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- Production of Portland cement clinker from French Municipal Solid Waste Incineration Bottom Ash
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- 11

12 Abstract

13 The amount of bottom ashes generated from the municipal solid waste incineration is increasing 14 considerably. Therefore their recycling is necessary in order to limit the landfilling that presents 15 environmental issues. The aim of this paper is to recycle Municipal Solid Waste Incineration Bottom 16 17 18 19 20 21 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 22 23 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 24 25 26 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.

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

28 1 Introduction

29 The cement industry is responsible for more than 6% of the total anthropogenic greenhouse gases 30 emissions. In fact the production of Ordinary Portland Cement "OPC" generates 842 kg CO₂/t of 31 clinker [1][2][3][4][5]. In addition to its huge energy consumption, the cement industry uses large 32 33 34 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 CO2 35 emissions. Different types of wastes have been studied in the literature in order to produce cement, 36 and mainly OPC cement, such as red mud [10], steel slag [11], sediments [12][13], recycled concrete 37 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.

45 The MSWI bottom ash (MSWI BA) represents 75-80% of the produced ashes [26][27][28]. Nowadays, 46 the landfilling presents the main option for MSWI BA disposal [29][30]. However, the landfill causes the 47 pollution of the groundwater and contaminates the soil in addition to the limited land space and the 48 problems related to the disposal cost and the regulation [31][32][33]. The literature describes different 49 methods to recycle the MSWI BA in civil engineering. The latter has been used as supplementary 50 cementitious materials and pozzolanic material [29][27][34][35], as an aggregate replacement 51 [19][28][36][37] or in alkali activated materials as a precursor [20][38][39], as a road based material 52 and in road construction [40][41][42].

53 54 The chemical composition of MSWI BA contains CaO, SiO₂, Al₂O₃ and Fe₂O₃ [43][44]. This composition makes the use of MSWI BA, as a secondary raw material, for clinker production 55 possible[45]. Li et al. analyzed different fraction from MSWI BA from Beijing to produce a type II 56 Portland cement and shows that 9% of this waste could be added to the raw meal [46]. Pan et al. also 57 used MSWI fly ash and bottom ash, after washing with water/acid as a pre-treatment, to produce 58 clinker by incorporating 1.75 and 3.5% respectively in the raw meal [47]. The percentages of 59 incorporation were determined based on the chloride content in the ashes in order to respect the limit 60 of chloride of 100 ppm required by the cement plant in Taiwan. They showed that this incorporation did 61 not affect the compressive strength and the cement produced is conformed to the Chinese National 62 Standard of type II Portland cement [47].

63 It is important to note that the MSWI BA has a very heterogeneous composition depending on several 64 factors such as the waste origin, the incineration process, the incinerator used, the pre-treatment 65 applied, etc. [48][49][50]. Therefore the percentage of substitution in the clinker raw meal differs from 66 one to another MSWI BA. Until today, there are no studies dealing with the use of MSWI BA generated 67 in France, as a secondary raw material for clinker production. Therefore the aim of this study is to use 68 Municipal Solid Waste Incineration Bottom Ash from the north of France, as a raw material in the OPC 69 clinker production. First a reference OPC clinker is produced without adding MSWI BA. Then 2 70 substitution rates of MSWI BA are applied: 5% and a second one corresponding to the maximum 71 72 73 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.

74 2 Materials and methods

75 The materials used in this study for the clinker production are the chemical reagents: CaCO₃, SiO₂, 76 Al_2O_3 , Fe₂O₃ and the MSWI BA from the north of France.

77 2.1 MSWI BA characterization

The MSWI-BA used in this study originates from the recycling platform RMN-Pr'eFerNord 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 µm. The
mineralogical characterization was made by X-ray diffraction (XRD) using a diffractometer Bruker D2
with Cu Kα radiation. The X-ray patterns were acquired in the 20 (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.

89 2.2 Clinker production at laboratory scale

90 Three Portland clinkers are produced in the laboratory with different percentage of substitution by 91 MSWI BA. The first is a reference clinker referred by OPC0 produced using analytical reagents 92 (CaCO₃, SiO₂, Al₂O₃ and Fe₂O₃) only. The theoretical mineralogical composition of OPC0 is shown in 93 table 1. This composition was chosen according to the study of Gineys et al [8] in order to produce an 94 Ordinary Portland Cement.

95

Table 1 : Theoretical composition of the targeted Portland clinker

C₃S (wt. %)	C ₂ S (wt. %)	C₃A (wt. %)	C4AF (wt. %)	C (wt. %)
65%	18%	8%	8%	1%

96

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, AI, Fe), with the aim to respect the targeted cement criteria represented in table 1.

101 The first substitution rate in the clinker raw meal by MSWI BA corresponds to 5% (OPC5). The second 102 one corresponds to the maximal amount of MSWI BA that could be incorporated in the raw meal while 103 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 Fe₂O₃ content (OPC12.4). Adding more than 12.4% of MSWI BA (maximum value) in the raw meal provides more Fe₂O₃ than needed and consequently the resulting clinker composition differs from the theoretical composition of the targeted Portland clinker.

110

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

Constituents	CaCO₃ (wt. %)	SiO ₂ (wt. %)	Al ₂ O ₃ (wt. %)	Fe ₂ O ₃ (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

111

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 cm²/g. To produce cements, gypsum was added to the clinker using equation 4 [52]:

- 118 Amount of gypsum used = Al_2O_3 content * [0.6/101.96] * [80/0.461] (equation 4)
- 119 Where, $101.96 = \text{mol. wt. Al}_2O_3$; $80 = \text{mol. wt. SO}_3$; $0.461 = \text{mol. ratio SO}_3/\text{natural gypsum used (for pure gypsum 80/172 is 0.466)}$

121 2.3 Clinker and cement paste characterization

122 The mineralogy of the synthesized clinkers was studied using X-ray diffraction (XRD). A Bruker D2 123 with Cu K α radiation was used. The X-ray patterns were acquired in the 2 θ (10-80 °) with a step size 124 of 0.02 ° and 1 s per step. A Rietveld analysis was also conducted using TOPAS software in order to 125 quantify the crystalline phases.

- 126 The Microscopic observations were conducted on polished section of clinkers using SEM type Hitachi 127 S-4300SE/N. Local chemical analyses were conducted using energy dispersive X-ray spectrometer
- 128 (EDS) with a Thermo Scientific Ultra Dry EDS detector coupled with the SEM. The polished sections
- 129 were made by vacuum impregnating a clinker monolith in an epoxy resin. After hardening, the samples
- 130 were polished with ethanol and then coated with carbon.

131 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].

145 3 Results and discussions

146 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).

149

Table 3: Bottom ash composition

Element	LOI 950°C	0	Ca	Fe	Al	Si	Р	S	CI	Pb
Percentage (%)	13.99	34.06	18.91	9.45	5.57	11.06	0.76	0.94	0.25	0.17
Element	К	Na	Ti	Cr	Mn	Mg	Cu	Zn	Ва	
Percentage (%)	0.6	1.25	0.44	0.1	0.19	1.13	0.24	0.63	0.11	
	soluble salts (mg/kg)									
	<	<20								
	2	2740								
Sulphate					5059					

150

151 The X-ray diffraction (figure 1) for MSWI BA shows that the principal phases are quartz, calcite, with 152 some minor phases such as hematite, gehlenite etc.



153 154

Figure 1: X-ray pattern for MSWI BA

155 The particle size distribution of MSWI BA is represented in figure 2. The results show that 90% of 156 particles are inferior to 70 μ m. In fact the finesse of the raw materials is important in order to have a 157



158 159

Figure 2: Particle size distribution for MSWI BA

160 3.2 Clinkers characterization

161 3.2.1 Mineralogical and chemical analyzes

162 The OPC0 reference clinker characterization by XRD is presented in figure 3. The result shows the 163 presence of the 4 crystalline phases of a Portland clinker, C₃S, C₂S, C₄AF and C₃A. The pattern shows

164 also that no secondary mineral phases are produced. Therefore the raw meal composition, the clinker 165 preparation process at the laboratory scale and the burning temperature are suitable for this study.



Figure 3: XRD patterns of the reference clinker OPC0

168 The MSWI BA based clinkers OPC5 and OPC12.4 are characterized and compared to the reference 169 clinker. The results are shown in the figure 4. As it can be seen from the patterns, the 2 MSWI BA 170 based clinkers, OPC5 and OPC12.4 present the same mineralogical phases as the reference clinker 171 OPC0. Consequently the partial substitution of the raw materials by MSWI BA does not modify the 172 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].

181

Table 4: XRF analyzes for the 3 produced clinkers

Element (%)	L.O.I (%)	0	Ca	Si	Fe	AI	Na	Mg	Р	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

182 In order to quantify the crystalline phases in the 3 synthetized clinkers, Rietveld analysis was 183 conducted. The results are represented in the table 5 and show that adding MSWI BA in the raw meal 184 produces a clinker with slightly less C₃S comparing to the reference clinker. This could be due to the 185 consideration in the calculation method that the four clinker phases are in their stoichiometric state 186 which is not quite realistic especially when incorporating wastes into the raw meal where the phases 187 can accommodate substitute ions [57]. Same results was obtained by Clavier et al. 2021 [43] when 188 incorporating bottom ash in the clinker. This was attributed to the operational variations associated 189 with lab-scale clinker production. In fact the C₃S tends to revert to C₂S when clinker is cooled too 190 slowly and therefore the speed of cooling showed be considered. In addition the Portland cement 191 chemistry is complex, therefore this complexity increases when a replacing the raw materials by a 192 heterogeneous material such as MSWI BA. The latter contains, as shown in table 3, some metallic 193 elements (i.e. Cu, Zn, Mg, Cl...) that could cause a reduction in the mineralization temperature, 194 favoring therefore the formation of C₂S over the C₃S phase[58]. Concerning the residual lime, its 195 content is between 0.05% and 0.21% (table 5) which confirms that the clinkering was complete 196 because these values are lower than what is specified in the standard NF EN 196-2. In addition, 197 according to the standard there is no risk due to these low free lime contents (<0.5%).

198

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

	C₃S (wt. %)	C ₂ S (wt. %)	C4AF (wt. %)	C₃A (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

199

200 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].



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207

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, C4AF (point 1), C3A (point 2), C2S (point 3) and C3S (point 4) were identified. The alite grains appeared to be angular whereas the belite one round. The oligibar with and without PA present in general almost a similar microstructures.

212 clinkers, with and without BA, present in general almost a similar microstructures.



213 214

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

215 3.3 Calorimetric study

216 The hydration reactions of the cement paste are exothermic and can be monitored by following the 217 rate of heat release using an isothermal calorimeter. Figure 8 shows the calorimetric curves for the 218 three cement pastes with and without BA incorporation: OPC0, OPC5 and OPC12.4. It can be seen 219 from the results that the three cement pastes have a similar curve shape. Three peaks were formed 220 during the first 36 hours of hydration. The first peak corresponds to the dissolution of the anhydrous 221 grains and appears immediately after adding water to cement. Then an induction period with low 222 thermal activity was observed for the three cement paste before appearance of the second peak. The 223 latter corresponds to the acceleration of the silicates and aluminates hydration and the formation of C-224 S-H and ettringite. A third peak is formed after the gypsum depletion and the transformation of 225 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

234 3.4 Mechanical performances

235 The compressive strength is measured in order to evaluate the mechanical performances of the 236 produced cement. Figure 7 represents the compressive strength at 1, 7 and 28 days for the three 237 cement pastes: OPC0, OPC5 and OPC12.4. These results show that at early age (1 day) the 238 compressive strengths is lower when MSWI BA is added (19.5, 14 and 10.7 MPa for OPC0, OPC5 239 and OPC12.4 respectively). This could be related to the retardation effect observed by isothermal 240 calorimetry at 1 day caused by the heavy metals and alkalis as previously shown in figure 8. This 241 could be related also to the amount of C_3S in the clinker (table 4) that is a slightly higher in the clinker 242 containing less MSWI BA. In addition the presence of alkali such as sodium and potassium could also 243 have a retardation effect on the hydration and influencing therefore the compressive strength [63]. 244 This difference in the compressive strength of the three cement pastes is less observed from day 7 245 because the effect of the delay of the hydration appears at very short term. It can be concluded that 246 using MSWI BA as a substitution for the raw materials for clinker production does not change 247 significantly the compressive strength of the cement pastes especially after 7 days.





249

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.





265

266

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

259 Conclusion 4

260 The aim of this paper is to recycle the MSWI BA from the north of France, in the raw meal to produce 261 Portland clinker. To reach this target, MSWI BA was grinded and characterized in order to identify its 262 composition. In addition to the reference clinkers made with chemical reagents, two other clinkers 263 were synthetized: OPC5 with 5% of MSWI BA and OPC12.4 with 12.4% that represents the maximum 264 amount of substitution. It has been shown in this study that:

- MSWI BA contains the main oxides CaO, SiO₂, Al₂O₃, Fe₂O₃ needed for the clinker production
- 267 For the clinker composition chosen, the maximum amount of MSWI BA that could be 268 incorporated in the raw meal is controlled by the Fe₂O₃ amount. The addition of 12.4% of 269 MSWI BA provides the total amount of iron oxide needed for the clinker production
- 270 Adding MSWI BA in the raw meal for the clinker production does not affect the mineralogical 271 composition comparing to the reference clinker. The main phases of Portland clinker $-C_3S$, 272 C₂S, C₄AF and C₃A in addition to free lime are obtained for all produced clinkers
- 273 The microstructure of the clinker with and without BA is quite similar despite the presence of 274 more porosity in the MSWI BA based clinker which could due to the clinkering process and the 275 presence of some heavy metals 276
 - 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.
- 277 278 The difference between the compressive strength of MSWI BA-based clinkers and the 279 reference are slight after 7 days of hydration.
- 280 Finally, this study reveals that the bottom ash could be recycled as a secondary raw material for 281 clinker production. The amount of bottom ash that could be recycled depends mainly on its composition and the type of cement targeted. 282
- 283 In our case we conclude that the French bottom ash has different chemical composition from the 284 ones studied in the literature (coming from Taiwan and Beijing [46] [47]). With this type of bottom 285 ash, it is possible to recycle 12.4% in CEM I Portland cement after adapting the raw meal 286 composition and without applying a treatment for the bottom ash.
- 287 Given the large variability of the bottom ash composition, due to the type and the origin of wastes 288 that differs from a country to another, it is important to extend the study to investigate the 289 variability of the bottom ash composition from a batch to another and the effect that could have on 290 its possible use as a raw material for cement production. In addition it is interesting to follow in 291 further studies the durability of the cement pastes made from MSWI BA based clinkers in order to 292 have a complete vision on the behavior of these clinkers.

293 **Declaration of competing interest**

- 294 The authors declare that they no known competing financial interests or personal relationships that 295 could have appeared to influence the work reported in this paper.
- 296

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