$ . The remaining solid sample was then removed using filter paper (Whatman 41) and the liquid sample diluted to a volume of 100 mL. Digestates were analyzed for trace metals using Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES) (Thermo-Jarrell Ash Trace 61E).

The digestion procedure to determine Hg speciation was derived from the sequential extraction procedure outlined in EPA Method 3200 (US-EPA, 2005); total Hg concentration was conducted in accordance with EPA Method 7474 (US-EPA, 2007). Five replicates (0.5 g) of each material type (PC, BFD, fine aggregate, and coarse aggregate) were weighed using an analytical balance with a precision of 0.01 mg and placed in polytetrafluoroethylene (PTFE) vials. Ten milliliters of the appropriate extraction solution for each of the Hg species were then added to the PTFE vials. The vials were then capped and heated to  $95 \pm 5^\circ\text{C}$  for a period 30 min in a microwave

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