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Review

Investigation of the hydrothermal treatment for maximizing the MSWI bottom ash content in fine lightweight aggregates

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HIGHLIGHTS

- Lightweight pellets including up to 80% MSWI BA are manufactured.
- Hydrothermal treatment improves mortars flexural but not compressive strength.
- Hydrothermal treatment decomposes Friedel's salt and generates Tobermorite phase.
- Standard curing is preferred for the contaminants retention than hydrothermal curing.

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ABSTRACT

The recycling of Municipal Solid Waste Incineration Bottom ash (BA) with a size below 4 mm is often difficult, due to its high contmination and porosity. To increase its reuse, the manufacture of lightweight aggregates represents a suitable option, as it takes advantage of the BA porous structure and uses cement for the immobilization of harmful contaminants. For highly contaminated ashes, hydrothermal treatment (HT) is known for further improving the immobilization of some heavy metals in the cementitious matrix. However, not many studies have investigated it as a curing method for pellets. This study addresses the application of highly contaminated BA fines in the production of lightweight aggregates. Firstly, pellets containing up to 80%wt. BA are manufactured and classified as lightweight aggregates, according to EN 13055. In the absence of HT, the formation of Friedel's salt helps the Cl^- retention, lowering the leaching by 65% compared to theoretical values. After HT, the formation of tobermorite on the pellet surface is beneficial for the flexural strength (+14%). However, the decomposition of phases such as Friedel's salt increases the leaching of Cl^-, Cu, Mo, and Zn compared to standard cured samples.

Contents

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1. Introduction

During the last years, the disposal and recycling of household waste have improved remarkably. Among the approaches, the application of municipal solid waste incineration (MSWI) has become more and more popular thanks to the use of Waste to Energy (WtE) plants, which reduce the volume of waste and allows energy production via heat recovery [41,52]. From this process, one main solid by-product is generated, accounting for 80% of the total incineration output: MSWI bottom ash (BA) [41]. Because of the high volume and the fact that it could cause contamination of the groundwater [37], landfilling as a disposal method for MSWI BA should be avoided. The need for alternative approaches for the recycling of MSWI BA led some European countries to consider its application as secondary building materials. Among them, the Netherlands regulates the legal limits for the leaching behaviour of heavy metals and salts from those materials, using the Soil Quality Decree [47].

Many studies have been conducted on the use of MSWI BA as secondary building materials, showing potential in the application as cement replacement, or as coarse and fine aggregates [7,10,13,9,14,37,41,49]. Despite extensive studies, the application of BA fines (below 4 mm) is difficult due to the high content of contaminants accumulating in this fraction [2]and their high porosity originating from the quenching process [15,38]. In order to take advantage of the porosity of those particles, a potential option is the manufacture of lightweight pellets or aggregates [16,18]. Both natural and artificial lightweight aggregates are used to produce low density concretes (300–1200 kg/m^3) but also structural concrete (1000–2000 kg/m^3), depending on the raw materials and techniques adopted to manufacture them [51]. Generally BA fines are mixed with Portland cement or common by-products such as fly ash [48], in order to reduce both the costs and the environmental impact of the pellets and enable immobilization of contaminants in the cement hydration products [28,29]. However, due to the low content of cement used in the pellet production, the immobilization of heavy metals, chlorides and sulfates might be insufficient. In order to increase the immobilization rate, some studies have shown that the application of hydrothermal treatment (HT) is beneficial, contributing to strength development [11,35], due to the formation of tobermorite and the densification of the cement matrix. Nevertheless, to the best of the authors' knowledge, no studies have analyzed the influence of hydrothermal treatment (HT) on the behaviour of pellets or have investigated the maximum BA content possible while using this treatment.

Therefore, this study will address the application of MSWI BA fines in the production of lightweight pellets, maximizing the MSWI BA content and minimizing the density. The optimal pelletization conditions (e.g., rotational speed, pan inclination, and water amount) are determined. The pellets' physical and chemical properties are measured, based on different recipes with an increasing amount of BA to determine which mixture can satisfy the requirement of EN 13055:2016 for the classification as lightweight aggregates. Finally, a hydrothermal treatment is applied to investigate its influence on the reaction products formation by using XRD. Together with the mechanical strength, the leaching behaviour is addressed according to EN 196-1 and EN 12457-2 respectively.

2. Experimental

2.1. Materials

In this study, the BA used is provided by and processed at the Heros Sluiskil plant (NL) (Fig. 1). Two different BA fractions are applied, in order to maximize the BA content of the pellets, and minimize the amount of cement used; the core of the pellet is made by BA-M (between 0.250 mm and 1 mm, Fig. 1), which aims to provide improved mechanical performance of the pellet [48]. BA-M is obtained by the fractioning of fine bottom ash (FBA, below 4 mm, Fig. 1) on a laboratory scale. Filter cake (FC, below 0.25 mm), obtained by the wet washing and centrifuging of the coarse bottom ash (CBA, 4–32 mm, Fig. 1), is applied as cement replacement for the manufacture of the external layer, coating the internal core of the pellet and limiting water absorption and contaminant leaching of the final lightweight aggregates. The suitability of this by-product as cement replacement has already been addressed in another study [9,14]. Due to the high replacement applied the FC is previously mechanically activated using ball mill as described in the study [9,14]. Applying 30 min mechanical activation lowers the FC water demand by around 70%, resulting in absorption behaviour similar to the one of plain PC. CEM I 52.5 R (ENCI, NL) was added in a later stage, as a coating of the formed pellets. Samples nomenclature will be structured as follow: P_n, for standard cured pellets having n as the rate of PC replaced by FC and P_nA for hydrothermally treated samples, having n as the rate of PC replaced by FC. P_0 represents the reference pellets where no FC is replaced in the external layer. The correspondent mortars will be named as Mor P_n or Mor P_nA, for standard and hydrothermal curing, respectively.

2.2. Methods

The fractions BA-M and FC are oven-dried for 72 h at 60°, to entirely remove the water without modifying the mineralogical phases present and evaluate the initial moisture content (MC). The chemical composition of the materials was measured by X-Ray Fluorescence (XRF) (PANalytical Epsilon 3 range, standardless OMNIAN method) on pressed powders. The determination of crystalline phases was done by X-ray Powder Diffraction measurements, with a D2 (Bruker) using a Co anode, a LynxEye detector, fixed divergence slits (0.12°) and a step scan size of 0.02. A primary and secondary soller slit with 2.5° were used. For the identification of the mineral phases X'pert HighScore Plus software has been used. The following phases have been detected: Ettringite (Aft, sulfoaluminate phase, ICDD number 00-009-0414), Calcite (Calcium carbonate, ICDD number 00-005-0586), Quartz (silica, ICDD number 00-046-1045), Portlandite (Calcium hydroxide, ICDD number 00-044-1481), Alite (tricalcium silicate, ICDD number 00-049-0442) Belite (dicalcium silicate beta, ICDD number 00-031-0298),

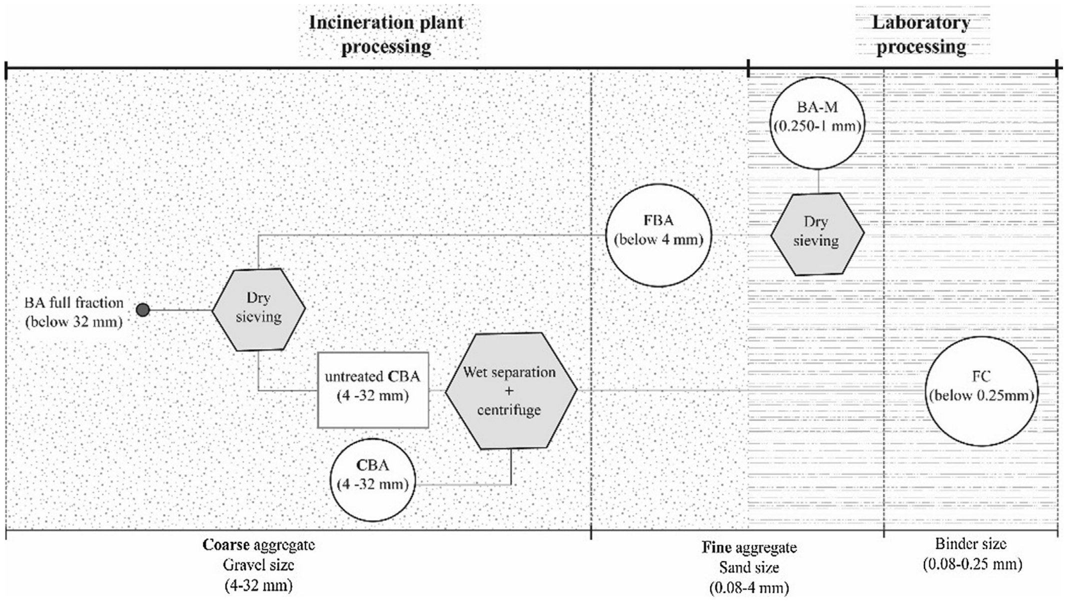


Fig. 1. Graphical representation of the treatments and fractioning of BA fine fraction used in this study.

Friedel's salt (ICDD number 01-078-1219), Gypsum (Calcium Sulfate Hydrate, ICDD number 00-021-0816), Tobermorite 11A (Calcium silicate hydrate, ICDD 00-045-1480). The particle density of the BA-M, FC and PC was determined by Helium Pycnometer (AccuPyc II 1340), and the loose bulk density by a conventional pycnometer, according to the EN 1015-10 [20]. The water absorption of the BA-M was measured by mass gain after 24 h, while the powders (PC and FC) water demand was determined by the Punkte test [32]. The particle size distribution of the coarser fraction (BA-M) was measured by sieving using a vibratory sieve shaker (Retsch: AS 450 Basic), according to DIN EN 933-2 [24]. The visualization of the particle morphology was achieved by Scanning Electron Microscope (SEM) (Phenom ProX), using a spot size of 4.0 and 15 kV voltage. The materials BET surface area and pore volume were measured by nitrogen physisorption measurement (Micromeritics, Tristar II 3020 V1.03). The quantification of the metallic aluminium was performed by digestion in alkaline conditions (3 M NaOH solution) for 24 h [44], measuring the volume of hydrogen released in the presence of the alkaline environment. The leaching assessment was performed on the loose pellets after 28 days, but also on the mortars manufactured using the pellets as a full sand replacement. The classification of lightweight aggregates was based on EN 13055:2016 [22], considering a loose bulk density below 1200 kg/m^3 as a reference value. The hydrothermal treatment (HT) is applied to half of the produced pellets after 7 days reaction in standard curing. The HT was performed according to Caprai et al. [12,11], in order to apply optimal conditions for maximizing both the environmental and mechanical performances of the pellets and also to limit the embedded energy of the final material. A total curing time of 6 h (1.5 h heating, 3 h 190 °C plateau and 1.5 h cooling), at 11 bars was applied on the granular pellets, placed in steel perforated trays.

2.2.1. Determination of optimal conditions for pellets production without FC

The determination of the optimal parameters for the production of the pellets was performed according to previous studies concerning pellet engineering [5,6]. The determining parameters for the proper production of pellets are the rotational speed and the angle of pelletization. Those factors influence the forces acting on each pellet, affecting the shape and strength of the final product [5]. The following equations were used for determining the so-called "critical revolutions per minutes" (n_{cr}) when gravitational and centrifugal forces act equally on the pellets:

$$m * g * \sin\beta = m * \frac{D}{2} W^2 \tag{1}$$

$$n_{cr} = \frac{42.3}{\sqrt{D}} \sqrt{\sin\beta} \tag{2}$$

where m is the mass of the individual pellet [g], g is the gravitational acceleration [m/s^2], β is the angle of the pan [°], R is the radius of the disk [m], w is the centrifugal acceleration in gradients/t^{2,} and 42.3 is a constant [6]. Using Eqs. (1) and (2) an optimal n_{cr} is calculated based on the chosen inclination angle. In addition to the n_{cr} calculated a rotation speed of ± 5 rpm is also considered and tested.

Therefore, three different inclination angles (50–55–60°) and rotation speeds (40–50–60 rpm) have been taken into account for achieving a PSD of the BA pellets close to the sand. At the same time, different water amounts are used during pelletization. Fixed water to binder ratio of 0.25 was set for the PC in the pellet. Additional water was used to pre-soak BA-M in order to reduce the excessive sorption of the pellet core. Three presoaking amounts were calculated, based on the 24 h water absorption of the BA-M (31% wt.) corresponding to 50%, 75% and 100% of this value (16% MC, 24% MC and 31% MC, respectively, Fig. 2).

The general scheme in Fig. 2 shows the various combinations evaluated. During the pellets manufacturing a rotating pan of 40 cm was used (EIRICH Disk Pelletizer Type TR04). The optimal rotation speed for the manufacture of lightweight fine aggregates was 40 rpm, while the chosen pan rotating angle was 50°. The choice of the optimal recipe and settings for the pellets' manufacture is based on the loose density (lightweight product) and particle size distribution (sand fraction) targeted for this study. All further experiments, with different Filter Cake (FC) amounts (10, 30, 50% wt.), were made with these settings. The ratios used and the PC and BA final content is presented in Table 1.

2.2.2. Mortars manufacture

The rheological behaviour of the mortars was evaluated using a Hägermann cone and a mortar flow table (Jolting table), according to EN 1015-3 [21], applying a liquid to solid ratio (L/S) of 0.5. The

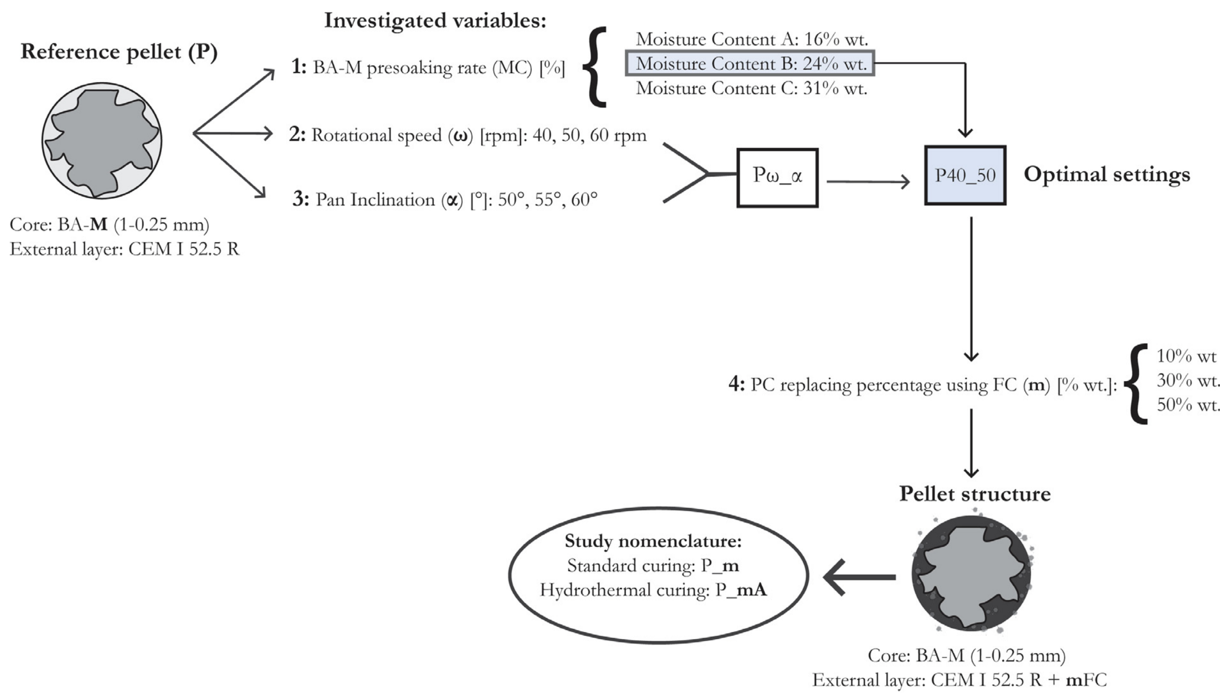


Fig. 2. Methodology applied in this study for the evaluation of the optimal parameters for the manufacture of pellets with the size of sand.

Table 1
Definition of the parameters used in the manufacture of pellets with increasing FC content in the coating layer.

	L/S*	w/B	Coating/Core*	PC mass percentage	BA-M + FC mass percentage
	–	–	–	%	%
P_0	0.24	0.25	0.75	42.9	57.0
P_10	0.24	0.25	0.75	38.6	61.4
P_30	0.24	0.25	0.75	30.0	70.0
P_50	0.24	0.25	0.75	21.4	78.6

*L/S stands for liquid–solid (Water/(PC + F**C** + BA – M), Coating/Core stands for the mass ratio (PC + FC/(BA – M)).

manufacture and testing of the mortars were performed according to the EN 196-1 procedure [23], using the pellets as 100% volume replacement of the standard sand. In order to apply a volume-based replacement, preliminary studies showed that its quantification using the particle density leads to a highly viscous paste, unable to be correctly cast. Therefore the volume of pellets needed for mortars was calculated based on the sand loose bulk density. It is mainly depending on the high volume occupied by the pellets (lightweight product), reducing the final proportion between paste and aggregates. The final L/S ratio of the mortars was fixed at 0.5. In the mortars, the sand to binder mass ratio was 3. The flexural strength results were based on the average of three specimens, while for the compressive strength an average of six samples was used.

Moreover, the mortars were cured in a high relative humidity chamber (RH 95%) to avoid its influence in the leaching behaviour of the material after 28 days. The curing was not performed in water, to avoid the leaching of contaminants during the curing period.

2.2.3. Leaching assessment

The determination of the leaching of contaminants in the presence of water was tested for the initial by-product (FC and BA-M, Section 3.1) and for the pellets (Section 3.5.1), using the one batch leaching test (EN 12457-2) [25,31], with a dynamic shaker (ES-SM-30, Edmund Buhler GmbH), in ambient conditions (L/S 10, 250 rpm, 24 h). Despite the high contamination of the MSWI ashes, a preliminary washing treatment has not been applied, due to the outcome of a previous study [12], underlining the triviality of this treatment for lowering most of the contaminants, in the presence of HT. The same test is applied for the evaluation of the final mortars containing the pellets as fine aggregates (Section 3.5.2). The leaching of PC describes the leaching of heavy metals of a paste after 28 days high RH curing, aiming to show the contribution of the PC in the leaching of the final product. After the evaluation of the mechanical performances, the mortars were sieved below 4 mm and tested using the one batch leaching test. The results have been compared to the Dutch Soil Quality Decree, which defines the leaching thresholds for heavy metals, chlorides, and sulphates [47], tested using the column leaching test (Percolation test – NEN 7383:2004 [43]. Other studies show that the one stage batch leaching test is sufficient for addressing the compliance with legislative limits values [30], as the leaching values obtained from it overestimate the contaminant concentrations (for the same L/S ratio) compared to the column test [45]. For comparing the retention of contaminants in standard curing conditions and after hydrothermal treatment (HT), theoretical leaching values of the pellets were calculated based on the equation below:

$$C_t = \frac{C_{ePC} * m_{PC} + C_{eFC} * m_{FC} + C_{eBA} * m_{BA}}{m_{tot}} \quad (3)$$

where

C_t is the theoretical concentration of a specific contaminant [mg/kg], C_{ePC}, C_{eFC} and C_{eBA} are the effective leaching concentration for a specific contaminant, shown in Section 3.1 [mg/kg], m_{PC}, m_{FC} m_{BA} and m_{tot} are the mass fraction and total applied during the pellets manufacture [kg].

3. Results and discussion

3.1. Characterization of the initial materials

The physical properties of the materials applied in this study are displayed in Table 2. Based on the loose bulk density, the by-product used as core (BA-M) is very porous, also confirmed by the high BET surface area and pore volume (Table 2). Its particle size ranges between 0.125 and 1 mm, and the initial moisture content is 8.8% wt. Due to the high porosity, the water absorption after 24 h is 32% of its weight. The MSWI Filter Cake (FC) is used as a cement replacement in the external layer, because of its particle size close to PC. FC has 32 times higher surface area than PC, due to the presence of ultrafine particles, residues from the incineration and quenching process [33]. Due to the wet centrifugation technique applied in the plant (Section 2.1), FC is provided with an initial moisture content of 28%. However, the BA-M moisture content is taken into account for the calculation of the presoaking water, during the pelletization process (Section 2.2).

Chemically, FC is characterized by a higher calcium content than BA-M mainly in the form of calcite (Fig. 3), due to the natural weathering and the brittle nature of this mineral accumulating it in the fine fraction (Table 3). As reported in Alam et al. [2], BA-M shows a higher content of silica, mostly in the form of quartz due to the harder nature of this mineral, collecting in the coarser fractions (Table 3). Among the major oxides, alumina ranges between 7 and 8% wt. in the by-products, but a little amount is in the form of metallic aluminum (0.1% wt.). Metallic aluminum can be detrimental for the application of BA as a secondary building material, due to the hydrogen gas released once placed in an alkaline environment [40]. Despite this, the limited amount of metallic aluminum measured in FC and BA-M did not result in crack formation and expansion behavior in pellets or mortars.

Table 3
Chemical composition of the materials applied in this study.

Oxides	FC % wt.	PC % wt.	BA-M % wt.
MgO	1.92	1.52	1.71
Al_2O_3	8.84	3.88	7.09
SiO_2	15.32	15.82	32.85
P_2O_5	1.12	0.49	1.28
SO_3	2.45	3.94	2.77
K_2O	0.88	0.58	1.13
CaO	32.72	68.38	17.98
TiO_2	1.90	0.28	1.17
MnO	0.47	0.09	0.12
Fe_2O_3	10.29	3.69	9.27
CuO	0.64	0.02	0.49
ZnO	1.16	0.12	0.55
Cl^-	0.35	0.02	1.11
Other oxides	0.54	0.29	2.0
LOI	21.4	0.99	19.8

Table 2
Physical characterization of the materials applied in this study.

Material		FC	PC	BA – M
Bulk density	g/cm^3	0.84	1.1	1.12
Particle density	g/cm^3	2.55	3.15	2.28
BET surface area	m^2/g	34.63	1.5	9.28
Water demand/Water absorption	% dw*	88.8	23.22	32.1
Total pore volume	cm^3/g	0.098	–	0.026
MCi	% dw*	28	0.1	8.8
Metallic Al	% dw*	0.1	–	
Particle size range	μm	0.4–250	0.2–90	125–1000

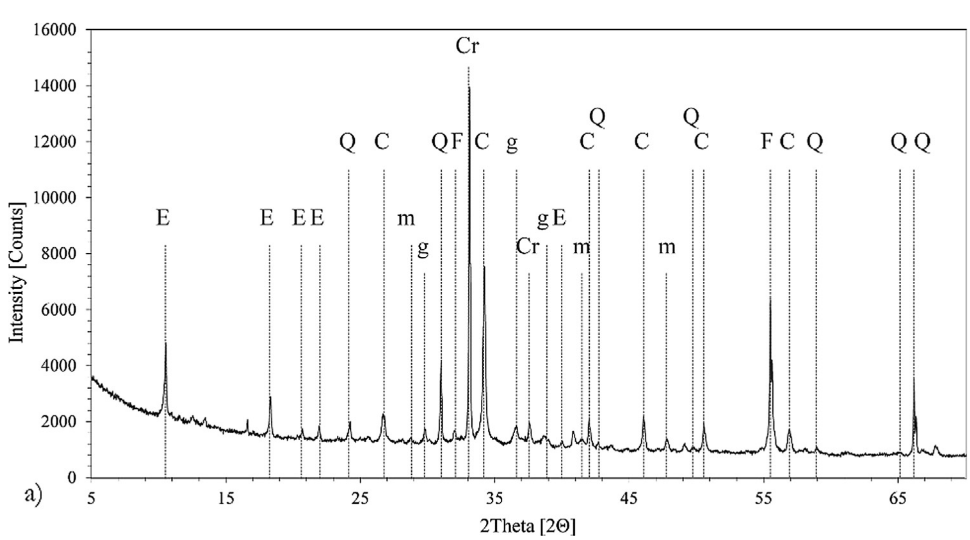


Fig. 3. Filter Cake (FC) XRD patter, including C – Calcite, Q – Quartz, Cr – Cristobalite (SiO_2), E – Ettringite, m – Magnetite (Fe_3O_4), g - Gehlenite ($Ca_2Al(AlSi)O_7$), F – Feldspar.

The leaching assessment of the by-products underlines the presence of several contaminants exceeding the Dutch soil quality decree (SQD) (Table 4). The highest leachability is shown by Cu, Mo, Sb, Cl^- and SO_4^{2-}. Despite the higher surface area, the leaching of Cl is lower for FC, mainly due to the wet treatment and centrifugation applied in the plant, which partially removes the readily soluble salts, including Cl^- [9,14]. On the other hand, the sulfate leaching is much higher for FC. It is generally known that sulfates tend to accumulate in the fraction below 0.25 mm, mainly in the form of ettringite or gypsum [3,11], leading to higher leaching for sulfate.

3.2. Physical properties of the pellets

The evaluation of the physical properties for the classification of lightweight pellets is shown in Fig. 4a) and b). Based on the classification given by the EN 13055 [22], the particle density (ρ_p) of all manufactured pellets is above the legislation threshold required for lightweight aggregates ($\rho_p < 2$ g/cm^3). Despite the internal porous structure of the core BA-M, the high particle density of the pellets is mainly caused by the high presence of iron-based phases characterizing the FC and BA-M chemical composition (Table 3). On the other hand, the (Fig. 4a) and b)) loose bulk density (ρ_b) if low enough to qualify as a lightweight aggregate. The pellet structure is characterized by a internal core, formed by the compaction of multiple BA-M particles, and an external mineral binder layer (Fig. 5a)). The presence of PC improves the overall particle shape of BA-M, smoothing the surface area and closing part of the porosity of the particles, without penetrating the core in depth. Fig. 5b) shows how the central core of the pellets (BA-M) preserves its high porosity and open structure, while the PC coats the outside of the core, limiting the water absorption. This particular structure of the pellet ensures a low, loose bulk density (Fig. 4a)), and allows the classification of these pellets as lightweight aggregates based on EN 13,055 ($\rho_b < 1.2$ g/cm^3), underlined in Fig. 4a) by the dashed line.

Comparing the particle size distributions (PSD) of the pellets, Fig. 6 shows that an increasing substitution rate of PC with FC increases the amount of smaller pellets. This trend can be explained by the reduction of the binder (PC) in the system, which limits the compaction and adhesion among smaller particles.

In this section, HT cured pellets are not discussed since the physical properties of the pellets were not affected by the hydrothermal treatment.

Table 4
Leaching assessment of the initial by-products by the one batch leaching test [31] in comparison to the Dutch Soil Quality Decree [2]. The values in bold indicate the elements overcoming the SQD threshold.

Elements	SQD Unshaped material mg/kg d.s.	FC mg/kg d.s.	BA-M mg/kg d.s.	PC mg/kg d.s.
pH	–	11.5	11.8	11.1
Antimony (Sb)	0.32	**2.4**	**1.2**	0.1
Arsenic (As)	0.90	<0.3	<0.3	<0.3
Barium (Ba)	22.00	0.4	0.8	13.8
Cadmium (Cd)	0.04	<0.02	<0.02	0.04
Chromium (Cr)	0.63	0.2	<0.02	0.02
Cobalt (Co)	0.54	<0.02	<0.03	0.03
Copper (Cu)	0.90	**1.3**	**5.42**	0.2
Lead (Pb)	2.30	0.1	<0.01	0.1
Molybdenum (Mo)	1.00	0.7	**2.04**	0.12
Nickel (Ni)	0.44	0.07	<0.05	0.06
Selenium (Se)	0.15	<0.2	<0.2	0.2
Tin (Sn)	0.40	<0.1	<0.1	0.1
Vanadium (V)	1.80	<0.1	0.11	0.1
Zinc (Zn)	4.50	0.4	0.15	0.05
Chloride (Cl)	616	**2966.0**	**7210.0**	35.80
Sulfate (SO_4^{2-})	2430	**21179.0**	**12960.0**	56.30

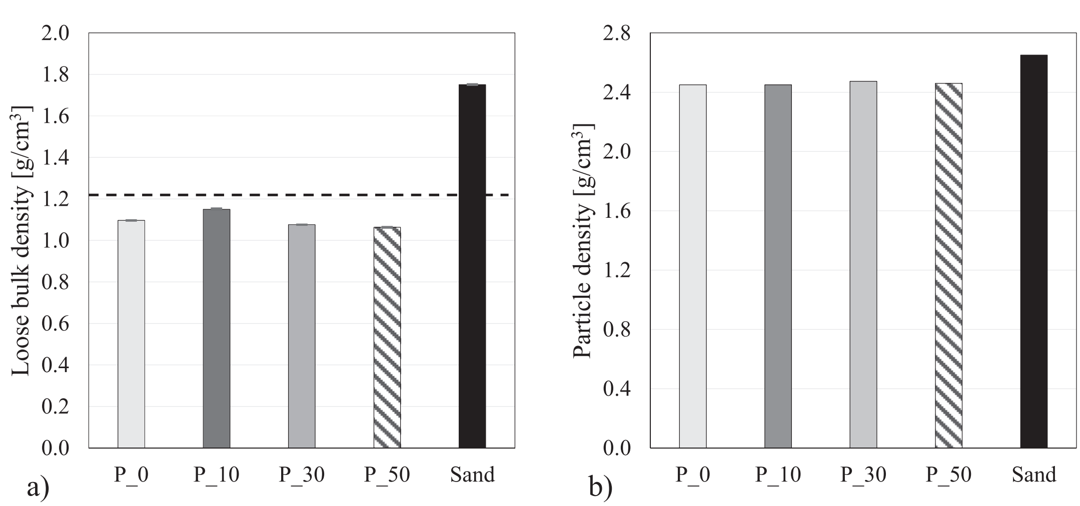
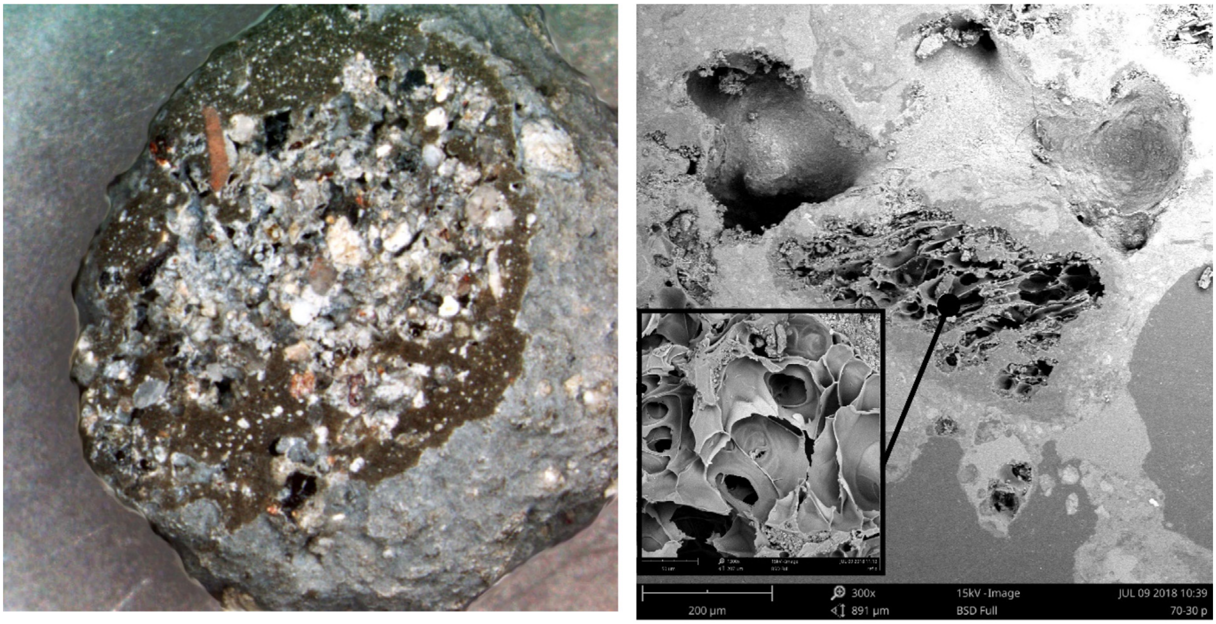


Fig. 4. a) Loose bulk density and b) Particle density of the manufactured pellets and BA-M used as core during the production. Sand is used as a reference.



a) b)

Fig. 5. a) Cross-section of the P_30, performed using an optical microscopy b) SEM pictures of the polished P_30 pellet, showing the internal structure of the pellet together with the enlargement of the porous core structure coming from the BA-M.

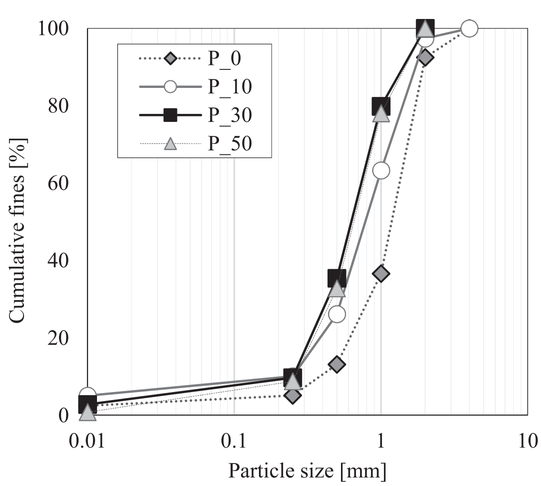


Fig. 6. Particle size distribution (PSD) of the manufactured pellets, including different FC replacement.

3.3. Evaluation of the reaction products

The XRD analysis of the pellets shows the presence of typical phases characterizing BA, such as quartz and calcite [3], but also unhydrated cement phases (e.g. C_2S, C_4AF), as well as hydrated phases such as calcium hydroxide (CH) and sulfoaluminate phases (e.g. ettringite (AFt) and monosulfoaluminate (AFm)) (Fig. 7a)–d)). During standard curing, the pellets show an increasing content of Friedel's salt (FS), a chloride containing Ca-Al double layer hydroxides (LDH), defined as hydrocalumite (HC) [27]. The presence of this reaction product is linked to the high chloride content detected in both FC and BA-M (Table 3), replacing the SO_4 ions of AFm phases with Cl^- [36]. The increasing intensity of the Friedel's salt peak indicates a greater content of this phase for a higher FC replacement, in agreement with the rising content of chlorides in the system. Compared to the standard cured samples, the decomposition of the Friedel's salt is visible in all HT cured samples, in agreement with the known decomposition temperature of LDH (85 °C, [27]). Compared to the results observed in a previous study testing HT on mortars blocks [11], the higher surface area exposed to hydrothermal treatment results in the complete decomposition of AFm phases and the consequent presence of gypsum in the XRD (Fig. 7a)–c)). However, the PC does not have the chemical composition needed for the formation of tobermorite phases (typically Ca/Si 0.83, Galvánková et al. [26], due to its high Ca/Si ratio (2.5 initially, 1.7 in C-S-H form [46]. Therefore, the formation of tobermorite is favored by the incorporation of reactive silicates coming from BA-M and FC, affecting the final Ca/Si during the treatment. HT is known for increasing the reactivity of normally unreactive silica-based phases such as quartz, creating favourable conditions for their dissolution and participation to the reaction products formation [4]. In this study, the reduction of the CH peak in the HT cured samples confirms the participation of those silica-based materials in the formation of C-S-H phases, such as tobermorite and xonotlite [4,11,50]. According to many studies, the presence of tobermorite should be beneficial for the development of higher mechanical strength, due to the lower calcium silica ratio of the matrix [39] and denser structure [9,14,34].

3.4. Mechanical performances

The spread flow diameter of the mixtures are similar independently on the FC content, and they all vary between ≈150–200 mm (Fig. 8a)). Differences in the rheological behaviour are related to the different loose bulk density (Fig. 4a)) and therefore replacement mass calculated of each fraction. Since the spread flow behaviour of the pastes is not affected by the presence of HT, only the standard cured samples are shown in Fig. 8a). The evaluation of flexural and compressive strength of mortars, including the manufactured pellets, is displayed in Fig. 8b) and c). In the

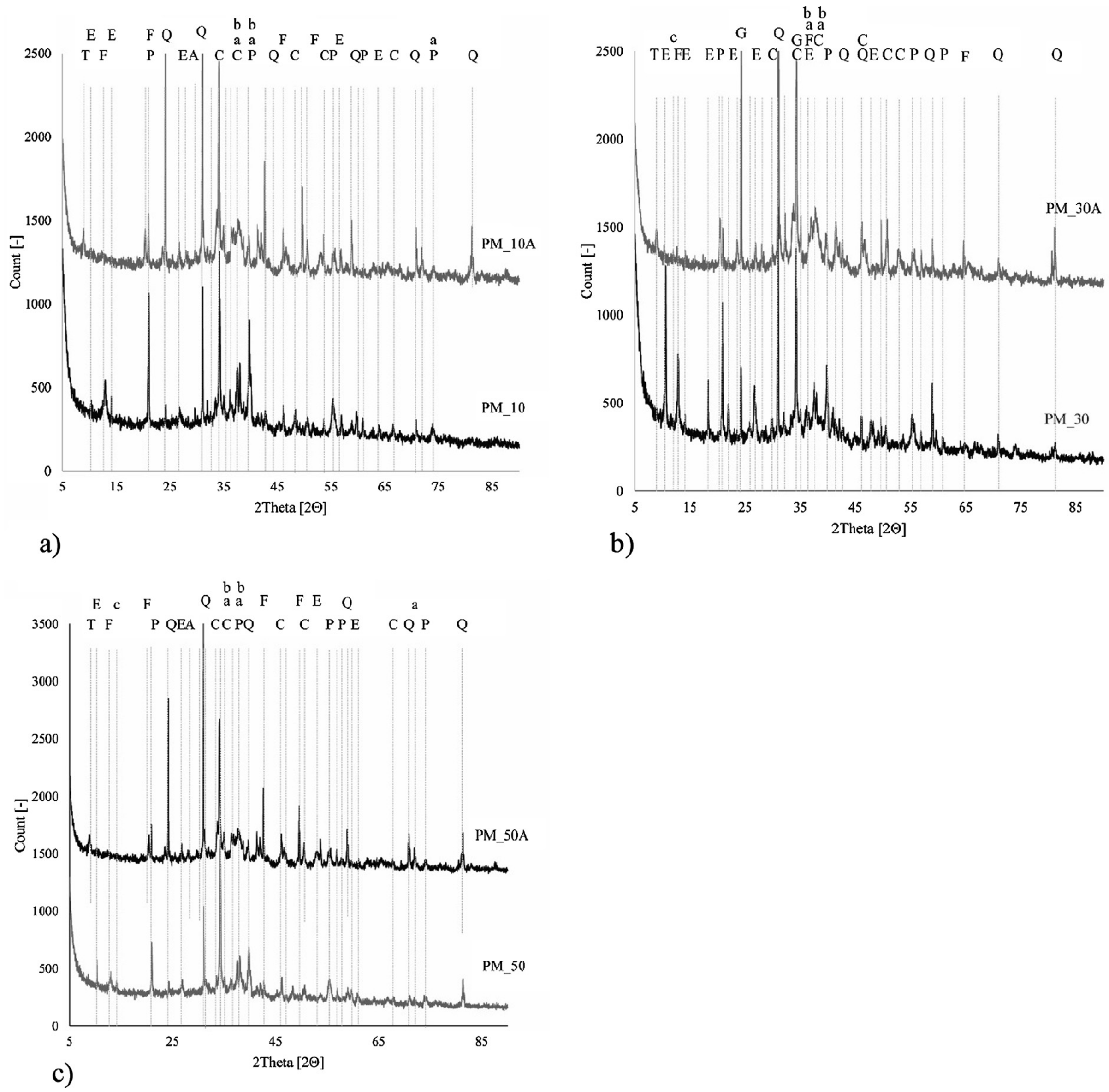


Fig. 7. Evaluation of the reaction products formed in the presence of hydrothermal treatment on the pellets in comparison with the untreated samples manufactured according to the same recipe a) P_10, b) P_30 and c) P_50. F-Friedel salt, P-Portlandite (Calcium hydroxide), E-Ettringite (Aft), C-Calcite, Q-Quartz, a-Alite, b-Belite, c- Ferrite (C4AF), G-Gypsum, T-Tobermorite.

presence of standard curing (Fig. 8b)), the performances of the mortars overcome the minimum 17.0 MPa compressive strength required for lightweight products, ranging between 25.5 and 35 MPa depending on the FC replacement rate. The pellets having only the PC as the external layer show the highest compressive strength (35.5 MPa). If PC is replaced with FC, the compressive strength decreases with increasing replacement (10% lower using P_10 and P_30, 28% lower using P_50), due to the high pore volume of the FC, contributing to the increase of the overall porosity of the pellets (Table 2).

Moreover, the less viscous paste obtained for P_30 and P_50 (Fig. 8a)) contributes to the increase of the matrix porosity, and thus to the lower compressive strength [17]. This trend is also displayed in the presence of HT, where the higher FC content leads to lower mechanical performance, for a minimum of 19.07 MPa for P_50A. Compared to standard cured pellets of the same design, HT lowers the compressive strength by 9%, 19% and 25% for P_10A, P_30A, P_50A mortars respectively (Fig. 8c)).

On the other hand, in the presence of pellets cured by HT, the flexural strength of the mortars improves by 14% and 2.8% for P_30A and P_50A, respectively, while a small drop is shown for P_10A (4.4%), compared to standard cured pellets. Despite the certain presence of tobermorite (Fig. 7, Section 3.3) after HT, the pellets are not able to withstand higher compression stresses, and they fail before the standard cured one. Since the tobermorite is derived from the conversion of cement reaction products (e.g. C-S-H) during the hydrothermal treatment [4], it is expected to be found mainly in the external layer of the pellets. This fact is supported by SEM analysis, where needle-like crystals are mainly observed externally to the pellet structure (Fig. 9a)), in correspondence of the cement layer. On the other hand internally, the formation of those minerals is not so evident (Fig. 9b)). The crystals are

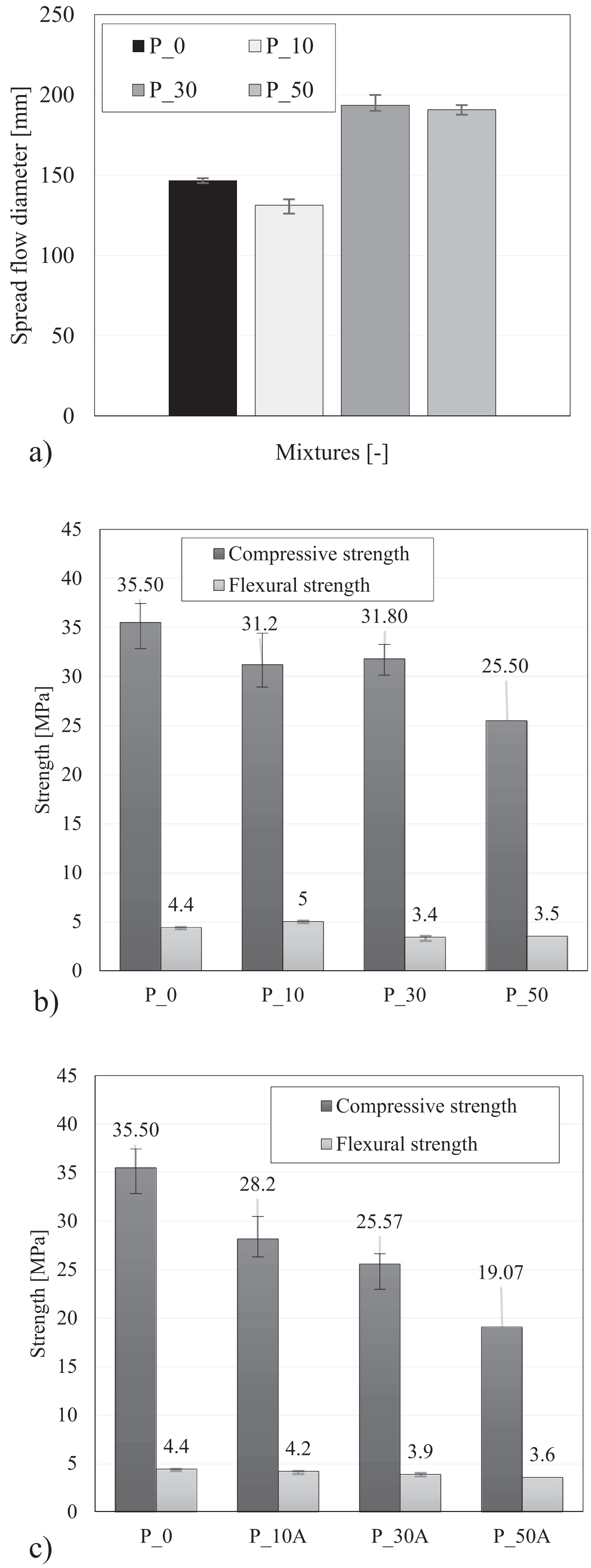


Fig. 8. a) Spread flow diameter of the mortars mixtures including MSWI BA pellets b) Compressive and flexural strength of the mortars including pellets cured in standard conditions c) Compressive and flexural strength of the mortars including pellets treated with hydrothermal curing for 6 h.

less densely organized, and they results smaller in size and length. Because of the absence of cementitious matrix inside the pellets (Fig. 9b)), those reaction products form in a lower amount, and they are not able to contribute to the formation of a denser structure, compromising the development of higher mechanical performances in compression. Despite this, the presence of needle-like crystals on the particle surface is beneficial for the bonding of aggregates and matrix, and it seems to contribute to the flexural strength of the mortar, improving the anchoring to the binder matrix. Based on the results obtained by the XRD analysis (Fig. 7, Section 3.3) and the similar shape [42], those minerals are identified as Tobermorite.

Finally, the high content of chlorides and sulfates available after the decomposition of AFm and Friedel's salt phases are expected to alter of the PC hydration either by accelerating the reaction of phases as C_3S [8] or by an increasing reformation of AFt phases especially in the ITZ, affecting its strength development.

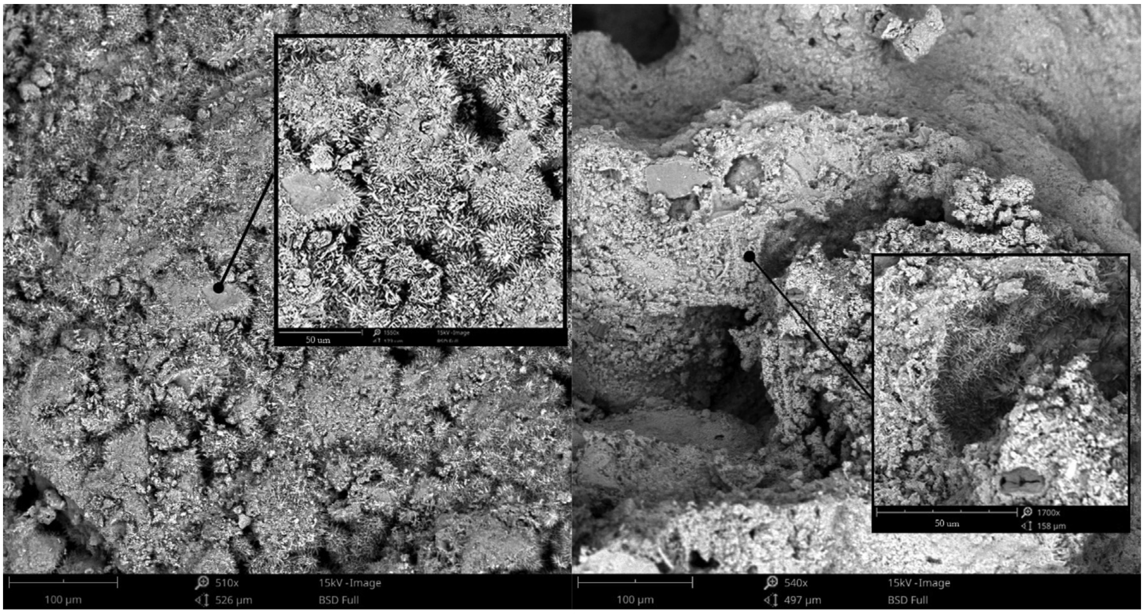
3.5. Leaching assessment of the pellets

3.5.1. Leaching comparison of the standard and hydrothermally cured pellets

The leaching behaviour of the pellets is tested as granular material (below 4 mm), according to EN 12457-2 [31]. The results shown in Fig. 10 describe only the elements above the legal limit and Ba, while the full range of heavy metals is presented in Appendix A.

Compared to the pure BA-M used as the pellet core, the leaching of the contaminants overcoming the legislative threshold (e.g. Mo, Cu, Sb, Cl, and SO_4) in the standard cured pellets are highly reduced. The chloride leaching from P_0, P_10, and P_30 is reduced by ≈65%, 75%, and 77% respectively. Despite this, the leaching of chlorides is above the Soil Quality Decree threshold for all standard cured pellets. Calculating the theoretical concentrations (C_t) for the leaching of Cl^- (Section 2.2, Eq. (3)), between 57% and 65% fewer chlorides have been released compared to the theoretical values, showing the efficiency of the immobilization in cement hydration products such as Friedel's salt (Section 3.3). The reduction in leaching takes place not only due to the chemical binding of contaminants in the reaction products [29] but also via sealing off the BA-M from direct contact with water. The leaching of copper increases with the presence of FC in the external coating, in agreement with the leaching behaviour shown in Section 3.1, Table 2. It reaches critical values for P_50 (1.4 mg/kg), suggesting that this pellets design is not suitable, even if an additional washing treatment of the FC is applied. The removal of heavy metals, such as Cu, is not effective using washing treatment due to its incorporation mainly in weathering products as calcite or organics [3], insoluble in low alkaline conditions (washing pH ≈ 11, Table 4).

After the 6 h hydrothermal treatment, the releases of chlorides and sulfates are increased. As shown in previous work [11], the presence of 6 h hydrothermal curing also induces the partial decomposition of AFt (but also AFm) which increases sulfate leaching, up to 28 times than the standard cured sample (P_50A). Moreover, as shown in Section 3.3, due to the high temperature applied the decomposition of Friedel's salt is promoted [27], causing the increase in chloride leaching by up to 158% (P_50A), compared to the standard cured sample. For lower contents of FC (P_10A), the sulfate leaching is only 3 times higher. As far as the heavy metals are concerned, after HT an improvement for the leaching of Ba (Fig. 10c)) is recorded, by up to 90% in P_50A. However, the release of other contaminants increases (Cu, Mo, and Zn leaching is increased 8, 1.7 and 176 times respectively) after treatment, showing that the HT is not always beneficial for the environmental impact of the final product. Finally, both PC and MSWI ashes contain Sb (Table 4), which during hydration is adsorbed in C-S-H or AFm phases forming antimonite-AFm ($Ca_4Al_2[Sb(OH)_6]_2(OH)_{12}*3H_2O$) [19,53]. Therefore, as previously discussed, the decomposition of AFm phases in the pellets after HT leads also to



a) b)

Fig. 9. SEM pictures of P_50A a) external surface in correspondence of the cement layer and b) internal structure.

an increase of Sb leaching out from HT lightweight pellets (P_30A and P_50A, Appendix A).

It is important to notice that the leaching behaviour is considered per mass of the final product, while the immobilization capacity depends mainly on the PC content. Therefore, compared to the HT reported in other articles, the lower PC content of the pellets seems to play the central part in the high leaching of SO_4, Sb and Mo, which increases after the HT (up to 96%, 60% and 41%, respectively) but lowered compared to the theoretical samples (up to 97%, 87% and 67%, respectively). A previous study has shown the inefficiency of HT for the immobilization of those ions even with higher PC content, having comparable leaching values to the standard cured samples [12].

3.5.2. Leaching of the mortars

After the use of the pellets as lightweight aggregates in mortars, the leaching behaviour of the final product is evaluated as granular material (Table 5). The chlorides leaching is highly reduced once the pellets are in contact with the cement matrix, but it does not satisfy the threshold values of SQD for Mor P_0, Mor P_50 and all the HT cured samples. Compared to the loose pellets, in the presence of a cement matrix, the Cl leaching reduces by 67% and 63% and sulfates by 52% and 67% for mortars made using P_10 and P_30, respectively, satisfying the legislation limit. Finally, Ba and Sb are problematic in the application of pellets manufactured using BA and CEM I 52.5 R. Trace elements such as Ba can replace Ca ions in C_2S and C_3S phases, due to the contamination of raw materials and fuels used in the clinker production [1,53] (Table 3, Section 3.1). Since the PC used in this study releases 13.8 mg/kg of Ba in the absence of BA, the leaching behaviour of the final pellet will be affected, causing it to exceed the legislation threshold for most of the samples. The high leaching measured for Sb in the mortars containing P_30A is mainly addressed to inhomogeneity of the sample. Comparing the results obtained after HT in the previous study and this one, it is clear that the different amount of PC used in the application plays a crucial role on the immobilization capacity of the final product. Moreover, another main difference in the two systems proposed is the BA content, ranging around 6% in Caprai et al. [12] and up to 78% in this study.

4. Conclusions

This article describes the manufacture of pellets, including a high content of MSWI BA. Hydrothermal treatment (HT) has been applied and compared to standard curing to improve the pellets mechanical and environmental performance. The following conclusions can be drawn:

- Using 80%wt. MSWI BA, lightweight pellets can be successfully produced, combining a coarser fraction (BA-M, 1–0.25 mm) as core and a fine one (FC, below 0.25 mm) as cement replacement for the outer coating. Having bulk density between 1.15 and 1.06 g/cm^3, they satisfy the requirement imposed by the EN 13055:2016 for lightweight aggregates classification.
- In standard curing, the pellets show the formation of Friedel's salt due to the high chloride content of both the core (BA-M) and the FC. Using HT, the decomposition of Friedel's salt and the formation of tobermorite are detected. Tobermorite presence is linked to the participation of reactive silicates from BA-M and FC, stimulated by the hydrothermal process.
- Mechanically, the mortars containing standard curing pellets achieve between 9% and 25% higher compressive strength than HT cured ones (between 25.5 MPa and 31.2 MPa), depending on the FC replacement. On the other hand, HT on pellets increases flexural strength up to 14% (3.9 MPa), thanks to the formation of tobermorite on the surface of the pellets, anchoring the pellet to the cement paste.
- In standard curing conditions, the formation of Friedel's salt and sulphoaluminate phases immobilize chloride and sulfate leaching, up to 30.9% and 98%, respectively. For heavy metals, the main contaminants retained are Sb, Cu, Mo and Zn having up to 90%, 81%, 80%, and 94% respectively lower leaching, compared to the theoretical values. However, the leaching of Ba increases up to 3 times, due to the presence of cement.
- Using HT, the decomposition of phases such as Friedel's salt causes an increase in the leaching of chlorides by up to 158%. The HT is also detrimental for the leaching of Cu, Mo and Zn increasing 8, 1.7 and 179 times respectively.

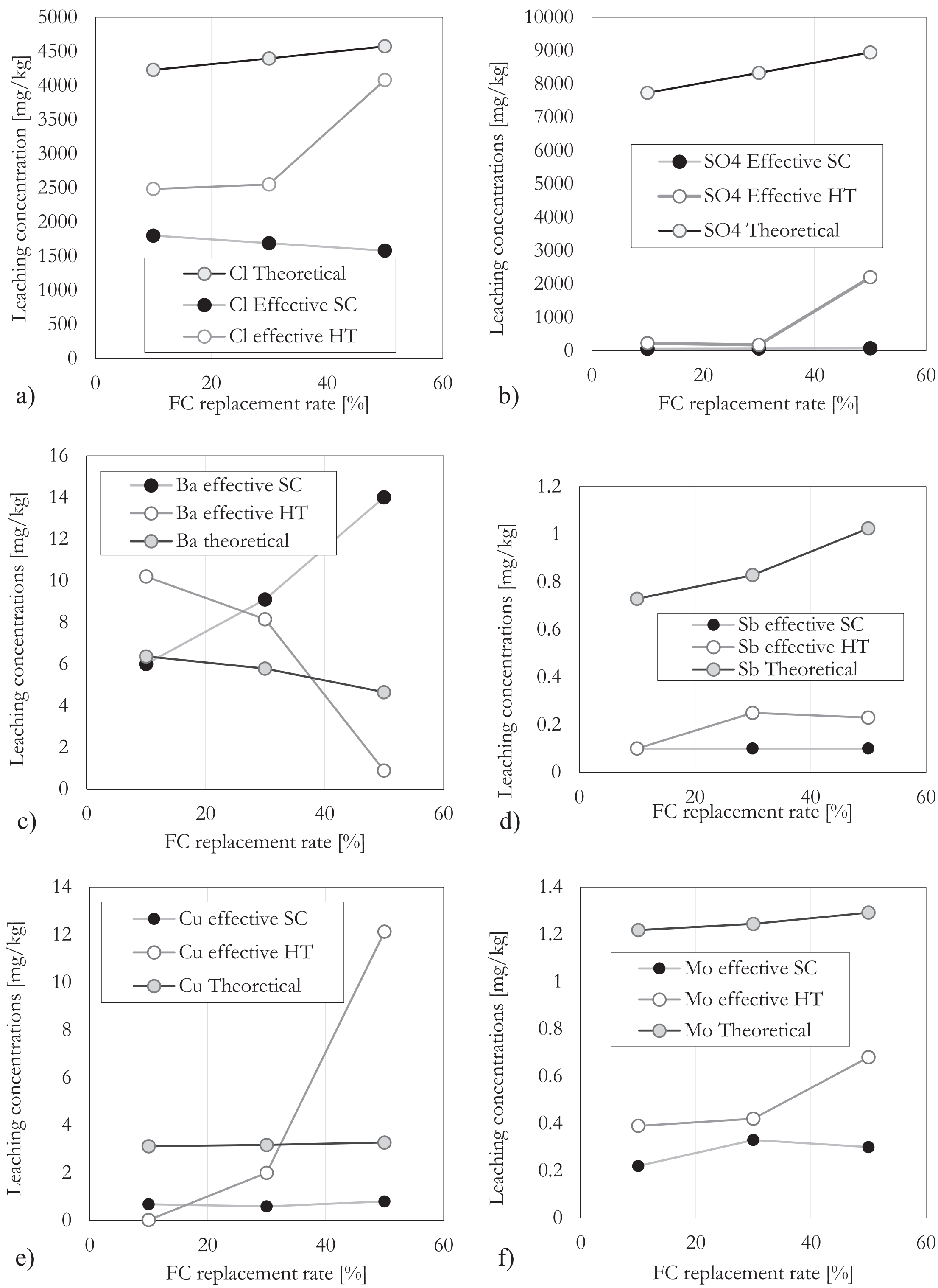


Fig. 10. Leaching comparison of the pellets cured in standard conditions and cured with hydrothermal treatment, with theoretical values calculated based on the leaching of each raw material. Values in bold are overcoming the current legislation for unshaped materials (Soil Quality Decree) [47].

- Once pellets are applied in mortars, the high content of PC helps the immobilization of Cl^- and SO_4^{2-} (up to 67% and 63%, respectively), but it causes a remarkable increase in barium leaching (up to 62% for P_50 compared to Mor P_50). For the reduction of this phenomenon, the use of a different cement type is recommended.
- The application of HT on pellets based on MSWI BA is not beneficial for mechanical and environmental performances, as it decomposes phases providing immobilization (Friedel's salt) and results in lower strength pellets. Moreover, it increases the overall embodied energy of the final product, resulting in higher CO_2 emission for pellets production. The lower

Table 5
Leaching assessment of the mortars, including pellets cured with both standard conditions and hydrothermal treatment. The values in bold indicate the elements overcoming the SQD threshold.

	Cl	SO_4	Ba	Cu	Mo	Sb	Zn
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
Mor P_0	**842.9**	34.2	25.89	<0.01	038	0.28	0.34
Mor P_10	591.8	31.9	**34.34**	0.1	<0.05	0.3	0.34
Mor P_30	579.0	24.1	**30.23**	<0.01	0.39	0.29	0.36
Mor P_50	**624.5**	26.1	**33.23**	<0.01	0.39	0.31	0.36
Mor P_10A	**884.19**	65.8	**22.86**	<0.3	0.34	0.15	0.34
Mor P_30A	**615.9**	67.3	**24.75**	<0.2	<0.05	**0.95**	0.58
Mor P_50A	**1015.5**	80.9	11.55	<0.4	<0.1	0.12	0.37
Unshaped limitation	616	2430	22.00	0.9	1.0	0.32	4.5

immobilization of the pellets, compared to the results previously obtained on mortars, depends on their lower cement content as well as on their much higher amount of the bottom ash.

Declaration of Competing Interest

The authors declare no competing financial interest.

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Appendix A

Complete table for leaching of contaminants of loose pellets manufactured including MSWI BA. The leaching assessment is performed according to EN 12457-2, one batch leaching test. Values in bold overcome the concentration of the contaminants allowed by the Soil Quality Decree.

Elements	SQD Unshaped material	P_0	P_10	P_30	P_50	P_10A	P_30A	P_50A
	mg/kg d.s.	mg/kg d.s.	mg/kg d.s.	mg/kg d.s.	mg/kg d.s.	mg/kg d.s.	mg/kg d.s.	mg/kg d.s.
pH	–	11.45	11.55	11.6	11.5	11.24	11.25	11.05
Antimony (Sb)	0.32	0.1	<0.1	0.1	0.2	0.1	0.25	0.23
Arsenic (As)	0.90	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3
Barium (Ba)	22.00	6.0	9.105	13.9	12.6	10.18	2.36	0.87
Cadmium (Cd)	0.04	<0.02	<0.02	<0.02	<0.02	**0.09**	**0.08**	**0.1**
Chromium (Cr)	0.63	0.02	<0.02	0.02	<0.02	0.32	0.43	0.41
Cobalt (Co)	0.54	<0.02	<0.03	0.03	<0.02	0.25	0.18	0.41
Copper (Cu)	0.90	0.68	0.59	0.79	**1.4**	<0.6	**2.0**	**12.16**
Lead (Pb)	2.30	0.1	0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Molybdenum (Mo)	1.00	0.22	0.33	0.34	0.4	0.39	0.42	0.68
Nickel (Ni)	0.44	<0.05	<0.05	0.06	<0.05	0.29	0.38	0.43
Selenium (Se)	0.15	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Tin (Sn)	0.40	<0.1	<0.1	0.1	<0.1	<0.1	<0.1	<0.1
Vanadium (V)	1.80	0.1	0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Zinc (Zn)	4.50	<0.01	<0.01	<0.01	<0.01	0.39	1.31	1.79
Chloride (Cl)	616	**2510**	**1800**	**1690**	**1580**	**2484.9**	**2550.8**	**4081.09**
Sulfate (SO_4^{2-})	2430	57.9	67.0	74.0	61.0	227.8	176.5	2203.8

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