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# Production of Portland cement clinker from French Municipal Solid Waste Incineration Bottom Ash

Joelle KLEIB<sup>1\*</sup>, Georges AOUAD<sup>2</sup>, Nor-Edine ABRIAK<sup>1</sup>, Mahfoud BENZERZOUR<sup>1</sup>

<sup>1</sup> Univ. Lille, IMT Lille Douai, Univ. Artois, Yncrea Hauts-de-France, ULR 4515 - LGCgE, Laboratoire de Génie Civil et géo-Environnement, F-59000 Lille, France

<sup>2</sup> Faculty of Engineering, University of Balamand, UOB, Al Koura, Lebanon

\* Corresponding author: Joelle KLEIB

Email: [joelle.kleib@imt-lille-douai.fr](mailto:joelle.kleib@imt-lille-douai.fr)

Univ. Lille, IMT Lille Douai, Univ. Artois, Yncrea Hauts-de-France, ULR 4515 - LGCgE, Laboratoire de Génie Civil et géo-Environnement, F-59000 Lille, France

## Abstract

The amount of bottom ashes generated from the municipal solid waste incineration is increasing considerably. Therefore their recycling is necessary in order to limit the landfilling that presents environmental issues. The aim of this paper is to recycle Municipal Solid Waste Incineration Bottom Ash from the north of France; as a secondary raw material for Portland cement clinker production. In this order Municipal Solid Waste Incineration Bottom Ash is characterized and then used to substitute raw materials for clinker production at laboratory scale. Substitutions rates up to 12.4% have been tested. The produced clinkers have been characterized by X-Ray Diffraction and the crystalline phases were quantified by Rietveld analysis. The microstructure has been studied by scanning electronic microscopy. After adding gypsum, the compressive strengths of the corresponding cements were measured at 1, 7 and 28 days. The mineralogical composition of the 28 days hydrated cement pastes were studied by X-Ray Diffraction. The results show that up to 12.4% of Portland cement raw materials can be replaced by the Municipal Solid Waste Incineration Bottom Ash without changing the mineralogical composition. In addition the compressive strengths of the cement pastes produced with Municipal Solid Waste Incineration Bottom Ash are equivalent to the one without bottom ash.

**Keywords:** Clinker; Compressive strength, Municipal Solid Waste Incineration Bottom Ash, Recycling

## 1 Introduction

The cement industry is responsible for more than 6% of the total anthropogenic greenhouse gases emissions. In fact the production of Ordinary Portland Cement “OPC” generates 842 kg CO<sub>2</sub>/t of clinker [1][2][3][4][5]. In addition to its huge energy consumption, the cement industry uses large amounts of raw materials for cement production [6]. Therefore the replacing of raw materials in the clinker raw meal by waste as secondary material, gains a lot of attention [2][7][8][9]. This replacement allows not only to preserve the natural resources and to recover wastes, but also to reduce the CO<sub>2</sub> emissions. Different types of wastes have been studied in the literature in order to produce cement, and mainly OPC cement, such as red mud [10], steel slag [11], sediments [12][13], recycled concrete aggregate [14], oil based mud cutting [15], oyster and scallop shell [16] etc.

In the European Union, the Municipal Solid Waste (MSW) production is significantly increasing. In 2015, each inhabitant generated about 477 kg of MSW per year [17] [18]. The incineration presents the most common method to treat these wastes as an alternative to landfilling [19] and reduces between 70 and 90% the volume of wastes [20][21]. The MSWI ash is a by-product from the combustion of MSW [22][21][23][24][25]. Two types of ashes are produced following the incineration operation: bottom ash and fly ash. The recycling of these ashes is a concern especially that their quantities are increasing considerably and due to their highly variable composition.

The MSWI bottom ash (MSWI BA) represents 75-80% of the produced ashes [26][27][28]. Nowadays, the landfilling presents the main option for MSWI BA disposal [29][30]. However, the landfill causes the pollution of the groundwater and contaminates the soil in addition to the limited land space and the problems related to the disposal cost and the regulation [31][32][33]. The literature describes different methods to recycle the MSWI BA in civil engineering. The latter has been used as supplementary cementitious materials and pozzolanic material [29][27][34][35], as an aggregate replacement [19][28][36][37] or in alkali activated materials as a precursor [20][38][39], as a road based material and in road construction [40][41][42].

The chemical composition of MSWI BA contains CaO, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> [43][44]. This composition makes the use of MSWI BA, as a secondary raw material, for clinker production possible [45]. Li et al. analyzed different fraction from MSWI BA from Beijing to produce a type II Portland cement and shows that 9% of this waste could be added to the raw meal [46]. Pan et al. also used MSWI fly ash and bottom ash, after washing with water/acid as a pre-treatment, to produce clinker by incorporating 1.75 and 3.5% respectively in the raw meal [47]. The percentages of incorporation were determined based on the chloride content in the ashes in order to respect the limit of chloride of 100 ppm required by the cement plant in Taiwan. They showed that this incorporation did not affect the compressive strength and the cement produced is conformed to the Chinese National Standard of type II Portland cement [47].

It is important to note that the MSWI BA has a very heterogeneous composition depending on several factors such as the waste origin, the incineration process, the incinerator used, the pre-treatment applied, etc. [48][49][50]. Therefore the percentage of substitution in the clinker raw meal differs from one to another MSWI BA. Until today, there are no studies dealing with the use of MSWI BA generated in France, as a secondary raw material for clinker production. Therefore the aim of this study is to use Municipal Solid Waste Incineration Bottom Ash from the north of France, as a raw material in the OPC clinker production. First a reference OPC clinker is produced without adding MSWI BA. Then 2 substitution rates of MSWI BA are applied: 5% and a second one corresponding to the maximum amount of MSWI BA that could be incorporated while respecting the targeted composition of the reference clinker. The mechanical properties of the resulting cement are compared to reference OPC cement produced as well as the hydration products.

## 2 Materials and methods

The materials used in this study for the clinker production are the chemical reagents: CaCO<sub>3</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and the MSWI BA from the north of France.

### 2.1 MSWI BA characterization

The MSWI-BA used in this study originates from the recycling platform RMN-PréFerNord located in Fretin in the region of “Hauts-de-France” in France. This platform treats between 150,000 and 180,000 tons yearly and is considered to be among the largest in France in terms of quantities and the techniques used for the process of preparation of the MSWI-BA. MSWI-BA was subjected to a

maturation period of two to four months in open air and was considered recyclable in the field of roads' construction according to the French regulation of 18 November 2011 [51]. The fraction 0-2 mm of Municipal Solid Waste Incineration Bottom Ash (MSWI BA) is crashed and then sieved at 100  $\mu\text{m}$ . The mineralogical characterization was made by X-ray diffraction (XRD) using a diffractometer Bruker D2 with Cu K $\alpha$  radiation. The X-ray patterns were acquired in the 2 $\theta$  (10-80 °) with a step of 0.02° and 1 s per step. The chemical composition was analyzed by X-ray fluorescence (XRF). The particle size distribution was determined using the laser granulometer COULTER type LS 230.

## 2.2 Clinker production at laboratory scale

Three Portland clinkers are produced in the laboratory with different percentage of substitution by MSWI BA. The first is a reference clinker referred by OPC0 produced using analytical reagents (CaCO<sub>3</sub>, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>) only. The theoretical mineralogical composition of OPC0 is shown in table 1. This composition was chosen according to the study of Gineys et al [8] in order to produce an Ordinary Portland Cement.

Table 1 : Theoretical composition of the targeted Portland clinker

C <sub>3</sub> S (wt. %)	C <sub>2</sub> S (wt. %)	C <sub>3</sub> A (wt. %)	C <sub>4</sub> AF (wt. %)	C (wt. %)
65%	18%	8%	8%	1%

For the MSWI BA based clinker, the solver option in Microsoft Excel was used to adjust and calculate the amount of the different raw materials [14] when substituting with MSWI BA. This option uses the chemical composition, determined by XRF for the different raw materials and MSWI BA (% wt. of Ca, Si, Al, Fe), with the aim to respect the targeted cement criteria represented in table 1.

The first substitution rate in the clinker raw meal by MSWI BA corresponds to 5% (OPC5). The second one corresponds to the maximal amount of MSWI BA that could be incorporated in the raw meal while respecting the theoretical composition of the targeted Portland cement.

Table 2 shows the mixes designs of the 3 clinker raw meals obtained by solver and the percentage of each component to add, in order to reach the theoretical composition of table 1. As it can be seen from the table 2, the maximum amount of MSWI BA that could be incorporated in the raw meal is controlled by the Fe<sub>2</sub>O<sub>3</sub> content (OPC12.4). Adding more than 12.4% of MSWI BA (maximum value) in the raw meal provides more Fe<sub>2</sub>O<sub>3</sub> than needed and consequently the resulting clinker composition differs from the theoretical composition of the targeted Portland clinker.

Table 2: Mixes designs for the 3 produced clinker raw meals

Constituents	CaCO <sub>3</sub> (wt. %)	SiO <sub>2</sub> (wt. %)	Al <sub>2</sub> O <sub>3</sub> (wt. %)	Fe <sub>2</sub> O <sub>3</sub> (wt. %)	MSWI BA (wt. %)
OPC0	79.98	15.35	2.92	1.75	0
OPC5	77.45	14.11	2.39	1.05	5
OPC12.4	73.71	12.28	1.61	0	12.4

The method to produce clinker consists to mix all materials together by adding water to better homogenize. The mix is then dried at 105°C and pressed at 5 kN into pellets to obtain a better clinkering process. Then the pellets are fired up 30 min at 200°C then to 1450 °C with a rate of 10°C. After 15 min of burning at the clinkering temperature, the clinker was cooled in the furnace. The obtained clinkers are ground until a Blaine specific area of 4500 cm<sup>2</sup>/g. To produce cements, gypsum was added to the clinker using equation 4 [52]:

Amount of gypsum used = Al<sub>2</sub>O<sub>3</sub> content \* [0.6/101.96] \* [80/0.461] (equation 4)

Where, 101.96 = mol. wt. Al<sub>2</sub>O<sub>3</sub>; 80 = mol. wt. SO<sub>3</sub>; 0.461 = mol. ratio SO<sub>3</sub>/natural gypsum used (for pure gypsum 80/172 is 0.466)

## 2.3 Clinker and cement paste characterization

The mineralogy of the synthesized clinkers was studied using X-ray diffraction (XRD). A Bruker D2 with Cu K $\alpha$  radiation was used. The X-ray patterns were acquired in the 2 $\theta$  (10-80 °) with a step size of 0.02 ° and 1 s per step. A Rietveld analysis was also conducted using TOPAS software in order to quantify the crystalline phases.

The Microscopic observations were conducted on polished section of clinkers using SEM type Hitachi S-4300SE/N. Local chemical analyses were conducted using energy dispersive X-ray spectrometer (EDS) with a Thermo Scientific Ultra Dry EDS detector coupled with the SEM. The polished sections were made by vacuum impregnating a clinker monolith in an epoxy resin. After hardening, the samples were polished with ethanol and then coated with carbon.

The reactivity of the cement pastes was followed by isothermal calorimetry performed at 20°C. The water/cement ratio used was 0.5. The calorimeter was a home-made calorimeter using fluxmeters that allowed the calorimeter to equilibrate in less than 5 min [53].

The hydrates of the 4 different cement pastes are identified by XRD analysis at 28 days after stopping the hydration by immersing the cement pastes - already grounded into powder – in ethanol. After few minutes of mixing with ethanol, a filtration is done using the Buchner filter. The powder is then collected and dried in the oven at 50°C.

The compressive strength was determined on cement paste small cubes 1\*1\*1 cm (due to the limited quantities of cement produced in the laboratory according to [51]) made of cement pastes with a water/cement ratio (w/c) of 0.5. Cement and water were manually mixed. The filling of the molds (polytetrafluoroethylene (PTFE) molds) was performed in two steps on a vibration table. The molds were kept for 24 h at 100% relative humidity at 20 °C. The paste cubes were demolded after 24 h and curing was continued at 100% relative humidity at 20 °C. The compressive strength was measured after 1, 7 and 28 days [53], [54].

### 3 Results and discussions

#### 3.1 MSWI BA characterization

Table 3 presents the bottom ash composition as well as the soluble salts. The results show that MSWI BA contains all the necessary major elements for the clinker production (Ca, Si, Al and Fe).

Table 3: Bottom ash composition

Element	LOI 950°C	O	Ca	Fe	Al	Si	P	S	Cl	Pb
Percentage (%)	13.99	34.06	18.91	9.45	5.57	11.06	0.76	0.94	0.25	0.17
Element	K	Na	Ti	Cr	Mn	Mg	Cu	Zn	Ba	
Percentage (%)	0.6	1.25	0.44	0.1	0.19	1.13	0.24	0.63	0.11	
soluble salts (mg/kg)										
Fluoride					<20					
Chloride					2740					
Sulphate					5059					

The X-ray diffraction (figure 1) for MSWI BA shows that the principal phases are quartz, calcite, with some minor phases such as hematite, gehlenite etc.

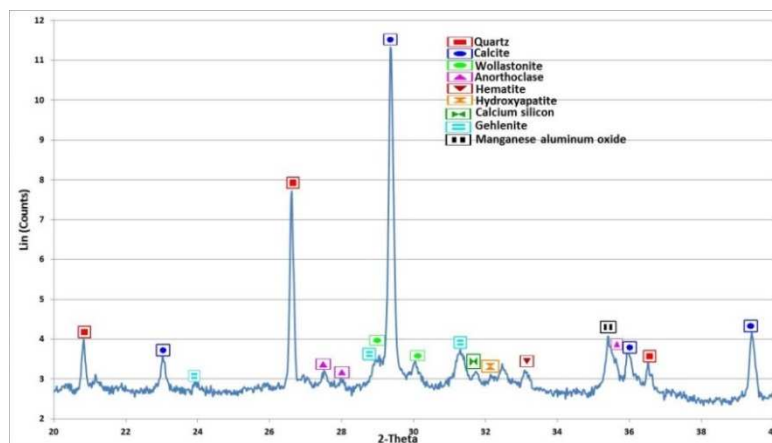


Figure 1: X-ray pattern for MSWI BA

The particle size distribution of MSWI BA is represented in figure 2. The results show that 90% of particles are inferior to 70  $\mu\text{m}$ . In fact the finesse of the raw materials is important in order to have a better reactivity.

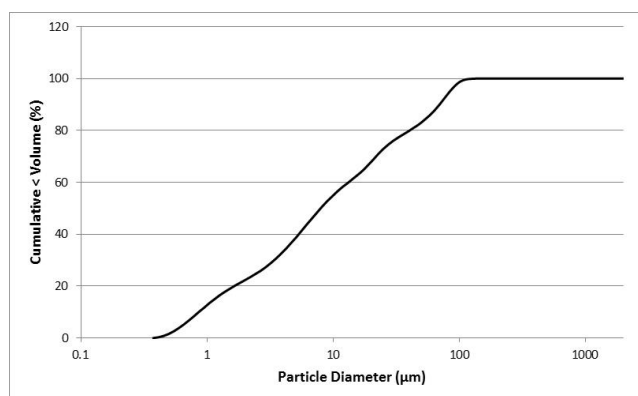


Figure 2: Particle size distribution for MSWI BA

## 3.2 Clinkers characterization

### 3.2.1 Mineralogical and chemical analyzes

The OPC0 reference clinker characterization by XRD is presented in figure 3. The result shows the presence of the 4 crystalline phases of a Portland clinker,  $\text{C}_3\text{S}$ ,  $\text{C}_2\text{S}$ ,  $\text{C}_4\text{AF}$  and  $\text{C}_3\text{A}$ . The pattern shows also that no secondary mineral phases are produced. Therefore the raw meal composition, the clinker preparation process at the laboratory scale and the burning temperature are suitable for this study.

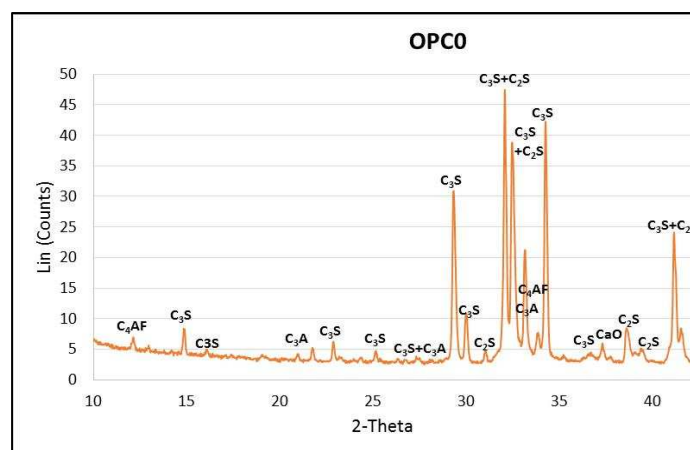


Figure 3: XRD patterns of the reference clinker OPC0



The MSWI BA based clinkers OPC5 and OPC12.4 are characterized and compared to the reference clinker. The results are shown in the figure 4. As it can be seen from the patterns, the 2 MSWI BA based clinkers, OPC5 and OPC12.4 present the same mineralogical phases as the reference clinker OPC0. Consequently the partial substitution of the raw materials by MSWI BA does not modify the mineralogical composition of the produced clinkers.

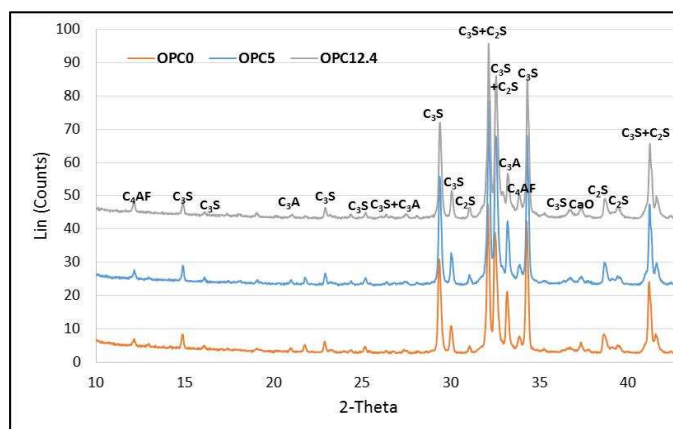


Figure 4: XRD results for the reference clinker OPC0 and for the MSWI BA based clinker OPC5 and OPC12.4

The three produced clinkers were subjected to an elementary analyzes by XRF in order to detect the presence of heavy metals and alkalis (Table 4). The results show that some heavy metals (Zn) and alkali (Na) slightly increases while increasing the MSWI BA substitution. The presence of these heavy metals could have an influence on the hydration mechanisms and on the setting time of the cement by forming a heavy metal hydroxide on the surface of the anhydrous cement grains, as suggested by Gineys et al. studies [55][56].

Table 4: XRF analyzes for the 3 produced clinkers

Element (%)	L.O.I (%)	O	Ca	Si	Fe	Al	Na	Mg	P	Ti	Zn
OPC0	3	38.8	47.3	9.9	1.8	2.2	-	-	-	-	-
OPC5	2.7	38.4	47.3	9.9	1.9	2.0	0.2	0.1	<0.1	<0.1	<0.1
OPC12.4	2.6	38.7	46.4	10.1	1.9	2.0	0.2	0.2	0.1	0.1	0.1

In order to quantify the crystalline phases in the 3 synthesized clinkers, Rietveld analysis was conducted. The results are represented in the table 5 and show that adding MSWI BA in the raw meal produces a clinker with slightly less  $C_3S$  comparing to the reference clinker. This could be due to the consideration in the calculation method that the four clinker phases are in their stoichiometric state which is not quite realistic especially when incorporating wastes into the raw meal where the phases can accommodate substitute ions [57]. Same results was obtained by Clavier et al. 2021 [43] when incorporating bottom ash in the clinker. This was attributed to the operational variations associated with lab-scale clinker production. In fact the  $C_3S$  tends to revert to  $C_2S$  when clinker is cooled too slowly and therefore the speed of cooling showed be considered. In addition the Portland cement chemistry is complex, therefore this complexity increases when a replacing the raw materials by a heterogeneous material such as MSWI BA. The latter contains, as shown in table 3, some metallic elements (i.e. Cu, Zn, Mg, Cl...) that could cause a reduction in the mineralization temperature, favoring therefore the formation of  $C_2S$  over the  $C_3S$  phase[58]. Concerning the residual lime, its content is between 0.05% and 0.21% (table 5) which confirms that the clinkering was complete because these values are lower than what is specified in the standard NF EN 196-2. In addition, according to the standard there is no risk due to these low free lime contents (<0.5%).

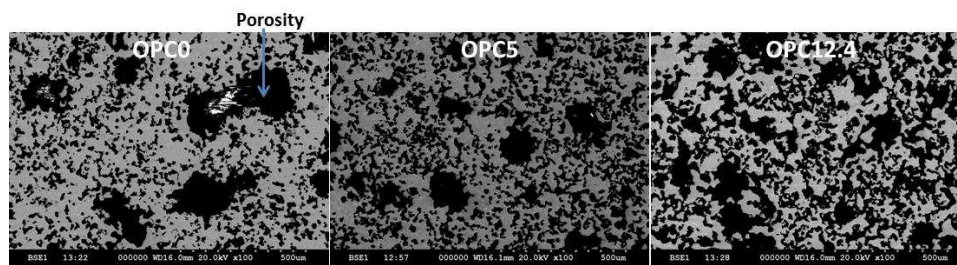
Table 5: Rietveld analysis for the 3 clinkers: OPC0, OPC5 and OPC12.4

	$C_3S$ (wt. %)	$C_2S$ (wt. %)	$C_4AF$ (wt. %)	$C_3A$ (wt. %)	CaO (wt. %)	GoF
OPC0	76.14	12.27	3.5	7.88	0.21	1.65
OPC5	74.01	15.21	4.07	6.66	0.05	1.44
OPC12.4	72.12	19.60	4.13	4.09	0.06	1.45

### 3.2.2 Microstructure observation

The microstructure of the 3 produced clinkers was observed by SEM/EDS on polished sections (figure 5). By comparing the 3 samples, it can be seen that the porosity (black areas) of the clinker matrix increases while increasing the incorporation percentage of MSWI BA. According to the literature, this behavior could be due to the presence of heavy metals (i.e. Zn) that could change the viscosity of the interstitial liquid phase of clinkers during the sintering process. In fact, zinc acts as a mineralizer [59], therefore the porosity of the clinkers could be influenced [60] [61].

Figure 5: Microstructural observations of the 3 clinkers: OPC0, OPC5 and OPC12.4



In order to identify the mineralogical phases of clinkers, elemental analyzes by EDS were conducted on OPC0 and OPC12.4 (figure 6). In both clinkers,  $C_4AF$  (point 1),  $C_3A$  (point 2),  $C_2S$  (point 3) and  $C_3S$  (point 4) were identified. The alite grains appeared to be angular whereas the belite one round. The clinkers, with and without BA, present in general almost a similar microstructures.

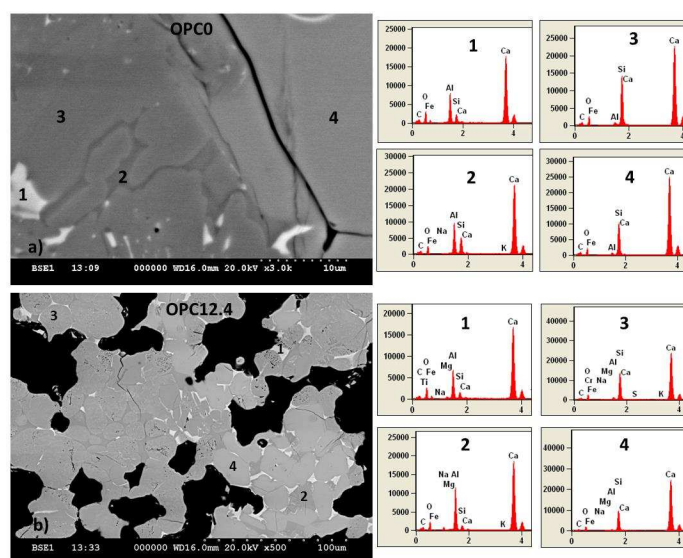


Figure 6: SEM/EDS spectra of the 2 clinkers OPC0 and OPC12.4

### 3.3 Calorimetric study

The hydration reactions of the cement paste are exothermic and can be monitored by following the rate of heat release using an isothermal calorimeter. Figure 8 shows the calorimetric curves for the three cement pastes with and without BA incorporation: OPC0, OPC5 and OPC12.4. It can be seen from the results that the three cement pastes have a similar curve shape. Three peaks were formed during the first 36 hours of hydration. The first peak corresponds to the dissolution of the anhydrous grains and appears immediately after adding water to cement. Then an induction period with low thermal activity was observed for the three cement paste before appearance of the second peak. The latter corresponds to the acceleration of the silicates and aluminates hydration and the formation of C-S-H and ettringite. A third peak is formed after the gypsum depletion and the transformation of ettringite to monosulfoaluminate hydrate.

By comparing the three curves a main difference can be detected related to the initial setting time. In fact the setting of OPC0 begins approximately 1 hour before OPC5 and OPC12.4. This could be due to the trace elements (i.e. Zn) that are present in the MSWI BA based clinkers as already shown in the XRF results (table 4). According to previous studies, Zn is well known to have a retarder effect on the



cement paste hydration [56][55]. In addition, the presence of alkalis such as Na could also induce a retardation effect [62].

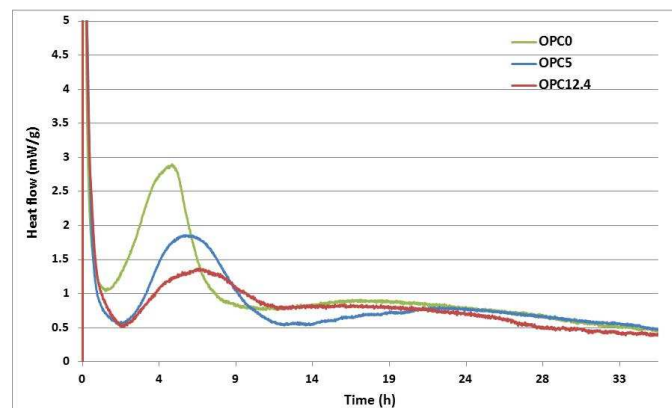


Figure 7 : The heat release for the three cement pastes: OPC0, OPC5 and OPC12.4

### 3.4 Mechanical performances

The compressive strength is measured in order to evaluate the mechanical performances of the produced cement. Figure 8 represents the compressive strength at 1, 7 and 28 days for the three cement pastes: OPC0, OPC5 and OPC12.4. These results show that at early age (1 day) the compressive strengths are lower when MSWI BA is added (19.5, 14 and 10.7 MPa for OPC0, OPC5 and OPC12.4 respectively). This could be related to the retardation effect observed by isothermal calorimetry at 1 day caused by the heavy metals and alkalis as previously shown in figure 8. This could be related also to the amount of  $C_3S$  in the clinker (table 4) that is slightly higher in the clinker containing less MSWI BA. In addition the presence of alkali such as sodium and potassium could also have a retardation effect on the hydration and influencing therefore the compressive strength [63]. This difference in the compressive strength of the three cement pastes is less observed from day 7 because the effect of the delay of the hydration appears at very short term. It can be concluded that using MSWI BA as a substitution for the raw materials for clinker production does not change significantly the compressive strength of the cement pastes especially after 7 days.

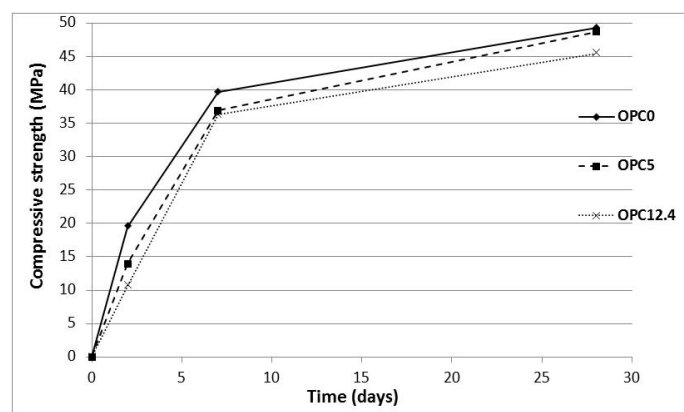
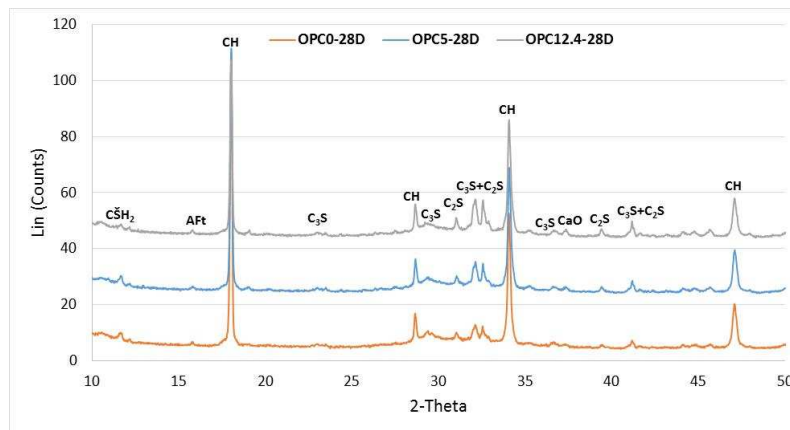


Figure 8: Compressive strength of the 3 cement pastes at 1, 7 and 28 days

The mineralogical assemblages of the hydrated cement pastes at 28 days were studied by XRD analysis after stopping the hydration (figure 9). The results show that the Portlandite CH is the main crystalline hydrate. This phase comes from the hydration of  $C_3S$  and  $C_2S$ . The patterns show also the presence of Ettringite AFt coming from the reaction of  $C_3A$  with gypsum.

The diffractograms of the 3 cement pastes (reference and the 2 cement pastes with MSWI BA) are similar. Therefore adding MSWI BA in the raw meal to produce Portland cement does not modify the mineralogical assemblage of the hydrated cement pastes.



**Figure 9: XRD analysis for the 3 cement pastes OPC0-28D, OPC5-28D and OPC12.4-28D at 28 days**

## 4 Conclusion

The aim of this paper is to recycle the MSWI BA from the north of France, in the raw meal to produce Portland clinker. To reach this target, MSWI BA was grinded and characterized in order to identify its composition. In addition to the reference clinkers made with chemical reagents, two other clinkers were synthesized: OPC5 with 5% of MSWI BA and OPC12.4 with 12.4% that represents the maximum amount of substitution. It has been shown in this study that:

- MSWI BA contains the main oxides -  $\text{CaO}$ ,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  - needed for the clinker production
- For the clinker composition chosen, the maximum amount of MSWI BA that could be incorporated in the raw meal is controlled by the  $\text{Fe}_2\text{O}_3$  amount. The addition of 12.4% of MSWI BA provides the total amount of iron oxide needed for the clinker production
- Adding MSWI BA in the raw meal for the clinker production does not affect the mineralogical composition comparing to the reference clinker. The main phases of Portland clinker –  $\text{C}_3\text{S}$ ,  $\text{C}_2\text{S}$ ,  $\text{C}_4\text{AF}$  and  $\text{C}_3\text{A}$  in addition to free lime are obtained for all produced clinkers
- The microstructure of the clinker with and without BA is quite similar despite the presence of more porosity in the MSWI BA based clinker which could be due to the clinkering process and the presence of some heavy metals
- The partial substitution of raw materials by MSWI BA provoked a delay of less than 1 hour in the setting time which could be attributed to the presence of heavy metals and alkalis.
- The difference between the compressive strength of MSWI BA-based clinkers and the reference are slight after 7 days of hydration.

Finally, this study reveals that the bottom ash could be recycled as a secondary raw material for clinker production. The amount of bottom ash that could be recycled depends mainly on its composition and the type of cement targeted.

In our case we conclude that the French bottom ash has different chemical composition from the ones studied in the literature (coming from Taiwan and Beijing [46] [47]). With this type of bottom ash, it is possible to recycle 12.4% in CEM I Portland cement after adapting the raw meal composition and without applying a treatment for the bottom ash.

Given the large variability of the bottom ash composition, due to the type and the origin of wastes that differs from a country to another, it is important to extend the study to investigate the variability of the bottom ash composition from a batch to another and the effect that could have on its possible use as a raw material for cement production. In addition it is interesting to follow in further studies the durability of the cement pastes made from MSWI BA based clinkers in order to have a complete vision on the behavior of these clinkers.

## Declaration of competing interest

The authors declare that they no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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