International Journal of Concrete Structures and Materials
Vol.7, No.2, pp.111–117, June 2013
DOI 10.1007/s40069-013-0042-3
ISSN 1976-0485 / eISSN 2234-1315

Use of Heavy Oil Fly Ash as a Color Ingredient in Cement Mortar

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(Received July 8, 2012, Accepted April 4, 2013)

Abstract: Heavy oil fly ash (HOFA) is a byproduct generated by the burning of heavy fuel oil. Chemical analysis showed that HOFA is mainly composed of unburned carbon with a significant amount of heavy metals. Due to toxicity, management of this waste poses a challenge to the industry personal. The present study investigates the possible use of HOFA as a black pigment or admixture in cement mortar aiming to produce ornamental brick. In order to investigate the change of cement mortar strength when HOFA is added, the standard compressive strength test with 50 mm cubes was performed. The results showed that the addition of 2–5 % of HOFA in cement mortar does not affect its strength. The leaching behavior of trace elements within HOFA and HOFA mixed mortar were investigated through laboratory batch leaching experiments. The results confirmed that HOFA can be utilized as a black pigment in ornamental brick, which is environmentally safe and provides good balance between color and brick properties.

Keywords: HOFA, reuse, ornamental brick.

1. Introduction

According to ASTM C618-08 fly ashes are distinguished in two classes (F and C). Class F ash is usually produced by burning anthracite or bituminous coal while Class C fly ash is normally produced by burning sub bituminous coal or lignite. The main criterion for the above classification is the sum of silicon dioxide (SiO_2), aluminium oxide (Al_2O_3) and ferric oxide (Fe_2O_3) which in Class F must be at least 70 % while in Class C must be higher than 50 %. Class F is rarely cementations when mixed with water alone possessing pozzolanic properties. Class F fly ash requires a cementing agent, such as Portland cement, hydrated lime, with the presence of water in order to react and produce cementitious compounds. Unlike Class F, Class C fly ash exhibit usually cementations in addition to pozzolanic properties, it does not require an activator. In the presence of water, Class C fly ash will harden and gain strength over time. The suitability for the use of fly ash in construction sector is determined based on certain criteria, such as the content of unburned carbon, reactive silica and alumina, sulfur trioxide levels and fineness. In recent years, the recycling of fly ash has become an increasing concern due to increasing landfill costs, strengthening environmental regulations, and current interest in sustainable development (Mofarrah and Husain 2013). Many applications have been developed for coal fly ash as a cement replacement material in concrete (Qian and Shi 2003; Collins and Jensen 1995). However due to different characteristics properties such application is uncommon for heavy oil fly ash (HOFA). The fly ash from heavy oil burning has a higher percentage of unburned carbon, less silica content, and a higher level of vanadium, nickel, and magnesium than coal fired fly ash. Due to higher proportion of unburned carbon and less silica content, HOFA does not acknowledge the good cementations property as coal fly ash. However, there are significant amount of HOFA is producing around the world that needs to me managed.

Burning heavy fuel oil yields about 3 kg of ash per kiloliter of oil (Tsai and Tsai 1997) and most of the ash (~90 %) is fly ash (FA), which is collected by ESPs or cyclones for final disposal or reuse (Hsieh and Tsai 2003). HOFA consists of inorganic substances such as SiO_2, iron oxide (Fe_2O_3), aluminum oxide (Al_2O_3) and 70–80 % unburned carbon (Kwon et al. 2005). It also contains heavy metals such as arsenic (As), cadmium (Cd), mercury (Hg), copper (Cu), vanadium (V) and nickel (Ni) which exist in the fuel at the outset, but these levels increase during burning (Mofarrah et al. 2012; Hwang et al. 1996). The bulk density of HOFA varies from 0.50 to 1.50 g/cm and its true density and porosity are reported as 2.15 g/cm and 10.31 % respectively (Kwon et al. 2005). Research of the characteristics and possible applications of heavy oil FA is still insufficient in the scientific literature. Many studies have focused on the recovery of vanadium and nickel from HOFA (Fytianos et al. 1998; Choi et al. 2002); however, after recovery the major part of FA (about 90–95 %) is dumped into landfills (Akita et al. 1995; Miura et al. 2001).

Although Canada has reduced the use of heavy fuel oil for power generation, in some parts of Canada the power industry still uses heavy fuel oil or a mixtures of heavy fuel oil and petroleum coke. According to Statistics Canada,

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Newfoundland and Labrador uses heavy fuel oil, or a mixture of heavy fuel oil and petroleum coke, to meet 3.4 % of its power needs, while Nova Scotia and Prince Edward Island uses 15.8 % and New Brunswick uses 36.1 % (Paul and Caouette 2007). This indicates a huge amount of HOFA is being generated by provincial power industries.

The current disposal practice of HOFA follows either dry or wet disposal procedures. For the dry disposal method, HOFA is transported by truck or conveyor from the power plant to the site of disposal. In case of wet disposal, HOFA is transported as slurry through pipe and disposed of into lagoons. Land disposal of HOFA could results air, soil and water pollution by wind erosion, seepage, or run off from landfills into soil. Indeed, poor management and uncontrolled disposal of HOFA may cause a dispersion of fine particles containing pollutants which are health hazard (Fernandez et al. 2003; Mohapatra and Rao 2001). For example, exposure to HOFA can result in coughing, bronchial irritation, and gastrointestinal diseases (Jiang et al. 2000; Andrew et al. 2002). HOFA forms an acidic solution on contact with water. Skin irritation may occur on contact with the HOFA. Handle, transport, and disposal of HOFA have also negative effects on environment and human health.

The objective of this study is to investigate the use of HOFA as a black pigment or admixture in cement mortar aiming to use in ornamental brick. Analysis of chemical and physical properties of HOFA also conducted in this study.

2. Materials and Methods

For this study, FA was collected from the electrostatic precipitator (ESP) at a power plant in New Brunswick, Canada. This power plant uses a mixture of petroleum coke, and heavy fuel oil at different percentage. The collected sample was produced from mixture of petroleum coke and 2 % heavy fuel oil. The physical properties of the sample, such as bulk density, specific gravity, and moisture content were analyzed following the testing method described by Wesche et al. (1989).

2.1 Quantitative Chemical Analysis

Quantitative chemical analysis was performed to accurately determine the concentration of the major elements in HOFA. To determine the trace element concentrations in HOFA, an inductively coupled plasma-mass atomic spectrometry (ICP-MS) examination was used. A combination of bulk HNO_3, and then Aqua regia (a mixture of HNO_3 and HCl at 1:3 ratios) was used for digestion. ICP-MS measurements were performed using a Perkin-Elmer DV instrument.

2.2 X-ray Diffraction Studies

X-ray diffraction was used to study the crystallographic structure and mineral composition of HOFA sample. In all cases, samples were analyzed using the powder X-ray diffraction technique. An Ultima IV X-ray diffractometer by Rigaku Americas Corporation operating at 40 kV and 44 mA was used for these measurements. The detector was scanned over a range of 2θ angles from 5° to 75°, at a step size of 0.02° and a dwell time of 2 s per step. The resulting powder diffraction patterns were analyzed utilizing JADE version 9 software developed by Materials Data, Inc. (MDI).

2.3 Scanning Electron Microscopy Analysis

The scanning electron microscopy (SEM) analyses were conducted with a quartz PIC image measurement system to observe the microstructure of the samples. Samples were mounted in epoxy resin and the surfaces were ground flat by 600 grit abrasive paper. The samples were then polished to achieve a smooth surface. The polished samples were placed into a vacuum and etched with argon gas (Ar) for 20 min. The microstructures of the sample were examined with SEM and photographs were taken.

2.4 Particle Size Distribution

Small amount of HOFA sample was placed into a laboratory centrifuge tube. The tube was then filled up with ~40 ml of 0.05 % $NaPO_3$ solution. After hand shaking, it was placed into a tray in a Branson ultrasonic bath model 5510 for overnight. The particle size of the prepared HOFA sample was measured with a HORBIA PARTICA laser scattering particles size analyzer model LA-950 using the wet dispersion method in $NaPO_3$ solution.

2.5 Batch Leaching Test

The main objective of batch leaching test (BLT) is to investigate the leaching behavior of toxic compounds in the concrete made with HOFA. The results of BLT will help in assessing the human health and environmental risks. Many leaching test protocols such as toxicity characteristic leaching procedure (TCLP), equilibrium leach test (ELT), acid neutralization capacity (ANC), have been cited in literature (USEPA 1989; ECAEC 1986; Scott et al. 2005). These tests are developed to simulate the leaching processes of waste materials in landfills or other disposal scenarios to evaluate potential risks to humans and/or ecosystem. TCLP developed by the United States Environmental Protection Agency (USEPA 1989), is widely used to classify hazardous solid wastes and evaluate the worst leaching conditions in a landfill. ANC and ELT are designed to simulate the leaching characteristics caused by precipitation on improperly designed landfills (ECAEC 1986). ELT is meant to evaluate the maximum leachate concentration under mild conditions while ANC uses acidic solutions to evaluate the leachate (ECAEC 1986). In order to evaluate the possible environmental effects of HOFA use as a color ingredient in concrete, this study performed ANC batch leaching tests (BLT) on the prepared color concrete.

Ornamental concrete can be used in landscape, which is ideal for driveways, walkways, patios, planters and retaining walls, etc. Since HOFA contained significant amount of leachable metals, the use of HOFA mixed concrete in landscape may leach toxic elements into the environment through rainwater or acid rain. Therefore, the result of leaching test has an important role in the use HOFA mixed concrete in landscape. The recent study Mofarrah et al.

(2012) revealed that most of the toxic elements in HOFA showed higher leaching values with lower pH level of the leaching solution. In order to evaluate the maximum leachate concentration, this study used ANC method using HNO_3 solutions (pH 3.0) as leaching media. All test apparatus and glasses were cleaned with an acid solution (e.g., 15 % HNO_3 solution) and rinsed using distilled water prior to the leaching experiments. The batch extraction procedures and liquid: solid (L/S) mixing ratio is shown in Table 1. To ensure continuous stirring of the samples, Phipps & Bird Stirrer model 7790-400 with 10 rpm was used over the test period. The mixtures from the above experiments were filtered through a 0.45 μm filter paper, and acidified with a nitric acid solution to make pH <2 for the chemical analyses.

2.6 Preparation of Concrete Samples

In order to investigate the change of the compressive strength of cement mortar when HOFA is added as a color ingredient, 50 mm cubes were prepared by mixing of Portland cement, sand and HOFA at different ratios according to the standard method (i.e., ASTM C109). The proportions of materials for the standard cubes were mixed as one part of cement to 2.75 parts of graded standard sand by weight. The HOFA was mixed to the mortar at 0 % (reference), 2, 5, 10, and 20 % by weight. The water cement ratio was maintained of 0.45 for all cases. Five groups (4 in each group) total 25 of cube samples were prepared namely: OC_0, OC_2, OC_5, OC_{10} and OC_{20}, respectively, with 0 % (reference), 2, 5, 10, and 20 % of HOFA. The prepared cube specimens were stored to air for 24 h. After this period each group of samples was kept in the separate tray and cured by cotton moisten with distilled water for 28 days.

The primarily aim of curing is to keep the mortar or concrete moist, by preventing the loss of moisture from it during the period in which it is gaining strength. Curing may be applied in a number of ways and the most appropriate means of curing may be dictated by the site or the construction method (CCD 2006). In this study the pre-washed moist cotton was applied as curing media in order to minimize leaching of metals during curing process. After 28 days, 3 cubes from each group were placed on the Automax 5 automatic concrete compression test machine to measure the compressive strength. The load was applied and gradually increased at the rate of 0.2 Mpa/s till the specimen fails. The remaining one sample from each group was separately placed in leaching solution (mentioned in Table 1) in order to perform the BLT. Some metals may be absorbed by moist cotton during curing process. To laminate this errors separate leaching test was conducted with the moist cotton and clean reference cotton.

3. Results and Discussion

The collected HOFA sample is true black in color. Its moisture content and density is 12.79 %, and 1.39 g/cm^3 respectively. The bulk density of the sample is very low (0.25 g/cm^3) compared to the density. The elemental analysis shows that the HOFA is mainly composed of carbon (i.e., 51.86 %) with minor components such as V, Ni, Fe, Cu, Zn, Sn, Pb, Cd, Cr, Mn, Co and As etc. Table 2 shows the chemical composition of the collected HOFA.

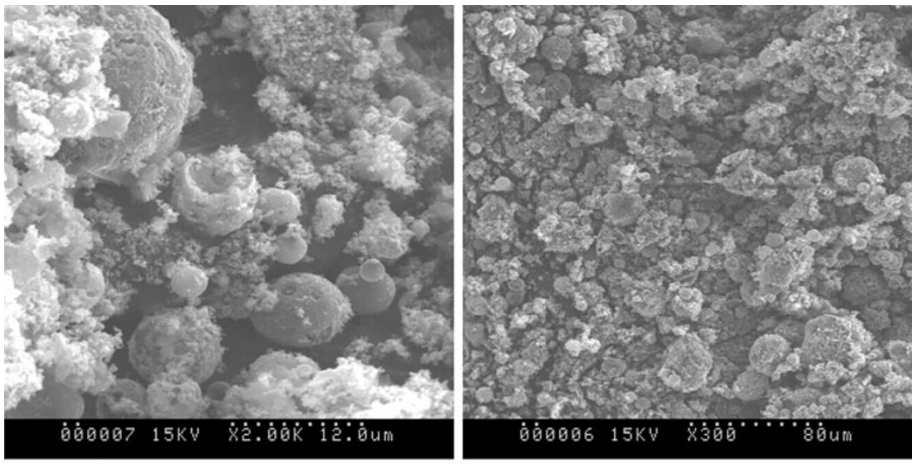
The morphological structures and mineral composition of HOFA were studied by ultrahigh resolution SEM and XRD

Table 1 Summary of heavy metal extraction procedure.

Method	Leachant	L/S ratio by weight	Extraction time (h)	Temperature (°C)	Reference
ANC	HNO_3 solutions (pH 3.0)	3:1	48 h	25	ECAEC (1986)

Table 2 Chemical composition of heavy oil fly ash.

Elements	mg/kg
Arsenic (As)	68.281
Cadmium (Cd)	1.588
Cobalt (Co)	247.79
Chromium (Cr)	107.6
Copper (Cu)	120.3
Mercury (Hg)	Not detectable
Nickel (Ni)	11,852.93
Lead (Pb)	116.095
Selenium (Se)	13.186
Vanadium (V)	34,487.12
Zinc (Zn)	592.131



(a) High magnification **(b)** Low magnification

Fig. 1 SEM analysis of fly ash.

respectively. The SEM micrographs Fig. 1a, b is shown the surface features of HOFA particles at 2000× magnification and 300× magnification respectively. These figures demonstrated that HOFA consists of spherical particles ranging in size from a few to several micrometers. The surface of particles is highly porous. These pores are individual, and randomly located on the particle surface. Generally, pores are formed by the explosion of gas inside the particle during burning and particle formation phase.

Although, HOFA sample exhibits an amorphous nature in the X-ray (Fig. 2), some distinct mineral and compound peaks (i.e., quartz, vanadium oxide) were observed. In this case, additional peaks were also attributed to carbon in graphite state. Particles size analysis confirmed the mean diameter of the FA sample is 8.56 (μm) with standard deviation of 5.21 (μm) as shown in Fig. 3. The particle size distribution is an important property of FA, with the smaller particles having greater surface areas. This property is also important during the interaction of the ash with different solutions, since it affects the mobilization of any trace elements on the surface of the particles. According to Itskos et al. (2009) the chemical and physical properties, and subsequently potential industrial utilization of FA, greatly depends on the particle size distribution.

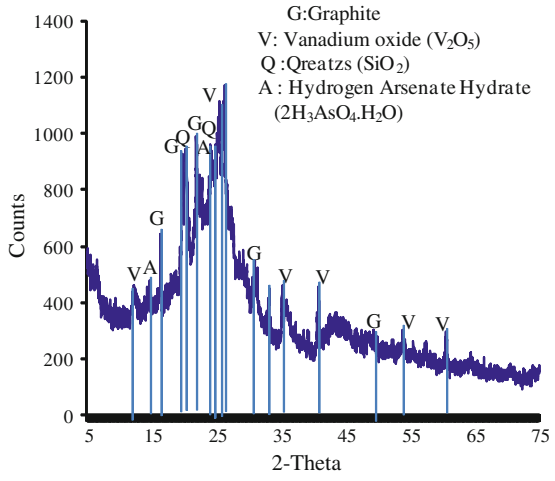


Fig. 2 X-ray diffraction analysis of fly ash.

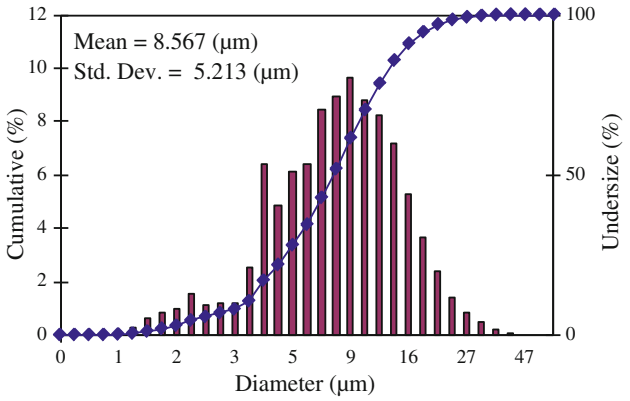


Fig. 3 Particles size distribution of fly ash.

When considering the utilization of FA as a black pigment or admixture in cement mortar, the mortar should possess suitable properties such as compressive strength. To investigate the possibility of HOFA use in cement mortar, the changes of compressive strength are measured by making cube blocks. The compressive strength of the study cubes after 28 days is shown in Table 3. The change of compressive strength of cubes is shown in Fig. 4. This figure demonstrates that the addition of 2 or 5 % HOFA in cement mortar has no significant difference in strength compared to the reference block; but the compressive strength significant decrease in the addition of 10 and 20 % of HOFA with mortar. The results show that the addition of 2 or 5 % of HOFA in cement mortar does not affect the quality of mortar. However, remarkable decreases in compressive strength were observed at 10 and 20 % HOFA in mortar.

Among the trace metals originally present in the HOFA (Table 2), only minor concentrations were obtained through leaching tests. BLT indicates that the elements in HOFA easily leachable into the environment by the rain water or acid rain. The study also pointed out that the amount of toxic metals leach by HOFA exceeds the permissible limit for drinking water recommended by DWT (2001), USEPA (1993). As can be seen Table 4, the maximum leach concentration (LC) of 0.041, 0.004, 0.283, 0.6, 77.9, and 0.116 mg/l respectively for As, Cd, Cr, Cu, Ni and Pb from the raw HOFA exceeds highest

Table 3 Change of comprehensive strength after 28 days.

% of FA	Comprehensive strength after 28 days (MPa)			
	Sample 1	Sample 2	Sample 3	Average
2	41.15	40.95	42.59	41.56
5	38.75	37.95	39.82	38.84
10	28.63	28.49	29.02	28.71
20	24.07	25.02	24	24.36
Reference cubes	42.96	41.32	42.59	42.29

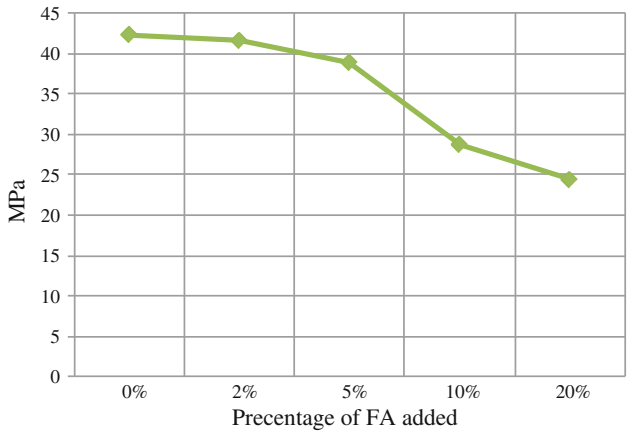


Fig. 4 Change of comprehensive strength in concrete cubes.

environmental permissible concentrations (0.01, 0.001, 0.05, 0.020, 0.05 and 0.05 mg/l respectively). Therefore, use of HOFA in the cement mortar must satisfy the environmental regulations. This was confirmed by BLT.

The leaching behavior of the 11 elements from HOFA mixed mortar materials are shown in Table 4. The results indicate that mixing of HOFA with mortar significantly decreases the LCs of most of the elements to below the permissible level. The results also show that the quantity of HOFA into the mortar has an influence on the LCs. Increased the percentage of HOFA in mortar increased the LCs of metals. However, the addition of 10 and 20 % HOFA in mortar shows higher LCs of some metals (i.e., Ni) compare to the permissible level. Furthermore the LCs of mortar with 2 or 5 % HOFA is adequately safe for the environment (i.e., compare with permissible limit shown in Table 4). In addition, 2 or 5 % HOFA in mortar does not pose any significant change in the mortar's compressive strength (Fig. 4). Even no surface degradation of the cubes after leaching test was observed. Based on the comparative results, it is clear that 2 to 5 % HOFA can be added to the cement mortar as black pigment.

4. Limitations of the Study

The age and curing environment has significant effects on the performance of mortar. This study only addressed 28 days mortar strength. Additional study is needed to

Table 4 Results of batch experiments.

Heavy metals	Detection limit (mg/l)	Peak concentration levels (mg/l) rounded to 3 digits					
		HOFA	OC_2	OC_5	OC_{10}	OC_{20}	Permissible limit[a]
As	0.001	0.041	ND	ND	0.001	0.001	0.01
Cd	0.001	0.004	ND	ND	0.001	0.001	0.001
Co	0	0.05	ND	ND	0	0	–
Cr	0.005	0.283	ND	ND	ND	0.006	0.05
Cu	0	0.6	ND	0	0	0.002	0.02
Hg	0	ND	ND	ND	ND	ND	0.002
Ni	0	77.91	ND	0.001	0.07	0.091	0.05
Pb	0	0.116	ND	0.001	0.003	0.003	0.05
Se	0.027	0.031	ND	0.001	0.001	0.001	–
V	0.001	303.912	ND	0.005	0.086	0.195	–
Zn	0.002	0.894	ND	0.001	0.003	0.006	5.00[b]

ND not detectable.

[a] DWT (2001).

[b] USEPA (1993).

evaluate the effects on age and curing environment on mortar strength. Although, the preliminary results based on the trial mixing proved that HOFA can be used as color ingredient in the ornamental bricks. However, this result may vary based on the characteristics of HOFA. Case by case study is necessary before large scale applications.

5. Conclusions

In this study the physical and chemical properties of HOFA was analyzed and a possible utilization of HOFA as a black pigment in cement mortar was investigated. Laboratory BLT was conducted to evaluate the environmental risks that may pose by ornamental brick made with HOFA. The results show addition of 2–5 % HOFA in mortar is adequately safe for the environment. As well, 2–5 % HOFA in cement mortar does not pose any significant change in the mortar's compressive strength. Based on the comparative results, it is concluded that HOFA can be used as a color pigment in ornamental brick. However, further experiments with quality and permanence of brick color are recommended.

Acknowledgments

Financial support provided by the Natural Sciences and Engineering Research Council (NSERC) of Canada is highly appreciated.

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