



Application of thermally activated municipal solid waste incineration (MSWI) bottom ash fines as binder substitute



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ABSTRACT

Untreated municipal solid waste incineration (MSWI) bottom ash fines (0–2 mm) have poor pozzolanic properties, and contain substances which can pose an environmental risk (e.g. heavy metals and salts). This study investigates combined treatments applied on bottom ash fines (BAF) to increase their reactivity. The treated BAF is compared with both untreated BAF and cement, and its contribution to cement hydration is investigated. Additionally, the utilization of the treated BAF in mortar as cement replacement is tested. Finally, the leaching properties of mortars containing treated and untreated BAF are estimated. According to the results obtained, the 28-day compressive and flexural strengths of mortar with 30% treated bottom ash are about 16% and 6% lower than the reference mortar, respectively. The leaching of contaminants from the crushed mortars with BAF are well under the limit values imposed by Dutch legislation.

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1. Introduction

Cement clinker production contributes highly to the worldwide CO₂ emission and continuous consumption of raw materials [1,2]. Considering the protection of the environment and sustainable development as a global focus, a reduction of the production and use of cement worldwide is meaningful. In recent years, a significant amount of research has been done on this topic, in which the application of secondary materials as cement replacement attracted extensive attention [3–6]. Furthermore, the successful use of fly ash and ground blast furnace slag in concrete promotes the research focus on other secondary materials, such as incineration wastes [7–10].

Municipal solid waste is incinerated to reduce its mass and volume (by 70% and 90%, respectively); meanwhile, energy is produced in the form of heat which can be applied for the generation of electricity [11,12]. The incineration of solid waste as an effective way to recover energy and decrease the demand of landfill attracts widespread attention [13–17]. However, a considerable amount of solid residues is produced after incineration (typically fly ash, air pollution control ash, bottom ash etc.), of which bottom ash

accounts for about 80% by weight [18]. Previous studies on the characteristics of municipal solid waste incineration (MSWI) bottom ash indicated its potential to be applied as building materials substitute in concrete, as cementitious material, sand or aggregates [19–26]. Nevertheless, it is also reported that the application of bottom ash as building material is hindered from the points of view of its environmental and mechanical properties. As an incineration residue, bottom ash contains certain amounts of heavy metals and salts (such as Sb, Mo, Cu, chlorides and sulphates) which pose an environmental risk [26,27]. Hence, the amounts of these heavy metals and salts need to be reduced before application in the construction field [28,29]. Furthermore, previous research also mentioned the expansion or swelling of concrete caused by the metallic aluminium contained in bottom ash when it is used as aggregate [30,31]. Therefore, a number of commonly used treatments are applied to upgrade the quality of MSWI bottom ash, which can clean the coarse bottom ash sufficiently to make its properties comply with the legislation, such as physical separation, washing treatment, weathering, etc. [32–34]. However, for the fine bottom ash (particle size smaller than 2 mm) which contain more leachable contaminants, these methods are not sufficient or cost effective because of their higher surface area and concentration of contaminants.

Several studies focused on the utilization of MSWI waste as

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binder, and it is reported that the bottom ash contributes very little to the cement hydration because of its low pozzolanic properties. According to the research of Li et al. [35], 30% of cement replacement by bottom ash reduces the compressive strength of mortars by about 26% after 28 days. However, the leaching properties of the concrete with MSWI bottom ash as binder are well under the limit value of Chinese legislation (GB 5085.3 [36]), which indicates the immobilization of the contaminants in concrete [37]. Therefore, it is considered that, if the reactivity of the MSWI bottom ash can be increased, its application as binder substitute would be enhanced.

According to previous research, thermal treatment on fine MSWI bottom ash is an effective way of increasing its reactivity, as well as reducing its leaching concentration of heavy metals. As reported by Qiao et al. [38], the milled bottom ash is an inert material and thermal treatment at 800 °C decreased the loss on ignition (LOI) and decomposed calcite, subsequently increasing the reactivity of bottom ash by generating new active phases (mayenite and gehlenite). Furthermore, thermal treatments of bottom ash are also proven to be efficient to reduce the leaching of heavy metals (such as Cu and Pb), by reducing the content of organics which have been linked to higher leaching of these elements [39,40]. Another connection can be made between the pH of the leachates of thermally treated samples and the variation in leaching of a number of elements, such as Mo, Sb, and Cu [41–45].

There are multiple studies on the properties, treatment and application of MSWI wastes [8,12,13,19,34]. However, research focused on the treatment and activation of MSWI bottom ash fines (0–2 mm) is scarce. Hence, the aim of this study is to evaluate the activation of MSWI fine bottom ash (0–2 mm) by thermal treatment, and its application as cement substitute in mortar. The characteristics of the treated bottom ash samples are determined by means of X-ray diffraction (XRD), X-ray fluorescence (XRF) and scanning electron microscope (SEM). Additionally, isothermal calorimetry is applied to investigate the hydration of the cement with treated bottom ash additions. Furthermore, the mechanical properties of mortars with treated bottom ash are analysed after different curing times. Finally, the environmental influence of the bottom ash-containing mortar is evaluated through leaching tests.

2. Materials and methods

2.1. Materials and treatments

The MSWI bottom ash (BA) was provided by a waste-to-energy plant (Attero) in the Netherlands, which has an annual incineration capacity of around 800,000 tons. The grid temperature is about 1000–1300 °C, with 3 separation lines of paper and plastic before the burning of the waste, which will be sent to special factories for reuse. After incineration, the solid residue that comes out from the boiler is quenched in water, followed by the ferrous and non-ferrous material removal by electromagnetic and eddy current separation, respectively. After separation, the residue is collected and termed MSWI bottom ash. In this study, the bottom ash fines below 2 mm (BAF) from the received bottom ash were chosen to be investigated, because of the higher concentration of contaminants compared with coarser particles.

The BAF was milled into powder to reduce its particle size and make it more homogenous using the planetary ball mill. Thermal treatment was performed on the BAF and the milled BAF in the muffle furnace, and the temperatures chosen were 550 °C and 750 °C, based on the research in Refs. [37,38], the heating capacity of the incinerator, as well as the temperature used to determine the loss on ignition (LOI) [46]. Thermal treatment at 550 °C was performed on the BAF before and after milling separately to investigate the influence of treatment order on the bottom ash properties.

It is hypothesized that the BA particles are covered by dust-like powders, which contain higher amounts of soluble contaminants, and can have a higher reactivity than the particle itself. In order to separate the fine powder attached on the surface of bottom ash particles for investigation, lower speed and short duration of milling were applied during the milling procedure. The treatments procedure is described as follows:

- Method 1 is to mill the bottom ash using a planetary ball mill with fixed milling duration and speed in order to reduce the particle size to a maximum 125 µm; the dark-grey powder obtained is labelled as M/BA.
- Method 2 and Method 3 are to thermally treat the milled bottom ash after Method 1 at 550 °C and 750 °C, respectively; reddish-brown powders are obtained, labelled as M5T/BA and M7T/BA, respectively.
- Method 4 is to heat the bottom ash particles at 550 °C firstly and then mill them using the same milling procedure as in Method 1, listed as 5TM/BA.
- Method 5 is to mill the thermally treated bottom ash (at 550 °C) for a shorter duration and lower speed compared to Method 1, followed by a sieving step to collect the material below 63 µm (5TMS/BA); particles above 63 µm are labelled as 5TMS+63/BA. Due to the fact that the dust/powder attached on the bottom ash particles could not be removed manually one by one or by machine evenly, the lower speed milling procedure was used in order to remove the attached powder from the particle surface by friction between the particles. The fines below 63 µm are chosen to be investigated in order to distinguish the powder from the crushed bigger particles during the milling.

The OPC used in this study was CEM I 52.5 R from ENCI HeidelbergCement (the Netherlands) and the sand was CEN normalized sand 0–2 mm according to EN 196-1 [47].

2.2. Analysis of material properties

The chemical compositions of the treated bottom ash and cement were determined by X-ray fluorescence (XRF). The X-ray diffraction (XRD, Cu tube, 40 kV, 30 mA, 3–75°, 0.02°/step, 0.2°/min, variable divergence slits V20) was applied to observe the crystalline phases present in the material.

The particle surface properties of the investigated materials were observed by scanning electron microscope (SEM, Quanta 650 FEG, FEI) and optical microscopy.

The particle size distribution (PSD) of all materials considered in this study was measured by laser diffraction (Mastersizer 2000 Malvern), and a helium pycnometer (AccuPyc II 1340) was used to measure the specific densities.

The amount of metallic aluminium in the treated bottom ash was evaluated with a method measuring the gas volume generated upon the chemical reaction of metallic aluminium with an alkaline solution; the setup for collecting the generated hydrogen is similar to the one described in Ref. [48].

2.3. Hydration properties study

The effect of the treated bottom ash on the cement hydration was investigated using an isothermal calorimeter (eight-channel TAM Air, Thermometric). Previous studies on the bottom ash show that its use as cement replacement should be limited under 30% [21,25,35]. Therefore, the treated bottom ash is mixed with cement with the mass ratio of 3:7, and the water to binder ratio is kept at 0.7, to facilitate the loading of the samples into the ampoules. Cement pastes containing different treated bottom ashes were

cured in water at 20 °C for 7 days, and subsequently small pieces were extracted from the hardened samples and dried with acetone. The 7 days samples were milled and the crystalline phases were investigated by XRD.

2.4. Mechanical strength of mortar samples

The treated bottom ash is pre-mixed with cement in a 3:7 ratio, and the mortar samples were prepared according to EN 196-1 [47]. The flowability of the fresh mortars was determined using the flow table test according to EN 1015-3 [49]. The fresh mortar was cast in 40 mm × 40 mm × 160 mm prism moulds and demolded after 24 h, and then cured in water at room temperature. The compressive and flexural strengths of mortar samples after different curing ages were tested (AUTOMAX 5). The obtained values were expressed as the averages of three samples.

2.5. Environmental impact evaluation

To estimate the environmental impact of the bottom ash before and after the treatments, as well as of the mortars containing bottom ash, leaching tests according to Dutch standard NEN 7383 (2004) [50] were conducted. The mortars containing bottom ash were crushed after 28 days curing to a fraction below 4 mm for the column leaching test. The liquid to solid ratio for the leaching test is 10 l/kg; the material was placed in a column container and water was forced to flow through the material from the bottom to top for a certain period. The concentration of chemical elements in the eluate was analysed using inductively coupled plasma-atomic emission spectrometry (ICP-AES) according to NEN 6966 [51], and the amount of chloride and sulphate was determined through high performance liquid chromatography (HPLC) following NEN-EN-ISO 10,304-2 [52]. The leaching values of studied elements were compared with the limit value (Table 4) according to the Dutch legislation [53]. The reflux destruction by HCl/HNO₃ on the investigated bottom ash samples were performed according to NEN 6961 [54] and then the concentration of Cu and Mo in the extracted solution were determined using inductively coupled plasma-atomic emission spectrometry (ICP-AES) according to NEN 6966 [51]. The amount of Sb was determined using atomic absorption spectroscopy (AAS) according to NVN 7323 [55]; the amounts of chlorides and sulphates were determined according to NEN-EN 1744-5 [56] and NEN-EN 1744-1 [57], respectively.

3. Results and discussions

3.1. Characteristics of materials

As presented in Fig. 1 (a) and according to the previous study [26], the raw BAF is a fraction with a similar particle size to sand, and has a dark-grey colour. There are quartz particles, iron, glass particles, as well as heterogeneous particles visually observed in bottom ash. Generally these BAF particles are covered by a dust-like layer. It is noticed that after the thermal treatment the colour changed to reddish and iron oxide particles could be observed. The dust-like layer of particles still exists on the surface of the particles, as shown in Fig. 1 (b). According to a previous study [58], after water quenching, the bottom ash particles consist about 60% particles which were coated by fine particles and other melted phases. This coating on the bottom ash particles results from the reaction occurred during the rapid water quenching process [59], which influence the leaching properties of the bottom ash. After treatment following Method 5, the bottom ash particles under 63 μm account for 34.7% wt. of the milled bottom ash (compared to the initial 2.4% wt.). The particles above 63 μm shown in Fig. 2 have

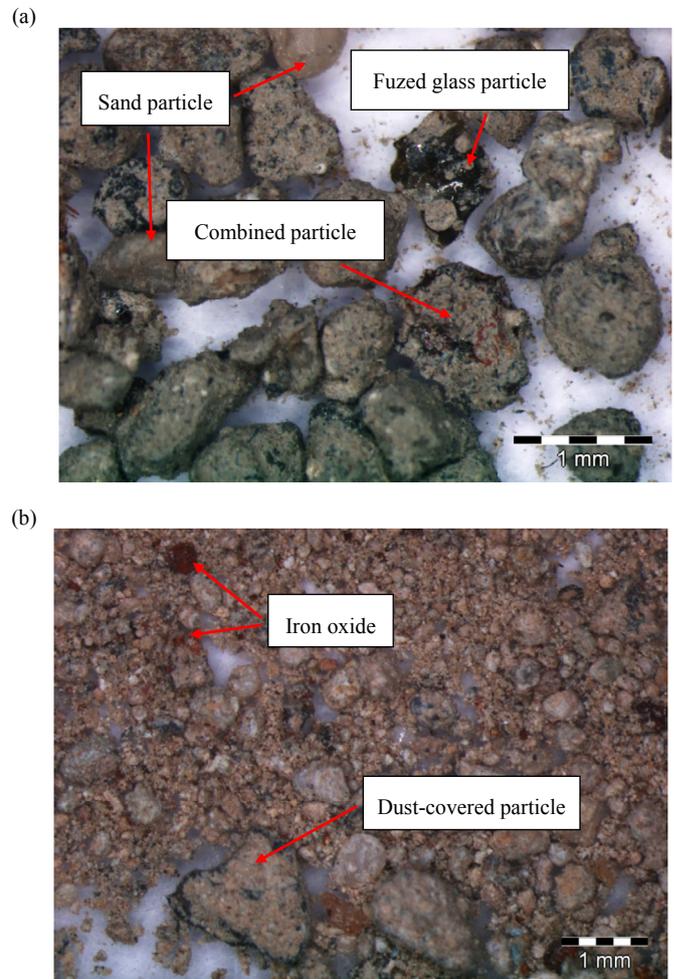


Fig. 1. MSWI bottom ash particles (a) before and (b) after 550 °C thermal treatment.



Fig. 2. BAF after treatment following method 5 (particles above 63 μm).

relatively cleaner surface than untreated and thermally treated bottom ashes (Fig. 1), as the attached dust-like layers on the surface were partially removed by the friction during milling. Furthermore, these particles are predominantly sand, glass, iron particles, and some plate-shaped metals were also observed (Fig. 3 is shown as an example).

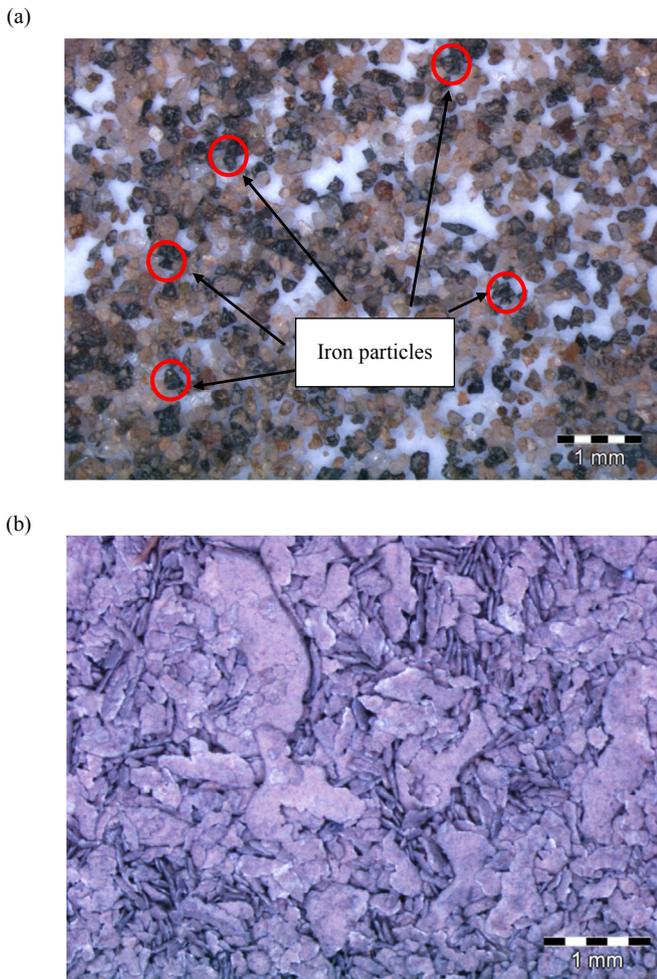


Fig. 3. (a) Iron particles and (b) plate-shaped metals sieved out in bottom ash particles above 180 μm after treatment method 5.

3.1.1. Chemical properties

The chemical compositions of the cement and treated bottom ashes are presented in Table 1. The treated bottom ashes consist of the same main oxides as cement (as assumed by XRF), and their

main constituents belong to the $\text{SiO}_2\text{--CaO--Al}_2\text{O}_3\text{--Fe}_2\text{O}_3$ system, which in total accounts for about 70%–90% wt. of the bottom ash [35]. However, the treated bottom ashes contain higher amounts of SiO_2 and less CaO than cement, and the content of Al_2O_3 and Fe_2O_3 is relatively high. A considerable amount of alkali and alkali-earth oxides, such as Na_2O and K_2O , MgO etc. exists in the treated bottom ashes. Similar results are reported elsewhere [22,60].

Comparing the samples M/BA, M5T/BA, M7T/BA, it can be observed that the content of Cl slightly decreased after the thermal treatment; moreover, the higher temperature leads to a higher decrease of its content. This result is consistent with the finding shown in Refs. [59,61]. In addition, this decrease is more noticeable when the thermal treatment is undertaken after the milling of bottom ash. As reported by Yang et al. [59] the Cl content is relatively higher in the heterogeneous particles in bottom ash formed during the water quenching procedure, which are unstable under thermal conditions. Therefore, the decomposition of quenched phases in these heterogeneous particles during the heating released the evaporable Cl. Moreover, the organic matter is burnt out during the thermal treatment, thus the organic-associated Cl [61] is evaporated. Furthermore, the milling increases the particle surface area, leading to the even and efficient heating of the samples, subsequently enhancing the Cl evaporation from the samples.

As expected, due to the larger specific surface area, the pre-milling before heating will increase the thermal treatment efficiency, proved by the lower loss on ignition of M5T/BA and M7T/BA than the one of 5TM/BA. In this study the difference between samples which were pre-milled and which were directly thermally treated is quantified through metallic Al content determination and mechanical strength of hardened mortars.

Differently from other treated bottom ash samples in this study, 5TMS/BA has a relatively higher content of CaO and Al_2O_3 , and lower content of SiO_2 and Fe_2O_3 . Moreover, its Cl and SO_3 contents are also the highest among all the studied bottom ash samples. According to the literature [62,63], the chemical compositions of MSWI bottom ash differ by size fraction, especially for Si_2O and CaO . The finer bottom ash particles have a higher content of SiO_2 and a lower CaO content, and this is further confirmed by comparing 5TMS/BA and 5TMS+63/BA. Additionally, the content of Cl and SO_3 is almost 1.5 times of that in other treated bottom ash samples in this study. Also, the dust-like layer removed from the bottom ash particles contains a higher amount of Cl than the bottom ash particles themselves, which is in line with the findings in

Table 1

Chemical compositions and densities of CEM I 52.5 R, M/BA, M5T/BA, M7T/BA, 5TM/BA, 5TMS/BA and 5TMS+63/BA obtained by XRF.

| Chemical compositions [% wt.] | CEM I 52.5 R | M/BA | M5T/BA | M7T/BA | 5TM/BA | 5TMS/BA | 5TMS+63/BA |
|--------------------------------|--------------|-------|--------|--------|--------|---------|--------------|
| SiO_2 | 17.11 | 35.98 | 40.55 | 41.20 | 35.80 | 24.49 | 50.74 |
| CaO | 64.99 | 19.34 | 19.49 | 20.03 | 21.67 | 28.29 | 16.02 |
| Al_2O_3 | 3.80 | 9.00 | 9.44 | 9.82 | 9.93 | 11.07 | 8.64 |
| Fe_2O_3 | 3.59 | 11.54 | 11.74 | 12.09 | 12.26 | 8.99 | 13.48 |
| MgO | 1.56 | 1.81 | 1.97 | 2.09 | 1.99 | 2.26 | 1.92 |
| Na_2O | 0.00 | 1.35 | 1.49 | 1.89 | 1.48 | 1.37 | 1.59 |
| K_2O | 0.16 | 1.15 | 0.94 | 1.09 | 1.10 | 1.22 | 1.06 |
| CuO | 0.02 | 0.43 | 0.50 | 0.53 | 0.55 | 0.65 | 0.48 |
| ZnO | 0.15 | 0.80 | 0.79 | 0.82 | 0.91 | 1.16 | 0.71 |
| P_2O_5 | 0.63 | 1.02 | 1.07 | 1.14 | 1.13 | 1.12 | 1.12 |
| TiO_2 | 0.27 | 1.11 | 1.13 | 1.16 | 1.15 | 1.31 | 1.14 |
| MnO | 0.09 | 0.16 | 0.17 | 0.17 | 0.18 | 0.19 | 0.17 |
| Cl | 0.02 | 0.66 | 0.64 | 0.56 | 0.74 | 1.06 | 0.43 |
| SO_3 | 3.96 | 4.95 | 5.24 | 5.23 | 5.87 | 8.82 | 2.51 |
| Other | 2.01 | 0.69 | 1.19 | 0.71 | 1.31 | 1.10 | ^a |
| LOI (550 °C) | 0.41 | 7.37 | 2.02 | 1.16 | 2.86 | 2.54 | ^a |
| LOI (950 °C) | 1.64 | 9.99 | 3.66 | 1.46 | 3.93 | 6.90 | ^a |
| Density [g cm^{-3}] | 3.10 | 2.74 | 2.91 | 2.97 | 2.87 | 2.85 | ^a |

^a Not measured.

Refs. [60,64]. The fine bottom ash particles have higher amounts of chlorides and sulphates due to the large specific surface and are aggregated on the surface of coarse bottom ash particles during the water cooling procedure, as shown in Fig. 1(a). As also explained in Ref. [58], the newly formed phases on the surface layer during water cooling and weathering enhanced the absorption sites of chloride and sulphate as well. It is commonly known that the fine particles contain higher amount of contaminants due to their large specific area. However, the comparison of 5TMS/BA and 5TMS+63/BA shows that the finer particles or dust layers sticking on the particle surfaces contribute to the higher amount of contaminants in the BAF. The same trend is observed for heavy metals (details are discussed in Section 3.4). Hence, the prevention of the finer dust sticking on the coarse particle might decrease the content of contaminants in MSWI bottom ash.

Fig. 4 illustrates X-ray diffractograms of the investigated samples. The identified predominant crystalline phases are quartz (SiO_2), anhydrite (CaSO_4), calcite (CaCO_3), hematite (Fe_2O_3) and magnetite (Fe_3O_4). The samples after thermal treatment have a relatively higher peak at about 26.6° , which represents the main quartz peak. This result is consistent with the investigation of Qiao et al. [38]. Furthermore, the peak heights corresponding to hematite and magnetite increased, especially in sample M7T/BA. This can be attributed to the fact that heating results in the oxidation of iron in BAF (as shown in Figs. 1 and 3(a)), and higher temperature could enhance this oxidation. It is noticed that the calcite peaks decreased after the 550°C thermal treatment, and are not present in sample

M7T/BA, which results from the decomposition of calcite at high temperature [65,66].

Qualitatively, no significant differences can be observed between 5TMS/BA, 5TM/BA and M5T/BA in the X-ray diffractograms. Nevertheless, in 5TMS/BA, the quartz peak height is the lowest, which is in line with the chemical compositions determined in this study (in Table 1, where 5TMS/BA is shown to have 24.5% SiO_2 , compared to 35.8% and 40.5% for 5TM/BA and M5T/BA, respectively). In addition, the peaks corresponding to anhydrite (CaSO_4) and calcite (CaCO_3) here are much higher than in other samples. Based on the chemical compositions of this sample, it could be concluded that part of the higher amount of the CaO (about 28.3% compared to 16–21.7% in other BAF samples in Table 1) can be attributed to the anhydrite (CaSO_4) and calcite (CaCO_3), and the high amount of SO_3 (about 8.8% compared to 2.5–5.9% in other BAF samples in Table 1) can be related to the amount of anhydrite.

To summarize, the BAF mainly contains crystalline phases like quartz, anhydrite, calcite and hematite, and there is no significant change after the 550°C thermal treatment. However, higher temperatures (around 750°C) enhance the iron oxidation and decomposition of calcite in BAF. Samples 5TMS/BA, consisting of the fine powder in BAF and the dust-like layer on the particles, possess lower quartz and higher calcite and anhydrite contents and they also contain higher concentration of Cl and SO_3 . Hence, a feasible method to prevent or separate the fine powders sticking on the coarse bottom ash particles is an interesting topic.

3.1.2. Metallic Al

According to previous research [30,48,67], the metallic Al in bottom ash can react with an alkaline solution and generate H_2 , eventually leading to cracks in concrete and high macro porosity in the cement matrix. However, the damage the gas causes in the structure depends on its generation rate and the available amount of metallic Al in the sample. Hence, in this study, the amount of metallic Al in the investigated samples is determined. The results are presented in Table 2.

It is apparent that the metallic Al content did not change significantly after the thermal treatment. M7T/BA has slightly less metallic Al than 5TM/BA. This should be due to the fact that the higher temperature enhances the oxidation of the metallic Al.

5TMS/BA contains relatively less metallic Al than all the other investigated samples. This can be due to a number of reasons: firstly, the metallic Al in the original bottom ash particles under $63\ \mu\text{m}$ is quite easily oxidized during the heating period; secondly, due to the good ductility of the metallic Al, it is deformed into plate-shape through the action of the milling balls (as shown in Fig. 3(b)), thus enlarging its surface which consequently could be sieved out. Therefore, to consider the removal and recovery of metallic Al in the fine bottom ash fraction, special milling equipment should be used, such as an impact mill.

3.1.3. Physical properties

Fig. 5 illustrates the SEM images of M5T/BA and 5TMS/BA as examples. It can be seen that most of the treated bottom ash particles have irregular shapes, which is a typical characteristic of the milled bottom ash. Moreover, the surface of the particles has flocculent matter attached to the particles, as reported in Ref. [35].

The specific densities of the samples are shown in Table 1. M/BA

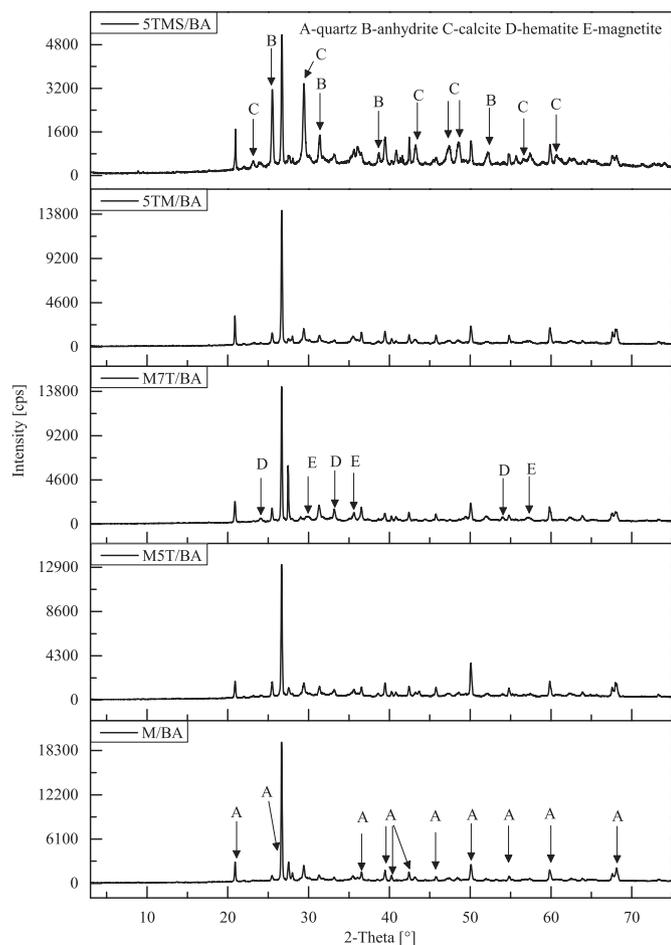


Fig. 4. XRD diffractograms of the treated bottom ash samples.

Table 2
Metallic aluminium content of bottom ash samples.

| Sample | M/BA | 5TM/BA | M7T/BA | 5TMS/BA |
|----------------------------|------|--------|--------|---------|
| Metallic Al [% wt. sample] | 0.44 | 0.48 | 0.46 | <0.2 |

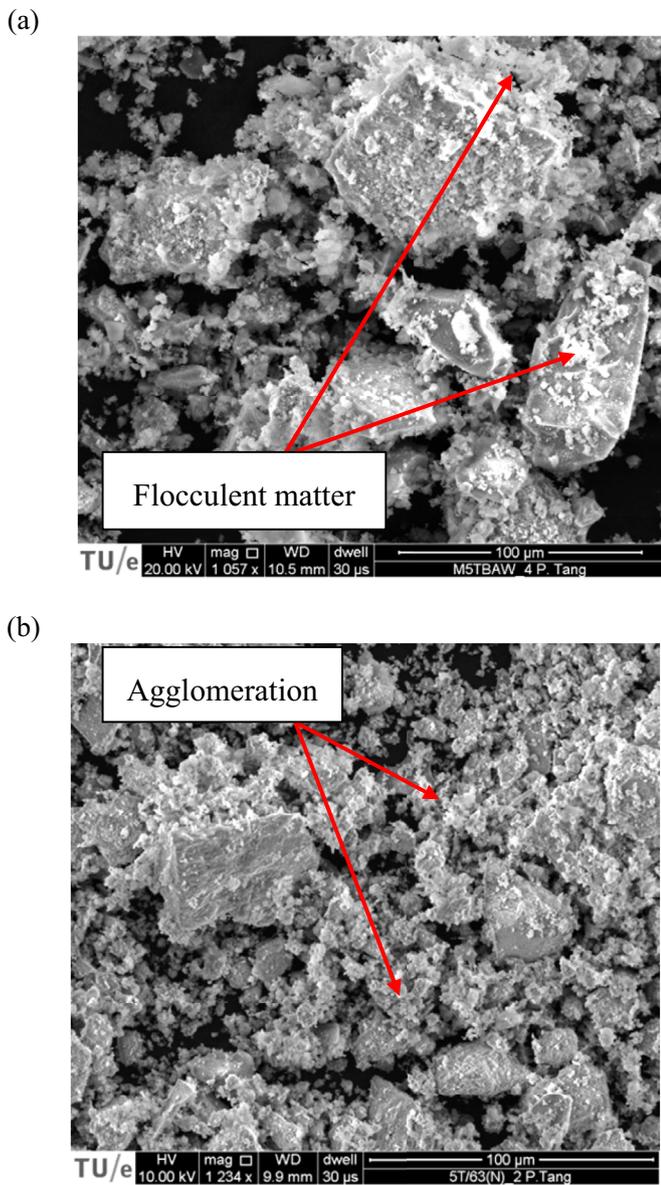


Fig. 5. SEM images of (a) M5T/BA and (b) 5TMS/BA.

has a slightly higher density than normal sand which is about 2.65 g/cm^3 . This is due to the fact that there are metal and ceramic particles in the BAF which could not be separated completely. The densities of thermally treated bottom ash increased. Moreover, a higher treatment temperature results in a higher density. The variation of the density after thermal treatment can be attributed to the combined effect of two opposing processes: burning the organic matter and the oxidation of the metals. The density of 5TMS/BA is lower than other thermally treated samples in this study, which could be due to the removal of iron and quartz particles during sieving (Fig. 3), as previously explained.

Fig. 6 presents the particle size distribution of the analysed materials. It can be observed that all the samples are coarser than cement powder, 5TM/BA excepted. Moreover, the thermal treatment on milled samples results in a coarse particle size, which is due to the agglomeration of fine particles during the thermal treatment [48]. In addition, the flocculent matter on the particles contributes partly to this phenomena (Fig. 5(a)). According to the measured PSD, sample 5TMS/BA has coarser particles compared

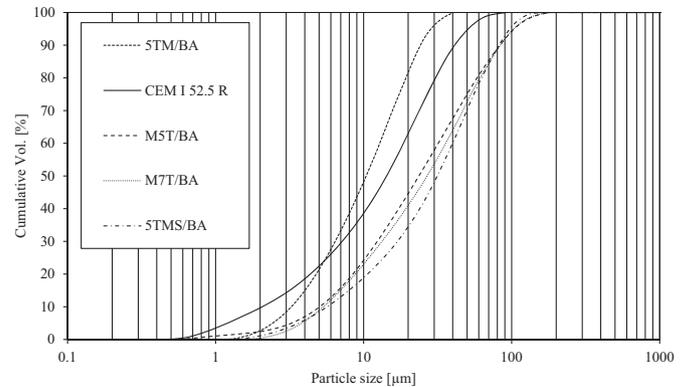


Fig. 6. The particle size distributions (PSDs) of the investigated materials.

with other bottom ash samples analysed in this study. However, this is a surprising result, because the 5TMS/BA only contains particles passing the $63 \mu\text{m}$ sieve. This could be because of the agglomeration of the powder (Fig. 5(b)), which in turn can be caused by the electrostatic interaction of particles.

To summarize, the order of milling and thermal treatment affects the particle size distribution of the samples. The high temperature of the thermal treatment on BAF contributes to the decomposition of calcite. Moreover, short time and a lower speed milling process, plus sieving, could reduce the metallic aluminium content in the chosen sample.

3.2. Hydration properties of bottom ash-cement mixes

In order to evaluate the influence of the thermally treated bottom ash samples and their contribution to the cement hydration, the heat development of cement with treated bottom ash (BA) during the hydration process are studied by isothermal calorimetry. The heat evaluation of the BA-cement mix, which is calculated on the basis of the total powder mass (cement and treated or untreated bottom ashes), is depicted in Fig. 7.

The hydration process of cement is typically divided into four periods, namely the initial stage, induction stage, acceleration stage and deceleration stage. Fig. 7(a) shows the heat flow of the investigated samples. It can be seen that the samples with treated or untreated bottom ashes have lower second maximum heat release rates (SMHR) than plain cement, while their SMHR are higher than 70% of the plain cement value. This indicates that the 30% bottom ash addition cannot be comparable to 30% of pure cement; however, they do contribute to the cement hydration to a certain degree. There is no difference in the aspect of the calorimetric curves of plain cement paste and BA-cement mixes (Fig. 7(a)). However, the additions of M/BA to cement lead to the retardation of cement hydration, so that the second maximum heat release rate (SMHR) is reached over three hours later than that of the cement paste (Table 3). For samples with 5TM/BA, this retardation is reduced by around 50 min, and the maximum heat release rate is higher than that of 70% of the plain cement and cement with sample M/BA. This means that the 5TM/BA has a more beneficial effect on the advancement of cement hydration than the M/BA samples. For samples with M5T/BA and M7T/BA, the maximum heat release rate is reached slightly earlier than in the case of plain cement. It can be noticed in Table 3 that the time needed for reaching the maximum heat release rate after the acceleration period of cement hydration, has a similar trend with that of the loss on ignition of the bottom ash samples at $550 \text{ }^\circ\text{C}$. The retardation effect of bottom ash on the cement hydration could be attributed to the organic matter

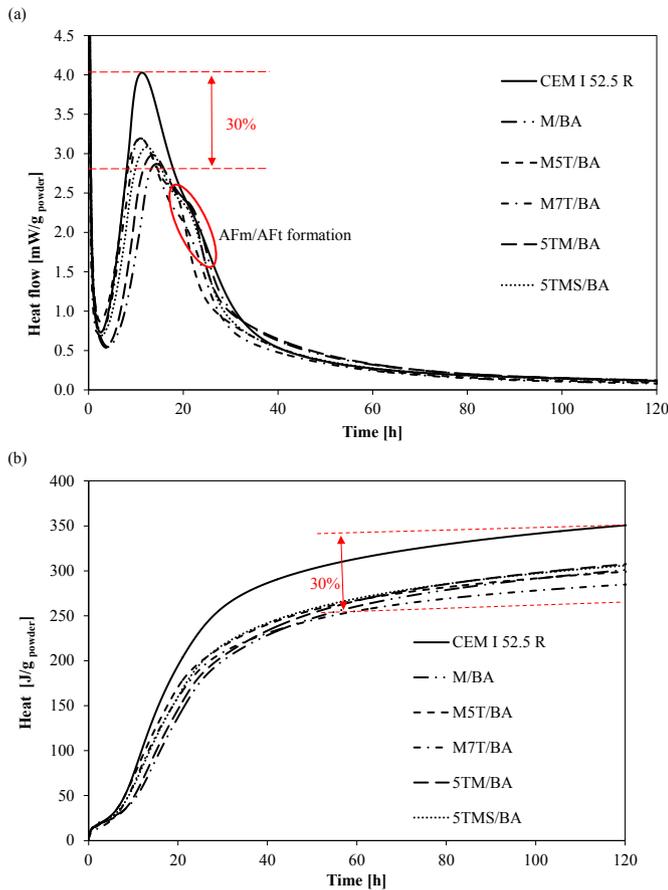


Fig. 7. The heat flow (a) and released heat (b) of cement with treated bottom ash relative to total powder amount.

Table 3
Calorimetric parameters of cement hydration and LOI of the investigated samples.

| | CEM I 52.5 R | M/BA | 5TM/BA | M5T/BA | M7T/BA | 5TMS/BA |
|--|--------------|--------|--------|--------|--------|---------|
| SMHR [hour] | 11.31 | 14.38 | 13.55 | 10.92 | 10.98 | 12.31 |
| Maximum heat release rate [mW/g _{cem}] | 4.03 | 2.87 | 2.98 | 3.20 | 3.19 | 3.08 |
| Total heat after 168 h [J/g _{cem}] | 366.65 | 317.21 | 323.35 | 311.07 | 295.53 | 319.98 |
| LOI 550 °C [% wt.] | 0.41 | 7.37 | 2.86 | 2.02 | 1.16 | 2.54 |

SMHR: duration for reaching the second maximum heat release rate.

contained in the bottom ash [68–70]. After the thermal treatment at this temperature, the organic matter is burned out, thus the retardation effect is reduced.

During the deceleration stage, there is a 'shoulder' on the hydration curve of samples with bottom ashes at around 17–25 h. A similar 'shoulder' can also be observed on the net cement hydration curve during the deceleration stage at around 22–27 h; however, this 'shoulder' is not clearly visible compared to the samples with bottom ashes.

According to [71,72], this 'shoulder' represents the formation of the AFm phase in pure cement paste. During cement hydration [72], the calcium aluminate (C_3A) reacts with calcium sulphate to form ettringite (Aft); after the calcium sulphate is completely consumed, due to the decrease of sulphate ions in the pore solution, the unstable ettringite converts to monosulphate; this normally occurs after 1 day or 2 days after the mixing of the cement paste; when there is a new source of sulphate ions come to be available in the pore solution, then the monosulphate reacts to form ettringite

again [73]. Therefore, in Fig. 7, the shoulder for pure cement at 22–27 h represents the formation of AFm. In the XRD pattern shown in Fig. 8 on the hydrated cement paste, the AFm phase is found, which also confirms this.

For the samples with ashes, there are several possibilities related to the sulphate consumption reaction. Firstly, the anhydrite in the ashes provides sulphates for the hydration of C_3A in the cement, which may result in more Aft formation [73]; secondly, the sulphate in the ashes does not have an influence on the reaction of C_3A , which means there will be AFm or little Aft found in the cement hydrates; thirdly, the sulphate in the ashes is a new resource for converting the AFm further to form Aft. Hence, the amount, dissolving rate and the type of the sulphate in the ashes are important parameters for its role in the cement reaction [74–77]. Therefore, the 'shoulder' for the samples with ashes could be attributed to the formation of Aft or AFm, which is mainly related to sulphate consumption during the cement hydration.

Fig. 7(b) and Table 3 show that the samples with bottom ashes achieved a lower total heat released based on the total mass of powder contents after the same hydration duration; their total heat released is however still higher than 70% of the plain cement value. The highest total heat (beside the plain cement sample) is reached by samples with 5TM/BA, followed by 5TMS/BA. It can be seen from the XRD pattern (Fig. 8) of the hydrated samples with and without ashes that there is AFm found in samples of plain cement, M5T/BA and M7T/BA. However, for the other samples with ashes, there is no AFm or Aft found. Considering the calorimeter results together, it can be explained that the samples with M5T/BA and M7T/BA may provide extra sulphate for cement to form AFm as explained above. For the other samples with ashes, the shoulder can be attributed to the second formation of Aft from AFm because of newly available sulphate; hence, these samples release a higher amount of heat during the cement hydration after 7 days than only 70% cement. Also, due to the consumption of the AFm and poor crystalline structure of AFm [78], there is no AFm or Aft found in the XRD

pattern of these samples.

Nevertheless, the hydration of cement is influenced by the organic content, the water to cement ratio, sulphate content, particle size and surface area, etc.; thus, the results should not only be explained by a single parameter. However, the total released heat results indicate that the addition of bottom ashes partially contribute to the cement hydration. Moreover, the retardation effect on cement hydration might be reduced by heating the milled bottom ash samples.

3.3. Mortar properties

As previous research reported [79], the lower pozzolanic properties of BAF and its retardation effect on cement hydration limit the amount to be used as cement replacement. Hence, based on literature, the replacement ratio of cement by treated bottom ashes is fixed at 30% in this study. The standard mortars are prepared according to EN 196-1 [47], and the fresh and hardened states

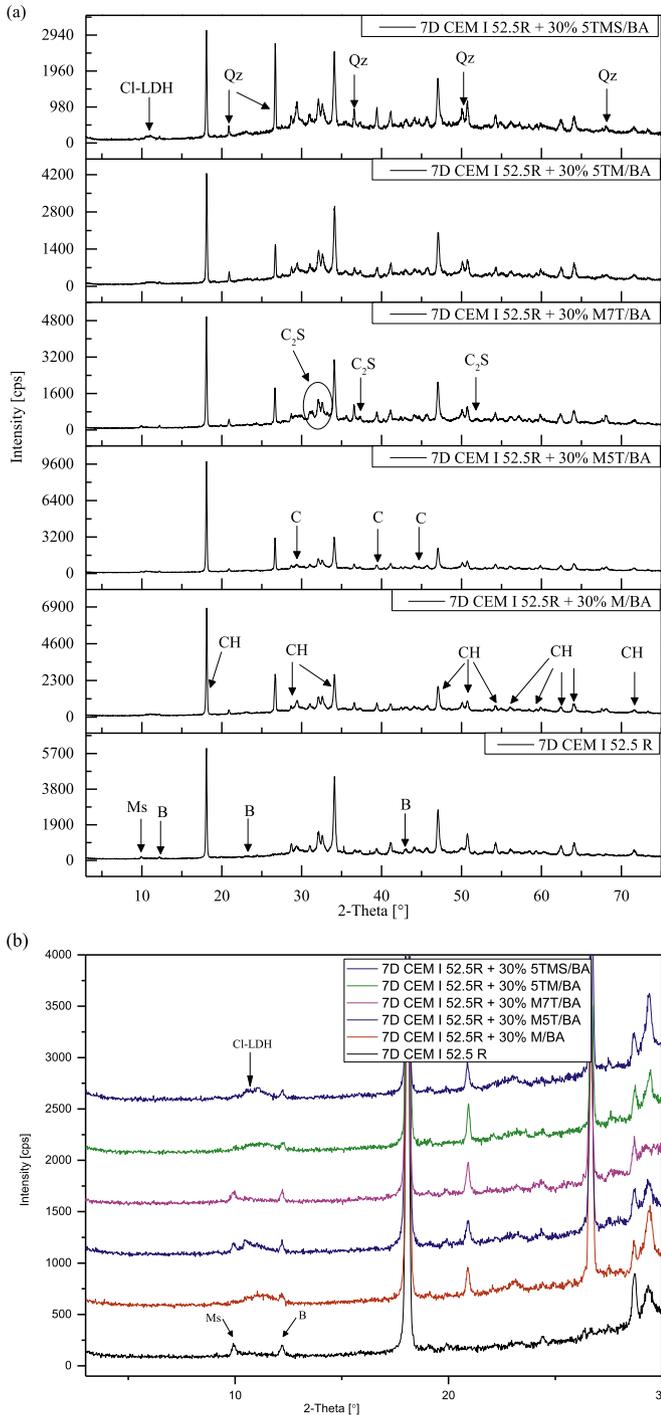


Fig. 8. XRD diffractograms of the cement pastes with bottom ashes after 7 days hydration (a) 3–75° (b) 3–30° (Ms – monosulphate, B–brownmillite, C–calcite, CH–portlandite, Qz–quartz, C₂S–dicalcium silicate phase, Cl-LDH-chloride layered double hydroxide).

properties are tested. The results are shown in the following section.

3.3.1. Flowability of mortars with treated bottom ashes

The test results show that the flowability of mortar decreases when treated bottom ash is added, except for the sample with M/BA (175 mm, compared with 170 mm of the reference). 5TMS/BA leads to the lowest flowability (160 mm), and M7T/BA leads to a higher

flowability (169 mm) than M5T/BA (165 mm). However, the quantitative differences on the flowability of the mortars are not significantly affected by adding the treated bottom ashes.

It is commonly known that the particle size distribution, particle shape, as well as particle texture of the alternative powder influence the water demand and subsequently the flowability of fresh mortar [35,80]. Hence, according to the previous investigations shown in this study (Figs. 5 and 6), these complex influential factors contribute to the phenomenon observed here.

3.3.2. Compressive and flexural strength

The compressive and flexural strengths of mortars containing bottom ash additions after various curing ages are displayed in Figs. 9 and 11.

It is demonstrated that both the compressive and flexural strengths of all the samples decrease at all curing ages [35]; when the treated bottom ash is applied, the mortar with 5TMS/BA gains higher strength than other bottom ash-containing samples. Moreover, for all the samples, the decrease of the flexural strength is not as significant compared to the reduction of the compressive strength.

For all the investigated samples, the compressive strength increases with increasing curing age. Furthermore, their differences to the reference are reducing with longer curing ages. It is noticed that the mortar with 5TM/BA gains higher compressive strength at all curing ages than the mortar with M/BA. This is attributed to the burning of organic matter in the BAF at 550 °C, which decreases its detrimental effect on cement hydration. Additionally, after 28 days curing, the decrease of compressive strength of the mortar with M/BA is about 36%, but only about 30% in case of the mortar with 5TM/BA, indicating the detrimental influence of M/BA and 5TM/BA. This is in agreement with the finding presented in Section 3.2.

However, the strength test results for the mortar with M5T/BA and M7T/BA are in conflict with the calorimetry results. These mortars have a relatively higher decrease of the compressive strength at all curing ages than other samples. For instance, after 28 days the compressive strength of mortars with M5T/BA and M7T/BA is about 30 MPa and 37 MPa, respectively (51% and 40% decrease compared to the reference, respectively). Their strength is even lower than mortar with M/BA. This higher reduction should be due to the inner cracks in mortars (as shown in Fig. 10) created by the H₂ generated upon the hardening of mortar. Nevertheless, inner cracks [67] are not found in mortars with M/BA, 5TM/BA and 5TMS/BA, of

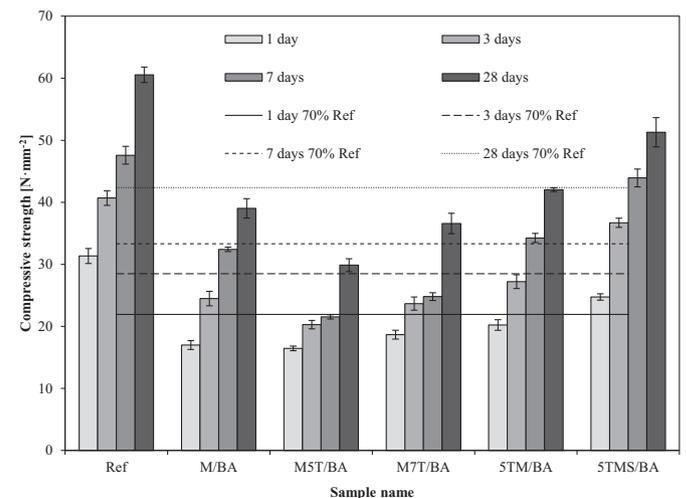


Fig. 9. Compressive strength of mortar containing bottom ash after 1, 3, 7, 28 days curing and the calculated 70% of reference lines.

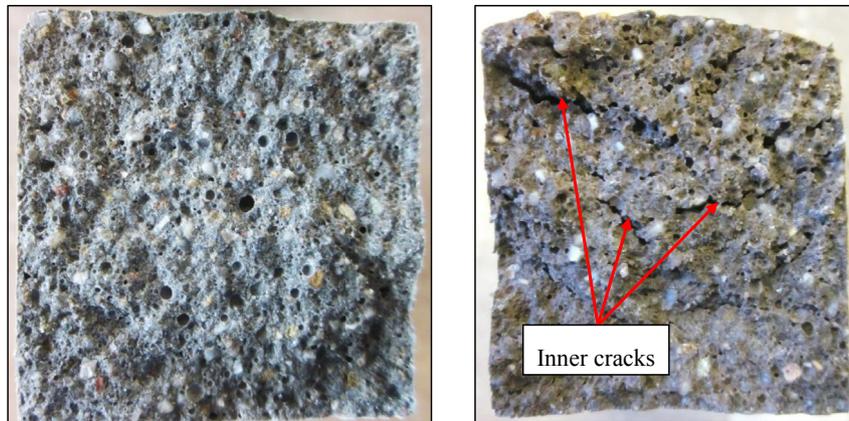


Fig. 10. The fracture surface of mortars (reference and M5T/BA).

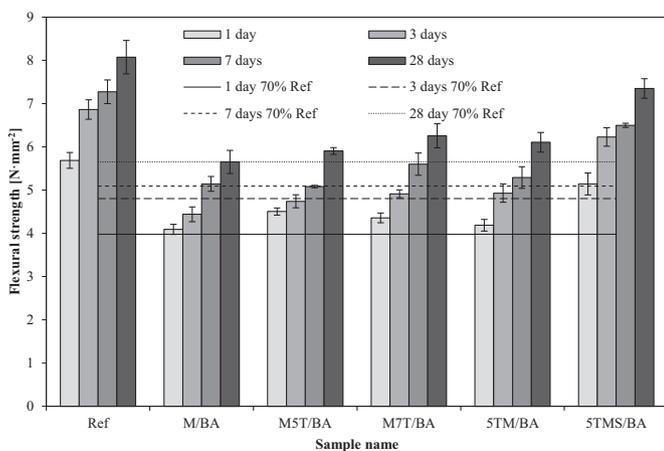


Fig. 11. Flexural strength of mortars containing bottom ash after 1, 3, 7, 28 days curing and the calculated 70% of reference lines.

which the first two have similar contents of metallic Al. This could be explained as follows: the thermal treatment on the milled bottom ash powder is more efficient on burning out the organic matter, as well as the oxidation of metallic aluminium. Hence, after thermal treatment, the surface of the metallic Al particles is oxidized [81]. Subsequently the reaction between metallic Al and alkalis takes a longer time, and the gas generated later could not be released before the hardening of mortar. Eventually, inner cracks form, which reduce dramatically the strength of mortar.

Mortar with 5TMS/BA gains the highest 28-day compressive strength, about 51 MPa, a 15% reduction compared to the reference. This result should be due to the following reasons: firstly, the particle size of the 5TMS/BA is controlled under 63 μm , which leads to a denser microstructure of the mortars. Secondly, it has the lowest metallic Al content in comparison with other treated bottom ash samples, so in turn less inner cracks are generated. Thirdly, it has a lower content of SiO_2 and a higher amount of CaO, which may participate in the cement hydration process.

The flexural strength of the mortar decreases when using bottom ash samples; however, the effect of the treated bottom ash on the flexural strength is more complicated than for the compressive strength. The flexural strength is significantly influenced by the cement hydration [82], and irregular particle shapes have a positive effect on flexural strength [83,84]. As the bottom ash has lower pozzolanic properties and more irregularly-shaped particles, part of the inert particles act as filler in the mortar matrix. Hence, there

is a better inter-locking effect between the cement paste and these particles, which contributes to a lower reduction of the flexural strength (Fig. 11). This finding is consistent with results reported by other researchers [85,86].

To summarize, the cement blended with 30% by M/BA can be used to produce low strength mortar, its compressive strength is comparable with that reported previously [35]. The cement with 30% 5TMS/BA could be used to produce a standard mortar which has a 28-day compressive strength of 51 MPa (compared to the 61 MPa reference).

3.4. Environmental impact evaluation

According to the Dutch legislation (Soil Quality Decree, SQD) [53], the leaching concentration of heavy metals and salts should be below the limiting values, considering the environmental impact and human health. The column leaching test is performed on the treated bottom ash samples, as well as on the mortar with treated samples to evaluate their environmental impact. The results are shown in Table 4.

Based on the evaluation of ten-year leaching data on the MSWI bottom ash in the previous work of the authors [26,87], it was summarized that the leaching concentration of most elements in the bottom ash from this plant is below the limits and the leaching of antimony (Sb), copper (Cu), molybdenum (Mo), chloride (Cl^-) and sulphate (SO_4^{2-}) mostly exceed the limit level. Hence, the leaching after the treatment is evaluated in this study and their total amount in the treated and untreated ashes are measured as well (Table 4).

It is indicated in Table 4 that the leaching of Sb and Cu in the bottom ash without thermal treatment exceeds the limit values (more than 4 times of the limit emission value). However, after the thermal treatment, their leaching concentration reduced significantly and is well under the limiting level. This could be due to the burning of the organic matter, which is related to the leaching of copper [88–90]. It can be seen that M/BA and 5TMS/BA have the same amount of Cu, however, M/BA has a higher leaching of Cu. This confirms that the thermal treatment decreases the leaching of Cu. Sample 5TM/BA has a higher total amount of Cu than 5TMS/BA, but lower leaching of Cu than 5TMS/BA. This is due to the fact that 5TMS/BA has more soluble Cu, which is mainly absorbed during the quenching process. 5TMS/BA, which concentrated the most contaminated fine powder in BAF, has a relatively higher leaching of Mo, Cl^- and SO_4^{2-} , which is in line with the finding in Refs. [58,59] that the dust-layer on the particle surface influences the contaminant leaching. In the case of Mo and Sb, the variation in leaching

Table 4

The Soil Quality Decree (SQD) [53] limits and leaching properties of treated BA samples and mortar with the BA after 28 days curing, the total amount of certain heavy metals and salts (d.s.–dry solid) and the pH of the leachate after column leaching of same samples.

| | SQD limit value [53] | Total amount [mg/kg d.s.] | | | Leaching value [mg/kg d.s.] | | | Mortar with M/BA | Mortar with 5TM/BA | Mortar with 5TMS/BA |
|-------------------------------|----------------------|---------------------------|--------|---------|-----------------------------|--------|---------|------------------|--------------------|---------------------|
| | | M/BA | 5TM/BA | 5TMS/BA | M/BA | 5TM/BA | 5TMS/BA | | | |
| Sb | 0.16 | 75 | 86 | 110 | 0.79 | 0.037 | 0.023 | <0.0089 | <0.0059 | <0.0089 |
| Cu | 0.9 | 3100 | 4000 | 3100 | 3.6 | <0.050 | 0.3 | 0.17 | <0.050 | 0.06 |
| Mo | 1 | 11 | 10 | 12 | 0.67 | 1.4 | 2.6 | 0.09 | 0.11 | 0.092 |
| Cl ⁻ | 616 | 3800 | 5300 | 9200 | 4400 | 3300 | 6600 | 350 | 290 | 450 |
| SO ₄ ²⁻ | 1730 | 18,100 | 24,000 | 45,700 | 16,000 | 20,000 | 23,000 | 180 | 180 | 270 |
| pH | | a | a | a | 7.8 | 10.39 | 10.18 | a | a | a |

^a Not measured.

can be attributed to the change in pH of the leachate. The pH data of the leachate of each sample is mentioned in Table 4. It can be seen that the samples that underwent thermal treatment will generate a much higher leachate pH. This difference explains both the decrease of Sb leaching and the increase of Mo leaching [41,44]. The chloride leaching of 5TM/BA is lower than that of M/BA, which confirms the removal of the highly soluble chlorides by the thermal treatment [59]. However, this reduction is not remarkable and the leaching is still above the limit level. The leaching of sulphates from the investigated samples exceeds the limit value of the legislation, and it seems thermal treatment did not influence the leaching of sulphate significantly.

The leaching properties of the mortars with treated bottom ash samples are also studied to investigate the immobilization of the contaminants in the samples. It can be seen in Table 4 that the leaching of all the investigated heavy metals and the salts is far below the limit leaching level. Hence, the mortars with 30% cement replacement by the treated bottom ash meet the Dutch legislation [53]. Moreover, the sulphate is more immobilized than the chloride in the mortar, when comparing their total amount and leached concentration (Table 4). This may further prove that the sulphate in bottom ash participates in the cement reaction as explained in Section 3.2, hence the leaching of sulphate in the mortar decreases more than that of chloride.

Consequently, in this study the leaching of Sb and Cu of the BAF is reduced to values under the limit by thermal treatment. Furthermore, the mortar with treated bottom ash complies with the Dutch national environmental legislation [53] considering the leaching of contaminants in future life cycles.

4. Conclusions

In this study, milling and thermal treatment are applied to upgrade the properties of fine MSWI bottom ash (0–2 mm), and the characteristics of the treated bottom ashes are investigated. Moreover, their effect on cement hydration and mortar properties containing 30% bottom ashes as cement replacement are illustrated. The following conclusions can be drawn:

- (1) The main chemical components of the MSWI bottom ash are SiO₂, CaO, Al₂O₃, Fe₂O₃ and the main crystalline phases are quartz, calcite, anhydrite and hematite. After the thermal treatment at 550 °C and 750 °C, qualitative differences on the chemical compositions are not identified. Higher temperature results in the decomposition of calcite and enhances the oxidation of iron. The amount of metallic Al is not reduced significantly after the thermal treatment. However, the short time and lower speed milling followed by sieving can separate the metallic Al from the fine powder chosen for the investigation. Additionally, this fine powder has a lower amount of SiO₂ and higher amount of CaO and Al₂O₃.

- (2) The hydration of the cement is retarded when milled raw BAF is added, which is due to the organic matter and heavy metals. Thermal treatment can reduce the retardation effect of the bottom ash on cement hydration. Furthermore, a sufficient thermal treatment on milled bottom ash enhances the contribution of the bottom ash on cement hydration and minimizes its retardation effect. The sulphate content in the bottom ash accelerates the hydration of C₃A and then increases the formation of AFm during the deceleration stage of cement hydration.
- (3) The 28-day compressive strength of the mortar with 30% treated bottom ashes replacement is 30–51 MPa, which is less than the reference (61 MPa). The highest strength is obtained in mortar with 5TMS/BA; the 30% cement replacement results in 15% compressive strength and 8% flexural strength reduction. Although an efficient thermal treatment increases the contribution of bottom ash on cement hydration, the oxidation of metallic Al prolongs the generation of gas. Eventually, the cracks in the hardened mortar reduce its strength.
- (4) Thermal treatment efficiently reduces the leaching of Sb and Cu, and slightly decreases the chloride leaching of the bottom ash fines. The mortars with 30% treated bottom ashes are environmentally acceptable according to the Dutch legislation, as the leaching of heavy metals and salts is well under the limit level. The fine dust-layers removed from the bottom ash particles have higher leaching levels of heavy metals, which confirms that these layers influence the heavy metals leaching of MSWI bottom ash.
- (5) The technique for the removal of metallic Al by slow milling (and the formation of metal plates which can be sieved out) was proposed and its applicability was proven.

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