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Use of Ponded and Fresh Ashes from China for the Production of Portland/Calcium Sulfoaluminate Clinkers

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ABSTRACT

This article summarizes the use of two fly ashes in the synthesis of Portland/calcium sulfoaluminate (OPC/CSA or A/CSA) clinkers. They are from the Shentou second power plant located in the Shanxi Province and from the Zhungeer power plant located in Inner Mongolia, China. The Zhungeer ash was collected dry, and the Shentou ash is from a pond. Their chemical compositions differ highly, especially the SiO_2 and Al_2O_3 contents. The high contents of silica and alumina make both ashes candidates as a partial or total substitute for bauxite, an expensive source of alumina, in the production of OPC/CSA clinkers. These particular hybrid clinkers are composed mainly of alite (C_3S) and calcium sulfoaluminate ($C_4A_3\acute{S}$), both phases responsible for the high early strength development in OPC and CSA cements, respectively. The production of high-quality OPC/CSA clinkers was produced with both ashes with the additions of hydrated lime, flue gas desulfurization (FGD) gypsum, fluorite, and bauxite at 1250°C for 60 minutes with final composition ranges of 29–41 wt% C_3S, 20–22 wt% C_2S, 30–45 wt% $C_4A_3\acute{S}$, and 1–4 wt% C_4AF.

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1. Introduction

The use of industrial by-products and coal combustion products, such as fly ash, bauxite, red mud, flue gas desulfurization (FGD) gypsum, and slag, in cement and concrete has been increasing during the past few years (Tzouvalas et al., 2004; Duvallet et al., 2009; Magallanes-Rivera and Escalante-García, 2014; Jewell et al., 2015; Kim et al., 2017), and growing effort in this direction is highly encouraged for beneficial environmental purposes. From 2016 ACAA data (American Coal Ash Association, 2018), it is estimated that 60% of fly ash produced in 2016 has been reused in various applications and materials, including concrete, cement, mineral fillers, structural fills, and others. However, the portion of the ash not recycled is thus landfilled in ash ponds. Recently, the U.S. Environmental Protection Agency's coal ash surface impoundment integrity assessment program (U.S. Environmental Protection Agency, 2018) evaluated and provided assessments to power plants, resulting in a large number of pond closings.

The successful use of fly ash in cement and concrete has been demonstrated by numerous studies, whereas the use of ponded ash in cement and concrete has been examined to a lesser extent. Indeed, addition of fresh fly ash to cement and concrete has been demonstrated to improve strength and durability (Chindaprasirt et al., 2005), workability (Ravina and Mehta, 1986), sulfate resistance (Torii et al., 1995), corrosion resistance (Hussain and Rasheeduzzafar, 1994), particle size packing (decreased permeability; Hussain and Rasheeduzzafar, 1994), and decreased water demand, and thus, water:cement ratio (Ravina and Mehta, 1986), among other properties. Reported work with utilization of ponded ash in cement and concrete is less common. Studies have been reported on the use of ponded ash as fine aggregates in cement

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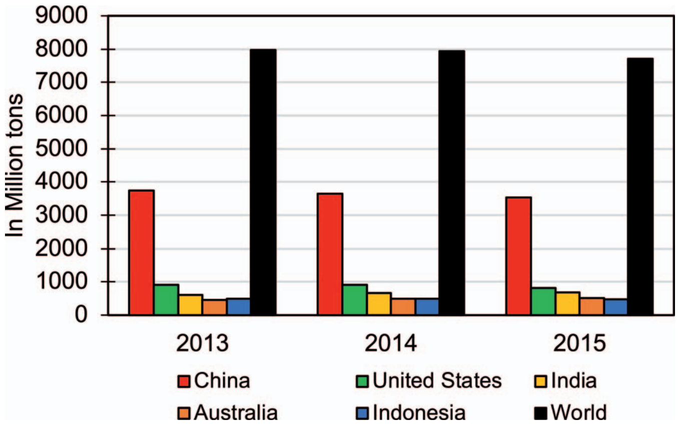

Fig. 1. Major coal producers (International Energy Agency, 2016).

Fig. 2. Google Earth image of large ash pond samples for study (appromiately 2800 m width).

concrete (Ranganath, 1995) or as a replacement for Portland cement (Cheerarot and Jaturapitakkul, 2004) with positive results, showing that even though ash has been exposed to weather for 24 months, ponded ash exhibited excellent pozzolanic properties.

China is the world's leader in coal production with 3527.2 Mt of coal produced in 2015, representing almost half of the world's production of coal at 7708.7 Mt (Figure 1), according to an International Energy Agency (2016) report. Consequently, the necessity to reuse and/or recycle coal combustion by-products like fly ashes, instead of landfilling this material, is of high priority for the country. As such, interest in the differences in ash characteristics and properties between countries and their potential reutilization is fundamental. Indeed, it has previously been found that ash from China is rich in alumina (Duvallet et al., 2016), which would potentially be an adequate replacement for bauxite, an expensive raw material used in the production of calcium sulfoaluminate (CSA) binders.

In this article, two sources of coal ash are characterized: (1) a ponded ash from the 4 × 500-MW Shentou Second Power Plant, located in Shuozhou City in the Shanxi Province of China, and (2) a dry (or "fresh") ash from the Zhungeer Power Plant, located in Ordos City at the junction of the Shanxi, Shaanxi, and Mongolia provinces in Inner Mongolia, China (Shenhua Guohua Zhungeer Power, 2017). Both ashes were characterized, providing information on their chemical and mineralogical composition, particle size distribution, and physical properties. After the characterization of both ashes, their potential use as raw material for the production of Portland/CSA (OPC/CSA or A/CSA) clinkers was assessed. OPC/CSA clinkers consist of clinker phases usually present in only OPC and CSA clinkers. Alite (or C_3S) is the clinker phase, characteristic of the main strength development in OPC, whereas calcium sulfoaluminate (or $C_4A_3\acute{S}$ or ye'elimite) is the clinker phase responsible for the high early strength development in CSA cements. The presence of both clinker phases into one clinker is made possible by the use of fluxes and mineralizers within the raw materials, as explained in previous research (Blanco-Varela et al., 1986; Giminez-Molina et al., 1992; Tobón et al., 2016). Although other methods have been attempted for the production of OPC/CSA clinkers (Xu and Sun, 2011; Ma et al., 2013; Londono-Zuluaga et al., 2017), none of them, to the knowledge of the authors, refer to the use of ponded ash as a raw material for the production of OPC/CSA clinkers. This final hybrid cement made of OPC and CSA clinker phases would theoretically have physical properties of both, through the combination of the high compressive strength development of alite and the rapid strength development of ye'elimite, while being produced at a lower temperature than OPC. The need to study this novel material is thus critical.

2. Materials, Methods, and Experiments

2.1. Materials

The materials used for this project are a ponded ash from the 4 × 500-MW Shentou Second Power Plant, mentioned above (see Figure 2), and a dry ash collected from the Zhungeer Power Plant. A typical U.S. Class F ash, collected from the landfilled ash at the Kingston power plant in Tennessee, is used for comparison. Their chemical compositions are presented in Tables 1 and 2. For the production of OPC/CSA clinkers, additional materials have been used, including hydrated lime, FGD gypsum, bauxite, and fluorite (reagent chemical grade, 99.5+% purity; Sigma-Aldrich), and their chemical compositions are presented in Table 3.

2.2. Methods and experiments

This paper is divided into two major studies. The first is dedicated to the characterization of the different ashes, which includes chemical, mineralogical, particle size, and imagery analyses compared with an American ponded ash. The second part is dedicated to the study of the production of OPC/CSA clinkers, with ashes from China as raw materials.

2.2.1. Characterization of ponded and fresh ash from China compared with an American ash

2.2.1.1. Chemical characterization

The major and trace elements in the samples were quantified with X-ray fluorescence analyses. The procedure followed ASTM D4326-13 standard (ASTM International, 2013) and was performed in a

Table 1
Major oxide composition of the ponded and fresh ashes from China compared with a standard American Class F ash

	Oxide composition (wt%)									
	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	Na_2O	K_2O	P_2O_5	TiO_2
Ponded ash	3.94	47.18	39.11	4.33	1.61	0.62	0.05	0.49	0.14	1.18
Fresh ash	4.00	32.82	54.46	2.76	1.41	0.59	0.05	0.42	0.22	1.94
American F ash	3.34	55.00	29.86	5.58	0.05	1.57	0.41	2.58	0.30	1.41

PW 2404 X-ray spectrometer supplied by Thermo Scientific Philips (Waltham, PA)

2.2.1.2. Mineralogical characterization (qualitative and quantitative analyses)

Mineralogical characterization was performed by X-ray diffraction (XRD) analyses, with a Philips X'Pert X-ray diffractometer (model PW3040-PRO) operating at 45 kV and 40 mA and utilizing Cu K-α radiation. Qualitative analyses were performed for the characterization of the ashes received, whereas quantitative (or Rietveld) analyses were performed for the characterization of the clinkers produced. For both analyses, samples were first ground mechanically in a small shatter box, and if necessary, ground further by hand with a mortar and pestle. The samples were dry mounted in aluminum holders. The step size for the qualitative XRD analyses was 0.017° at 0.035°/s, over 8–60° 2θ, and for quantitative XRD analyses, it was 0.017° at 0.013°/s, over 8–90° 2θ. The crystalline phases were identified using the International Centre for Diffraction Data (ICDD, Newton Square, PA).

2.2.1.3. Particle size distribution

The particle size distribution of ashes was determined by sieving the ashes through 50-, 100-, and 200-mesh sizes (i.e., 300, 150, and 75 µm, respectively).

2.2.1.4. Scanning electron microscopy

Scanning electron microscopy (SEM) was used for the study of the physical shape of the ash particles. A Hitachi S-4800 SEM was operated at a voltage of 15 kV and a current of 20 µA. The ashes were deposited on top of a carbon tape on an SEM holder and sputtered with gold for 180 seconds.

2.2.2. OPC/CSA clinker production

The type of raw materials, their chemical compositions, the quantities used for the production of OPC/CSA clinkers, and their final theoretical clinker compositions are detailed in Tables 3 and 4. Two OPC/CSA clinkers were produced: one with the ponded ash (partial substitution for bauxite), hydrated lime, FGD gypsum, and bauxite, and another produced with the fresh ash (total substitution for bauxite), hydrated lime, and FGD gypsum. Bauxite was used in the composition with ponded ash to obtain a relatively high content of ye'elimite, which without bauxite would not have reached around 30 wt%. Fluorite was added to improve the formation of alite (C_3S) and assure that C_3S coexists with calcium sulfoaluminate ($C_4A_3\acute{S}$) (Duvallet et al., 2017). The raw materials were mixed with a mortar and pestle until complete homogenization with addition of deionized water (10 wt%). Small 28 × 7-mm disks were then produced with a load of approximately 11,340 kg (25,000 represents a pressure of 180 MPa) and dried in an oven at 60°C for a few hours. The disks were placed in platinum crucibles and fired at 800°C for 30 minutes, followed by 1200°C/1250°C for 60 minutes, and finally quenched in air. Samples were crushed in a shatter box and submitted for XRD/Rietveld analyses to quantify the final clinker composition.

3. Results

3.1. Characteristics of ponded and fresh ashes from China compared with an American ash

3.1.1. Chemical composition

The chemical composition of ashes is presented in Tables 1 and 2, along with the chemical composition of a standard American Class F fly ash as a comparison. Both ponded and fresh ashes are similar in terms of oxide contents, with the exception of SiO_2 and Al_2O_3. The ponded ash contains more SiO_2 than the fresh ash, and the fresh ash contains more Al_2O_3 than ponded ash. Both ashes from China can be classified as F fly ash (ASTM International, 2012) because their total content of (SiO_2+Al_2O_3+Fe_2O_3) is above 70 wt%. However, both ashes contain high amounts of silicon oxide and aluminum oxide and low amounts of iron oxide when compared with the American ash.

3.1.2. Mineralogical composition

On the basis of XRD analyses shown in Figure 3, both ashes from China contain amorphous materials, as well as mullite, ettringite, gypsum, calcite, and quartz. The particularity between both ashes is the presence of calcium aluminate (CA_2) and alumina (Al_2O_3) in the fresh ash, which may be due to the high content of aluminum oxide within the chemical composition, as shown in Table 1. In comparison, the standard American Class F ash contains mullite, quartz, and amorphous phases.

Table 2
Trace elements in the composition of the ponded and fresh ashes from China compared with a standard American Class F ash

	Trace elements (ppm)															
	As	Ba	Cd	Co	Cr	Cu	Mo	Mn	Ni	Pb	Rb	Sb	Sr	V	Zn	Zr
Ponded ash	<1	240	<1	12	56	15	184	174	13	40	270	4	458	154	32	317
Fresh ash	8	278	<1	19	54	31	221	160	1	76	468	5	1213	359	58	597
American F ash	57	1518	<1	25	108	178	126	163	107	76	44	2	1549	429	185	4

Table 3
Chemical composition of the raw materials used for the preparation of Portland/calcium sulfoaluminate clinkers, mixed with ponded and fresh ashes

	Raw material composition (wt%)									
	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	Na_2O	K_2O	P_2O_5	TiO_2
Hydrated lime	93.02	2.79	2.23	0.6	0.1	3.0	0.16	0.1	0.01	0.1
FGD gypsum	40.79	1.3	0.28	0.1	57.89	0.21	0.02	0.04	0.01	0.1
Bauxite	0.22	9.1	80.23	8.61	0.33	0.27	0.01	0.01	0.3	2.99

3.1.3. Particle size distribution

The particle size distribution of both ponded and fresh ashes from China is presented in Figure 4, along with a standard American Class F ash for comparison. The ponded ash was not easily separated, and most of the ash particles (82 wt%) were between 75 and 300 µm, which may be due to the agglomeration of particles from solution and precipitation of gypsum in the pond. The fresh ash, in contrast, was easy to sieve, and most of the particles (85 wt%) were less than 75 µm. Additionally, the fresh ash from China is highly similar to the American F ash in terms of size distribution, even though the American ash is a ponded ash. Indeed, the American ponded ash did not alter in composition or size distribution by being landfilled, which demonstrates that the American ash did not react with water during the landfilling process.

3.1.4. Physical properties

The shape of the particles from both ashes from China and the standard American ash was analyzed with a scanning electron microscope. Particles from the ponded ash were both angular and spherical, as shown in Figure 5. The particles of the fresh ash were mostly angular, with no spherical particles present, as seen in Figure 6. However, as shown in Figure 7, the particles of the standard American Class F ash are highly spherical. This differences in morphology are related to the coal origin and the parameters and processes in place during the combustion of coal.

On the basis of characterization tests, both ponded and fresh ashes from China are excellent candidates for use as raw materials, especially as a bauxite replacement, for the production of OPC/CSA clinkers, as per their chemical composition with high content of both aluminum oxide and silicon dioxide.

3.2. Use of ponded and fresh ashes from China for the production of OPC/CSA hybrid cements

OPC/CSA hybrid cements are composed mainly of C_3S and $C_4A_3\acute{S}$ (ye'elimite, or also called Klein's compound), which are the major clinker phases in Portland and CSA cements, respectively. Both C_3S and $C_4A_3\acute{S}$ are responsible for strength development in their respective cement compositions: C_3S hydrates to calcium silicate hydrate (C-S-H), responsible for most of the strength development in

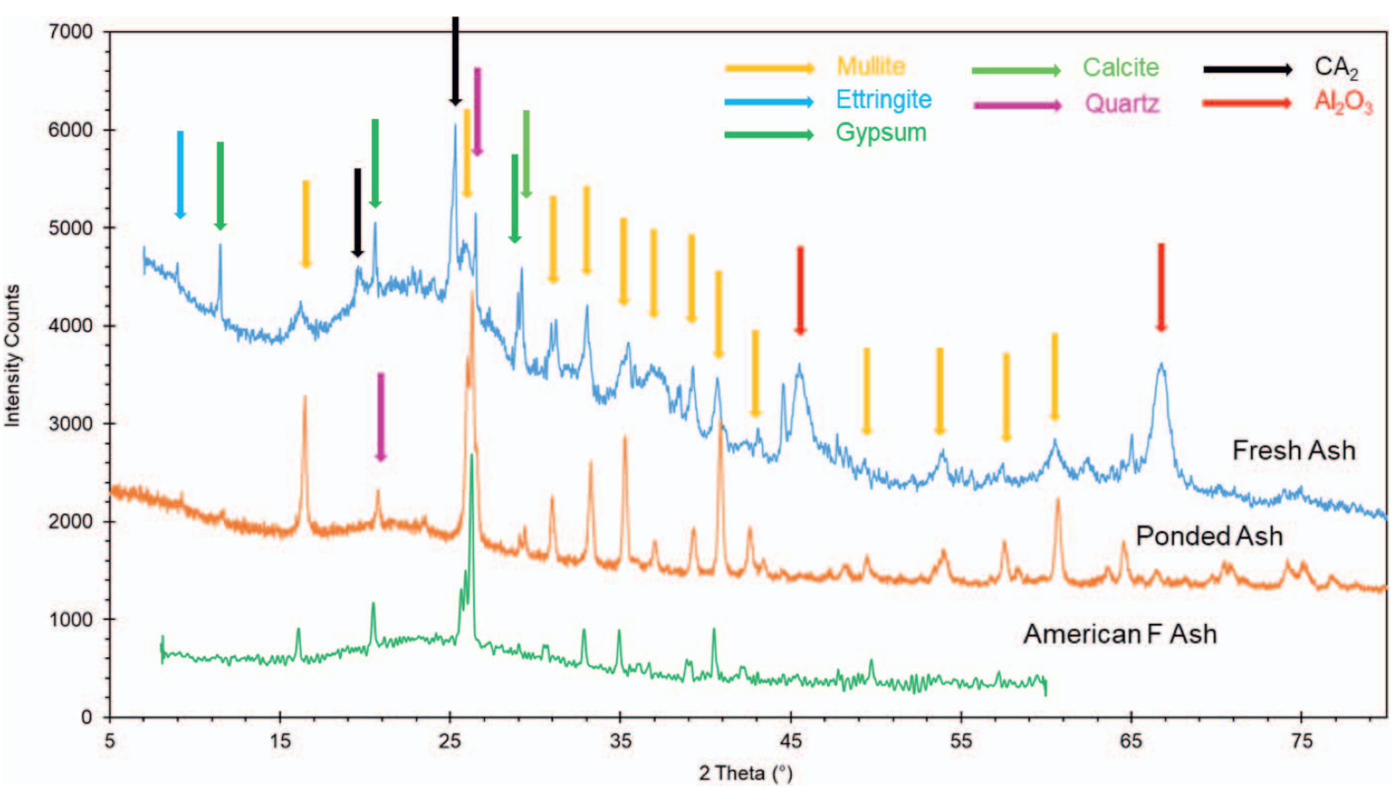

Fig. 3. X-ray diffraction patterns of ponded and fresh ashes from China.

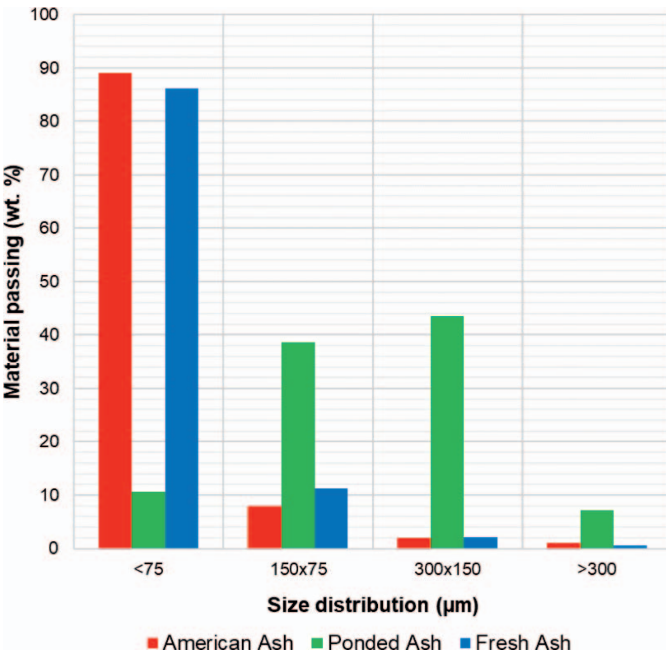

Fig. 4. Particle size distribution of the ponded and fresh ashes from China compared with a standard American Class F ash.

Table 4
Compositions of Portland/calcium sulfoaluminate clinkers made with ponded ash and fresh ash

Raw materials	With ponded ash (wt%)	With fresh ash (wt%)
Hydrated lime	65.5	58.4
FGD gypsum	6.3	9.7
Bauxite	5.0	–
Ponded ash	22.5	–
Fresh ash	–	31.3
Fluorite	0.7	0.6

ordinary Portland cement (Bogue and Lerch, 1934), whereas $C_4A_3\acute{S}$ hydrates to ettringite, responsible for the high early strength development in CSA cements (Sharp et al., 1999). The production of OPC/CSA clinkers require the use of raw materials with high contents of calcium, sulfate, silica, and, most of all, alumina. Bauxite

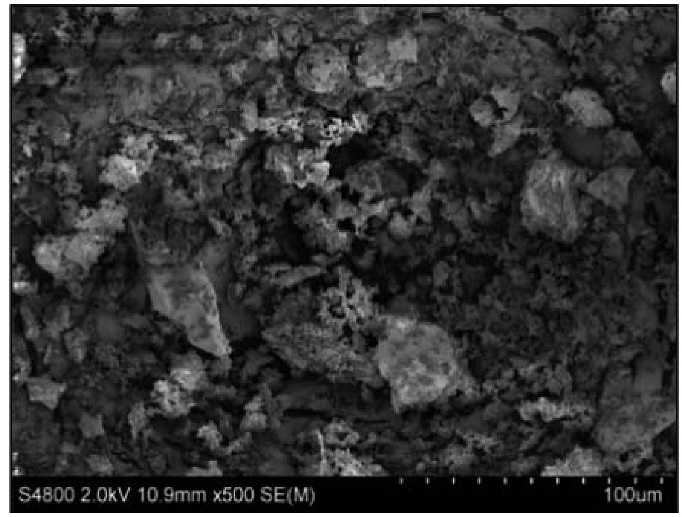

Fig. 6. Scanning electron microscopy (SEM) image of particles of the fresh ash from China.

Table 5
Theoretical and Rietveld analyses of clinkers made with ponded and fresh ashes from China fired at different temperatures for 60 minutes

	With ponded ash (wt%)		With fresh ash (wt%)		
	Theoretical	Rietveld 1250°C[1]	Theoretical	Rietveld 1200°C	1250°C
C_3S	≈60	40.2	51.0	12.3	29.3
C_2S	≈0	21.3	0.1	36.3	21.4
$C_4A_3\acute{S}$	29.8	32.0	40.0	39.1	43.3
C_4AF	6.1	3.2	4.0	0.9	2.0
$C\acute{S}$	0.6	2.0	1.9	0.6	–
MgO	2.0	1.2	1.8	1.7	1.2
C_3A	–	–	–	1.0	1.4
f_{CaO}	–	–	–	4.6	1.4
$C_{12}A_7$ (mayenite)	–	–	–	3.6	–

Note: f_{CaO} = free lime content.
[1] The sample with the ponded ash was fired twice at 1250°C for 60 minutes and ground again between both firings.

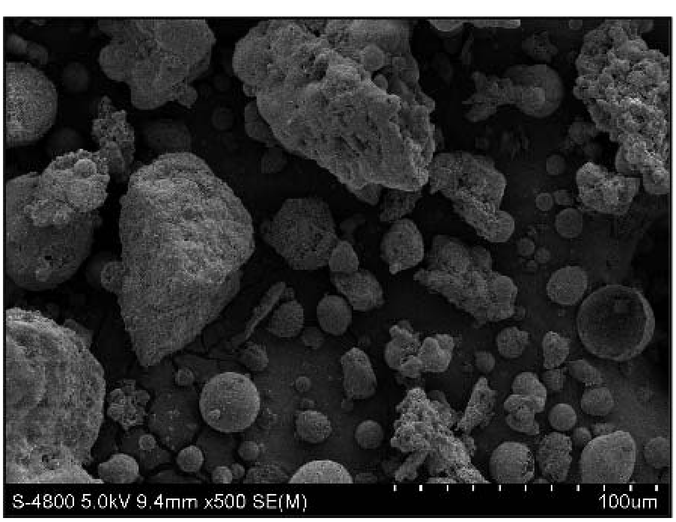

Fig. 5. Scanning electron microscopy (SEM) image of particles of the ponded ash from China.

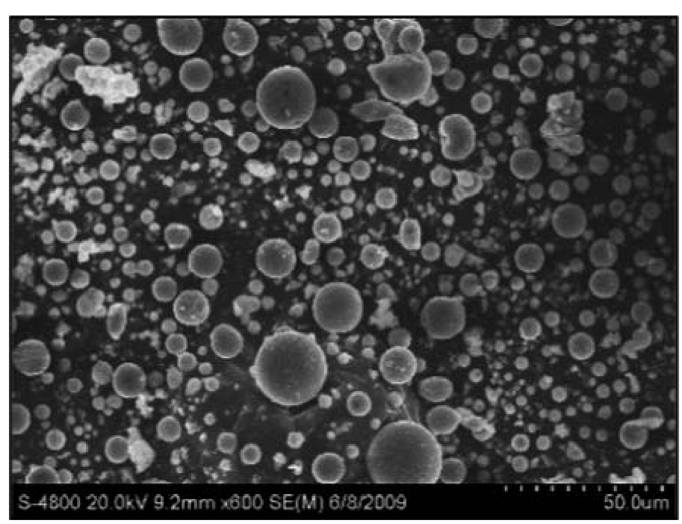

Fig. 7. Scanning electron microscopy (SEM) image of particles of a standard American F ash.

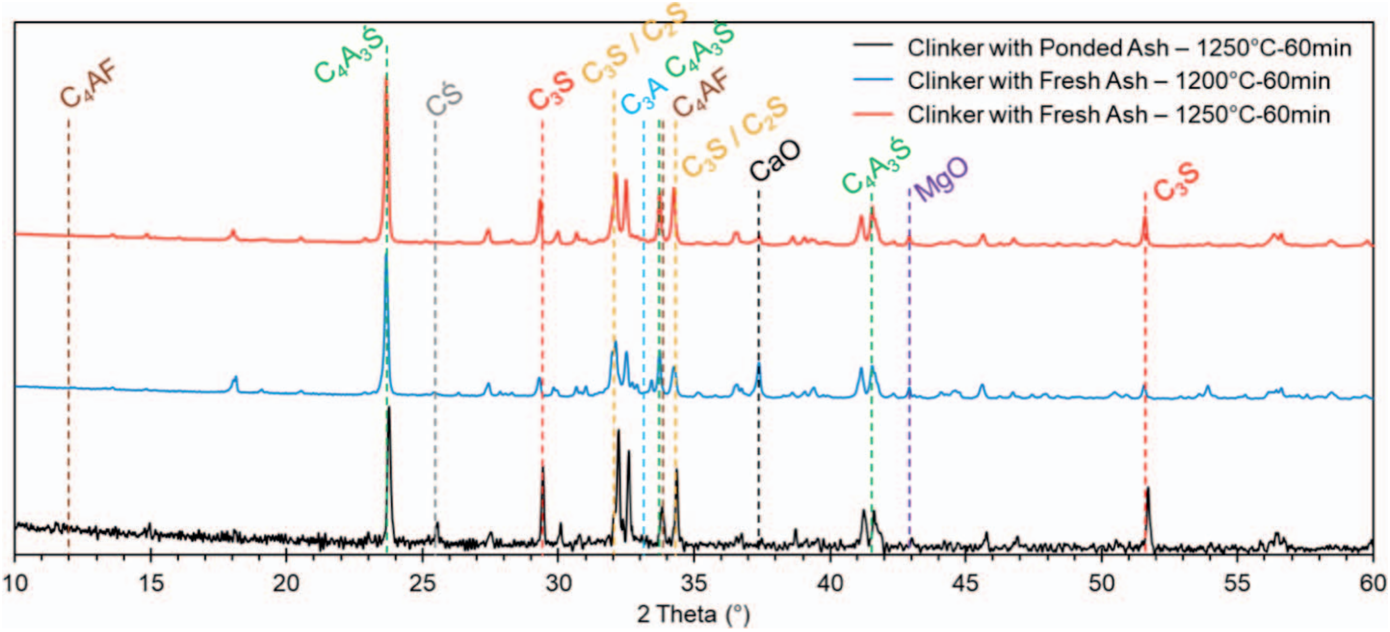

Fig. 8. X-ray diffractograms of clinkers made with ponded and fresh ashes fired at different temperatures.

is usually used as a source of alumina for the production of CSA clinkers, although this material is expensive. Both ashes from China contain important quantities of alumina and would seem to be a viable partial or total substitute for bauxite in the production of OPC/CSA clinkers.

XRD and Rietveld results from the samples made with both ashes from China fired at different temperatures are presented in Figure 8 and Table 5. For the clinker made with fresh ash, the low firing temperature of 1200°C was not high enough, and some free lime (4.6 wt%) was still present at the end of the clinkerization process. As a general rule, if the free lime content exceeds 2.0 wt%, clinkerization is considered not complete. However, the 1250°C firing temperature improved the clinker composition by decreasing the quantities of free lime (now at 1.4 wt%) and belite and increasing the amount of alite. When comparing the Rietveld data from Table 5 with the theoretical values from Table 4, the amounts of calcium sulfoaluminate are highly similar, with more than 30 wt% for the clinker made with ponded ash, and around 40 wt% for the clinker made with fresh ash, when fired at 1250°C. Regarding the amounts of belite and alite, the values are not close to the theoretical values, although when adding both belite and alite, totaling to 40 wt% for the clinker made with ponded ash and 60 wt% for the clinker made with fresh ash, it is then close to the theoretical values. A higher firing temperature or a longer dwelling time would help by improving the formation of alite, although it may also decompose the calcium sulfoaluminate into calcium aluminate phases ($C_{12}A_7$ and CA) and O_2 and SO_2 (Puertas et al., 1995; Hanein et al., 2015).

4. Conclusions

The ponded and fresh ashes from China are both Class F fly ashes and present similar chemical composition, with high amounts of aluminum oxide and silicon oxide, making them candidates for use as an affordable replacement for the expensive bauxite in the production of OPC/CSA clinkers. The clinkers made from both ashes demonstrate that they can be used successfully to produce OPC/CSA hybrid clinkers. The next step of this project will be to produce large amounts of these clinkers and test the hydration processes and the mechanical properties of these cements.

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