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# Integral recycling of municipal solid waste incineration (MSWI) bottom ash fines (0–2 mm) and industrial powder wastes by cold-bonding pelletization

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ABSTRACT

The cold-bonding pelletizing technique is applied in this study as an integrated method to recycle municipal solid waste incineration (MSWI) bottom ash fines (BAF, 0–2 mm) and several other industrial powder wastes. Artificial lightweight aggregates are produced successfully based on the combination of these solid wastes, and the properties of these artificial aggregates are investigated and then compared with others' results reported in literature. Additionally, methods for improving the aggregate properties are suggested, and the corresponding experimental results show that increasing the BAF amount, higher binder content and addition of polypropylene fibres can improve the pellet properties (bulk density, crushing resistance, etc.). The mechanisms regarding to the improvement of the pellet properties are discussed. Furthermore, the leaching behaviours of contaminants from the produced aggregates are investigated and compared with Dutch environmental legislation. The application of these produced artificial lightweight aggregates are proposed according to their properties.



## 1. Introduction

Nowadays, the sustainable development of building materials is attracting worldwide attention (Omer, 2008; Brunner and Rechberger, 2015) considering resource saving and environment protection. In industries and building materials field, reduce, reuse and recycle are the primary ways to achieve the environmental and economic sustainable development goals (Kothari et al., 2010; Ahuja, 1997). The use of industrial wastes is widely studied and successfully applied in some cases, and the inherent properties of these wastes show their suitability to be used as building materials (Rivera et al., 2015; Tam et al., 2005; Al-Jabri et al., 2009; Xuan et al., 2016a, 2016b). However, the application of some wastes is limited before proper treatments due to their environmental impact (Fedje et al., 2010; Arickx et al., 2006).

Municipal solid waste incineration (MSWI) bottom ash is a primary solid by-product from incineration plant, which has a potential to be used as building materials (Tang et al., 2015). Series of treatments are already available to clean the coarse bottom ash (above 2 or 4 mm) which make this coarse fraction suitable to be used as aggregate in concrete (Keulen et al., 2015). Nevertheless, the sand-sized bottom ash fraction (BAF, under 2 or 4 mm) remains to be a problem due to the fact that it contains higher amounts of contaminants (such as Cu, Sb and Mo) and salts (such as chlorides and sulphates) which exceed the leaching limits according to legislation (Tang et al., 2016). Landfill is not preferable due to lack of space, waste of potential resource and risk of leakage. Hence, suitable application and treatment are in demand. However, the efficiency of the current treatments on this sand-sized fraction is limited because of its small particle size and higher surface area (Rahman and Bakker, 2013) and the treatment cost is too high comparing with its application potential. Thus, new methods or applications should be explored for BAF. There are other industrial wastes, such as coal fly ash (FA), paper sludge ash (PSA) and washing aggregate sludge (WAS) which can be used or recycled as building materials (Mozaffari et al., 2009; González-Corrochano et al., 2010). Nevertheless, the environmental impact of contaminants is also one of the issues need to be considered during their application. Considering the solid wastes properties and their application potentials, an integrated recycling method is of interesting to be developed which can combine these wastes together, maximize their advantages (pozzolanic properties, etc. (Aubert et al., 2006), meanwhile minimize their disadvantages (leaching problems, etc. (Tang et al., 2015).

Currently, the cold bonded pelletizing technique is widely used to recycle industrial waste powders for producing artificial aggregates, and the raw materials (powder level) used are extended to

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various waste powders in recent years (Geetha and Ramamurthy, 2010a; Tsai, 2012; Gesoğlu et al., 2007, 2012; Thomas and Harilal, 2015; Dutta et al., 1997; Ferone et al., 2013). It is proved to be an effective way for recycling powdered wastes, however, there is hardly research done on the wastes which have a particle size larger than powder (<125 μm), such as sand-sized fractions (particles under 2 or 4 mm).

Hence, in this study, the cold bonded pelletizing technique is applied as an integral recycle method for municipal solid waste incineration (MSWI) bottom ash fines (0–2 mm, BAF), coal fly ash (FA), paper sludge ash (PSA) and washing aggregate sludge (WAS). Nowadays in the Netherlands, these solid wastes need to be used as useful/valuable materials instead of landfill, to reduce their environmental impact. An artificial lightweight aggregate is successfully produced based on this technique and the combination of the selected raw materials. Moreover, the influence of the raw materials on the properties of the aggregate and the methods to improve the aggregate properties are investigated. The leaching behaviour of these aggregates are evaluated and compared with Dutch legislation. Finally, the application potential of these aggregates is suggested.

## 2. Materials and methodology

The materials used in this study are shown in Fig. 1. The binder applied is Ordinary Portland Cement (OPC) CEM I 42.5 N (ENCI, the Netherlands). The industrial waste powders chosen here are combustion fly ash (FA), paper sludge ash (PSA) and washing aggregate sludge (WAS) from a power plant, a paper recycle company and a gravel washing factory in the Netherlands, respectively. The ground granulated blast furnace slag (Gao et al., 2015) (GGBS, ENCI, the Netherlands) is used as OPC alternative in one of the recipes. The municipal solid waste incineration (MSWI) bottom ash fines (BAF) are provided by a waste-to-energy plant in the Netherlands (Moerdijk, Attero.) which has around one million tonnes annual waste processing capacity of solid waste. The BAF has a particle size smaller than 2 mm, chosen by sieving from the wet MSWI bottom ash heap in the plant. Polypropylene fibre (PPF) with a length of 3 mm, density of 0.91 kg/m$^3$ provided by FBG (the Netherlands) and Bonar (England) is used as reinforced fibre.

The X-ray fluorescence (XRF, Epsilon 3, PANalytical) and the X-ray diffraction (XRD, Cu tube, 40 kV, 30 mA, 3–75°, 0.02°/step, 0.2°/min) are employed to determine the chemical compositions and crystalline phases present in the materials, respectively. The main chemical compositions (shown in Table 1) of these industrial wastes belong to the $SiO_2$-$CaO$-$Al_2O_3$-$Fe_2O_3$ system. The crystalline phases of the raw materials are shown in Fig. 2. WAS as a waste sludge from washing aggregate contains mainly $SiO_2$, and a considerable amount of $Al_2O_3$ and $Fe_2O_3$; the main crystalline phases in WAS are quartz, magnetite, clay mineral of the chlorite family, and feldspar. PSA consists high amount of CaO, and $SiO_2$ and $Al_2O_3$, its main crystalline phases are calcite, portlandite, gehlinite, and calcium silicate. The main chemical compositions in FA are $SiO_2$, and then higher amount of $Al_2O_3$ compared with the other waste materials in this study; its crystalline phases are quartz and mullite. BAF contains quartz, calcite, hematite, feldspar and anhydrite.

The particle size distributions (PSDs) of the powders are measured using laser diffraction (Mastersizer 2000 Malvern) and the PSDs of the particles (BAF and produced aggregates) are determined according to EN 933-2 (1995). BAF has a particle size under 2 mm, around 50% between 0.5 and 2 mm. GGBS has a very similar PSD as OPC, and WAS has a coarser PSD than OPC while similar as PSA. The PSD of FA is coarser than PSA (Fig. 3). The specific densities of the materials are measured using a helium pycnometer (AccuPyc II 1340) and shown in Table 1; and the bulk density of the produced aggregates are determined according to EN1097-3 (1998). The influence of PSA and FA on the OPC hydration is studied by eight-channel isothermal calorimeter (TAM Air, Thermometric).

The disc pelletizer (Fig. 4) is used for the pelletization process, the diameter of the disc is 100 cm and its collar height is 15 cm. The raw materials are homogenously mixed in a concrete mixer (BAF as received contains a water content of around 18%, it is used without drying), and then around 9–12 kg mixed material is fed to the disc. The water is sprayed during the first several minutes, and the disc is continuously running for the generation and compaction of granulates. The process is continuously running by adding mixed materials and then collect the produced aggregates which dropped off automatically in order to simulate the practical procedure in industry. The produced pellets are sealed in buckets at room temperature before test. By adjusting the pelletizing parameters (speed, angles, etc.), the particle size of the produced aggregate can be modified. The water absorption of the aggregates is determined according to EN 1097-6 (2013) and the crushing resistance of the aggregates is measured according to EN 13055-1 (2002) (Annex A, procedure 1). The cross sections of the pellets are observed using optical microscopy and scanning electron microscope (SEM, Quanta 650 FEG, FEI).

To estimate the environmental impact of the BAF and the generated aggregate, column leaching tests according to Dutch standard NEN 7383 (2004) were performed. The liquid to solid ratio is kept at 10 L/kg; during the test, the water is forced to flow through the material from the bottom to the top of the container

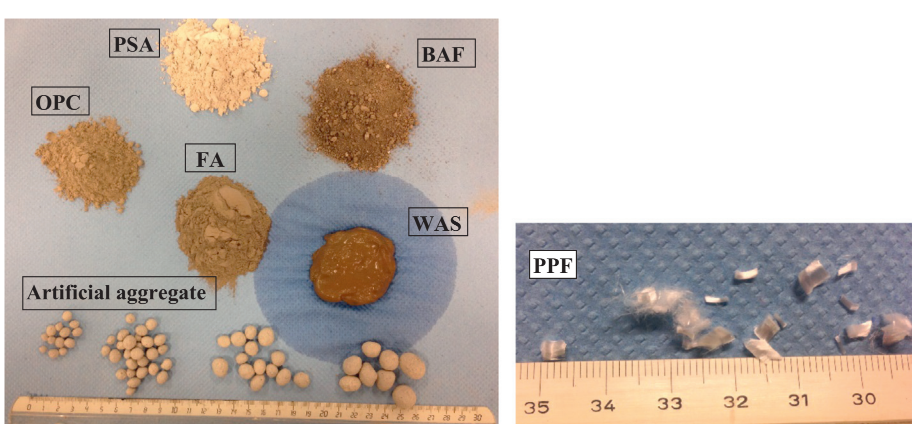



**Fig. 1.** The raw materials used and aggregate produced.

**Table 1**
The chemical compositions and specific densities of the raw materials, Ordinary Portland Cement (OPC), ground granulated blast furnace slag (GGBS), washing aggregate sludge (WAS), paper sludge ash (PSA), coal fly ash (FA), municipal solid waste incineration (MSWI) bottom ash fines (BAF).

| | OPC | GGBS | WAS | PSA | FA | BAF |
|---|---|---|---|---|---|---|
| LOI | 2.16 | 1.25 | 3.64 | 14.59 | 5.31 | 9.17 |
| CaO | 67.85 | 40.01 | 1.46 | 54.94 | 6.18 | 18.58 |
| $SiO_2$ | 14.85 | 30.19 | 73.31 | 13.64 | 45.20 | 39.13 |
| $Al_2O_3$ | 3.62 | 12.69 | 11.29 | 8.64 | 27.49 | 7.60 |
| $Fe_2O_3$ | 3.33 | 0.60 | 6.00 | 0.99 | 6.63 | 12.93 |
| $Na_2O$ | [a] | [a] | 0.61 | [a] | 0.96 | 1.02 |
| $K_2O$ | 0.82 | 0.62 | 1.85 | 0.46 | 2.17 | 1.09 |
| MgO | 1.57 | 9.08 | 0.89 | 2.09 | 1.36 | 1.93 |
| $P_2O_5$ | 0.39 | 0.00 | 0.07 | 0.30 | 0.78 | 0.86 |
| $TiO_2$ | 0.30 | 1.40 | [a] | 0.74 | [a] | [a] |
| MnO | 0.08 | 0.27 | 0.12 | 0.03 | 0.07 | 0.17 |
| CuO | 0.02 | [a] | 0.00 | 0.07 | 0.02 | 0.38 |
| ZnO | 0.07 | 0.00 | 0.01 | 0.08 | 0.05 | 0.66 |
| Cl | 0.11 | 0.06 | 0.00 | 2.24 | [a] | 0.29 |
| $SO_3$ | 4.46 | 3.55 | 0.16 | 0.99 | 1.70 | 4.33 |
| Others | 0.36 | 0.28 | 0.59 | 0.19 | 2.08 | 1.89 |
| Density ($g/cm^3$) | 3.1 | 2.93 | 2.68 | 2.71 | 2.32 | 2.69 |

[a] Not detected.

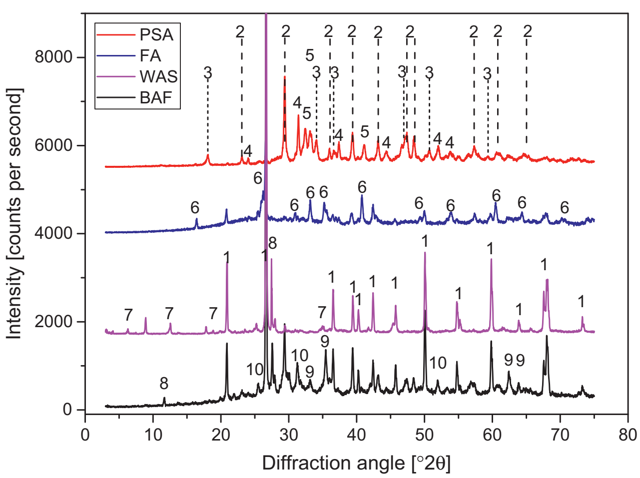



**Fig. 2.** The XRD pattern of the waste raw materials (1: Quartz; 2: Calcite; 3: Portlandite; 4: Gehlenite; 5: Calcium silicate; 6: Mullite; 7: Chlorite; 8: Feldspar; 9: Hematite; 10: Anhydrite) (PSA = paper sludge ash, FA = coal fly ash, WAS = washing aggregate sludge, BAF = municipal solid waste incineration bottom ash fines).

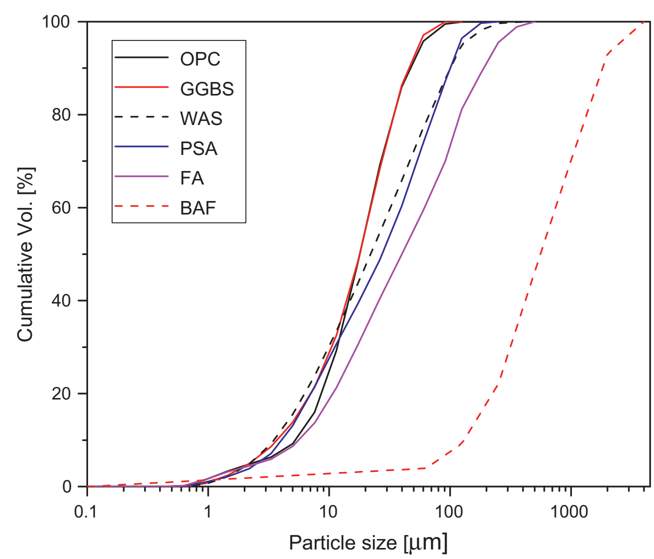



**Fig. 3.** The particle size distribution of the raw materials used, Ordinary Portland Cement (OPC), ground granulated blast furnace slag (GGBS), washing aggregate sludge (WAS), paper sludge ash (PSA), coal fly ash (FA), municipal solid waste incineration (MSWI) bottom ash fines (BAF).

for a certain period. The concentration of chemical elements in the eluate is analysed using inductively coupled plasma-atomic emission spectrometry (ICP-AES) according to NEN 6966 (2005), and the content of chloride and sulphate is determined through high performance liquid chromatography (HPLC) following NEN-EN-ISO 10304-2 (1996). The leaching values of studied elements are compared with the limit values according to the Dutch legislation (Quality Decree, 2008).

## 3. Results and discussion

### *3.1. Compatibility of industrial solid wastes during the integrated recycle*

In other researchers' studies (Baykal and Doven, 2000; Manikandan and Ramamurthy, 2007), it is concluded that the binder amount and the moisture content of the raw materials have significant influences on the pelletizing procedure and the properties of the produced aggregates. Moreover, the amount of binder generally used is around 10% wt. of total solids (Baykal and Doven, 2000; Colangelo et al., 2015). In order to compare the results with those reported in literature, the OPC content for our first trial mixture (sample M1) is fixed at 10% wt. of total solids. The washing aggregate sludge (WAS) in this study has a water content of around 50%, considering the amount of liquid needed for producing the aggregates (normally 21–31%, Gesoğlu et al., 2007, 2012), the total solid amount of WAS is fixed at 5% of the total solid raw materials. WAS has a fine particle size (as shown in Fig. 3) and is a water solid mixture, which can play a role as wetting and gluing agent at the pre-mixing stage to increase the growing speed of the pellets. To study the possibility of using BAF (contains coarser particles than powders) as one ingredient for producing artificial aggregates by pelletization and compare the properties of the pellets produced by others, coal fly ash (FA) is used as one powder ingredient and the amount of BAF for Mix 1 is chosen and fixed between 10 and 40% of the total solids. It is worth to mention that the exact value of BAF used in this study is confidential of the waste-to-energy plant (Attero) at this stage, hence cannot be shown yet. Moreover, the use of BAF amount can be various due to its property variety in different plants. Therefore, a range of

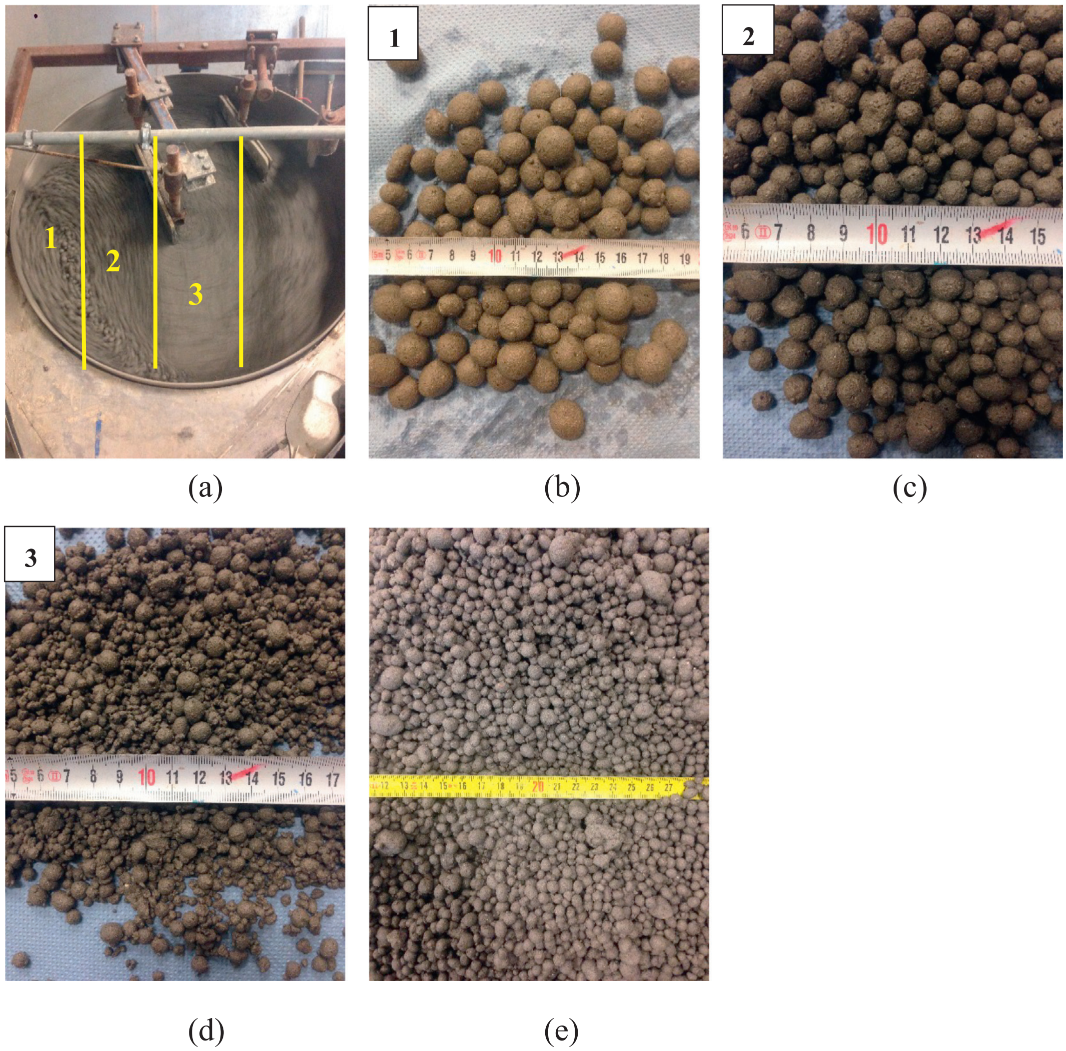



**Fig. 4.** The device and produced aggregates (a) zone 1–2: area to collect the pellets; zone 3: area to add the raw mixed materials; (b) pellets collected from zone 1; (c) pellets collected from zone 2; (d) pellets collected in zone 3; (e) pellets collected from zone 1–2 for future use.

BAF amount is given as indicator for others' research and the exact value we use for our research is in between this range. The proportions of solid materials of the first trial mixture is shown in Table 2.

The particle size distributions of the aggregates produced (M1) are shown in Fig. 5. The particle size of M1 is mainly between 2 and 16 mm, of which particles between 4 and 16 mm accounts for around 96% wt. The bulk density of this produced aggregate is around 840 $kg/m^3$ (Fig. 6) and water absorption is around 21% (Fig. 6). According to EN 13055-1 (2002), the bulk density of the aggregates lower than 1200 $kg/m^3$ is categorized as lightweight aggregate. The crushing resistance of this aggregate is increasing with the curing time and this increase is relatively slow after

**Table 2**
The recipe design for artificial aggregate production (proportions of dried solid materials).

| Samples | Binder | Powders | | | Coarser particles | Glue agent | Reinforcement |
|---|---|---|---|---|---|---|---|
| | OPC | GGBS | FA | PSA | BAF[a] | WAS | PPF |
| | [% total solid] | [% OPC] | [% total FA + PSA] | | [% total solid] | [% total solid] | [vol.%] |
| M1 | 10 | 0 | 100 | 0 | 10–40 | 5 | 0 |
| 5C | 5 | 0 | 50 | 50 | 40–75 | 5 | 0 |
| 10C | 10 | | | | | | |
| 15C | 15 | | | | | | |
| 6C | 6 | 67 | | | | | |
| No PSA | 10 | 0 | 100 | 0 | 40–75 | 5 | 0 |
| No WAS | | | 50 | 50 | | 0 | |
| 0.5% PPF | 10 | 0 | 50 | 50 | 40–75 | 5 | 0.5 |
| 1.0% PPF | | | | | | | 1.0 |
| 2.5% PPF | | | | | | | 2.5 |
| 4.5% PPF | | | | | | | 4.5 |

[a] The amount of BAF is fixed and the exact value is in the range shown in the table.

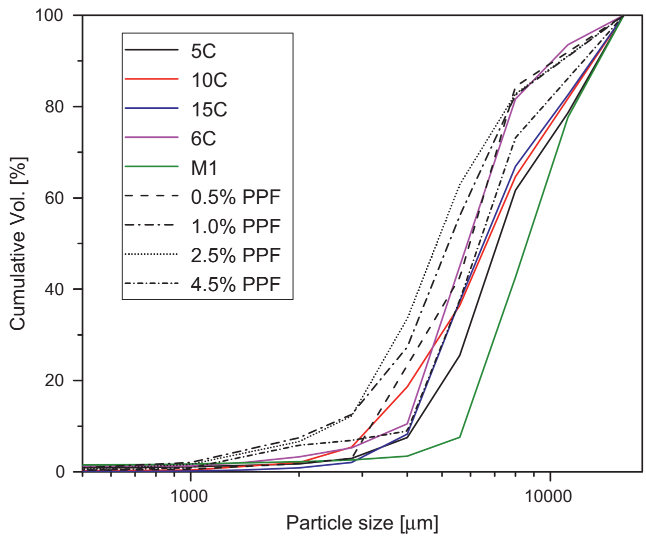



**Fig. 5.** The particle size distributions of the produced aggregates (see Table 2).

7 days curing. The crushing resistance is around 5.7 MPa (Fig. 7) after 28 days curing. The cold-bonding aggregates produced with similar methods as reported in Colangelo et al. (2015), Cioffi et al. (2011) and Ferone et al. (2013) have a bulk density around 1200–1600 kg/m$^3$ and water absorption about 8–20%, and the crushing resistance is between 2 and 6 MPa. Hence, it is observed that the aggregate (M1) in this study has a lower bulk density and higher water absorption, which indicates that M1 has higher porosity; while it has a higher crushing resistance compared with others' products, this may be attributed to the addition of MSWI bottom ash fines (BAF) instead of powders only. The mechanism of the contribution from BAF can be explained by the following hypothesis: firstly, the BAF can act as 'aggregates' in the pellets which enhance the skeleton strength of pellets (Fig. 8(a)), and BAF has irregular particle shape which can have better bonding strength between the paste matrix (Fig. 8(c) and (d)); secondly, BAF has smaller surface area compared with powered materials with same volume, which means less binder is needed to bind the particles. Hence, M1 can reach higher crushing resistance with same binder amount, although it has a higher porosity.

From the above shown results, it can be addressed that BAF and the chosen industrial powered wastes (coal fly ash and washing aggregate sludge) have good compatibility to be recycled using pelletization technique, and a type of artificial lightweight aggregates with comparable or even better properties than the ones in literature can be produced.

It is reported that the cold bonded lightweight aggregate can be used in concrete as aggregate replacement (Güneyisi et al., 2015a; Gesoğlu et al., 2006). However, it is also noticed that the strength of the pellets has a remarkable influence on its application in concrete (Thomas and Harilal, 2015; Colangelo et al., 2015; Güneyisi et al., 2015b), such as decreasing the concrete strength due to its lower strength compared with natural aggregates, which limits the amount of artificial aggregate that can be used. Hence, the investigation of methods to improve the properties of the cold-bonding lightweight aggregate by integrated recycle of industrial powder wastes and BAF in this study is focused, a comprehensive study on which is shown in Section 3.2.

### *3.2. Investigation of pellet properties improvement*

In nowadays research, the general methods for improving the pellet strength are mainly adjusting the pelletizing parameters (Baykal and Doven, 2000; Harikrishnan and Ramamurthy, 2006), changing the raw materials' properties (Thomas and Harilal, 2015; Dutta et al., 1997; Baykal and Doven, 2000; Manikandan and Ramamurthy, 2007) and curing condition (Manikandan and Ramamurthy, 2008; Gomathi and Sivakumar, 2015), etc. In the present study, the parameters investigated are binder amount (samples 5C, 10C and 15C), proper waste powder materials as additives (samples 6C, No PSA and No WAS), BAF (samples M1 and No PSA) and polypropylene fibre (samples 0.5–4.5% PPF). The design of the mixtures and labels of the samples are shown in Table 2. The properties of the produced aggregates are shown and discussed below.

#### *3.2.1. The particle size distributions (PSDs) of the produced aggregates*

For all the samples, there is very small amount of pellets above 16 mm, and these big pellets are not well compacted which have irregular shape and weak pellet strength. This fractions generally accounts for less than 5% of the total pellets produced in our study, therefore this fraction is sieved out (as performed in pilot plant).

During the pelletizing procedure, it is observed that the generation and growth of pellets in sample M1 is earlier and faster than other samples, while samples with PPF take longer time to grow. The generation speed of pellets during the pelletization follows the order as: M1 > 10C (5C, 6C, 15C) > No WAS (No PSA) > samples with PPF. However, this difference is very minor. It can be addressed that increasing the BAF amount and addition of PPF decrease slightly the generation speed of the pellets, while adding PSA or WAS result in shorter time needed for the generation of pellets during the procedure.

The particle size distributions of all the aggregates under 16 mm produced in this study are shown in Fig. 5. It can be seen that pellets below 2 mm accounts for approximately less than 7% of the total for all the mixtures, and the pellets produced with PPF have a slightly higher amount of particles under 2 mm than other samples. It can be seen from the PSD graph that the M1 sample has a coarse particle size than other mixtures, while the samples with PPF have finer particle sizes. This can be explained by the following reasons: firstly, M1 has a higher power/BAF ratio, which means more water layers will be formed on the small particles, this water layer make the pellets grow fast, hence the pellets are bigger than other samples within the same pelletizing duration. It is also noticed that M1 has some particles which are irregular, similar as shown in others' work (Colangelo et al., 2015); while the particles of the samples except M1 in this study are more roundish as shown in Fig. 4. This may indicate that the pellets containing only powder materials are more difficult to be compacted during the pelletizing procedure than the ones with BAF. For the samples with PPF, the hydraulic property of the fibre influences its compatibility with all the solids, and the PPF needs more powder to compact them together which may result in hollows in the pellets as shown in Fig. 8(b).

From the above shown results, it can be concluded that the use of PSA or WAS can shorten the growth time of pellets, the decreasing of BAF content also benefit the growth of pellets, however the freshly produced pellets are more difficult to be compacted, thus the pellets' shape is more irregular. The aggregates produced with higher powder/BAF ratio achieve bigger particle size than others and the addition of PPF slightly disturbs the growth of pellets.

#### *3.2.2. Bulk density and water absorption of the produced artificial lightweight aggregates*

The bulk density and water absorption are essential parameters for evaluating the cold-bonding lightweight aggregates, which has a significant influence on its application.

It can be seen in Fig. 6 that the pellets of all the samples have a bulk density around 835–927 kg/m$^3$, which is below 1200 kg/m$^3$

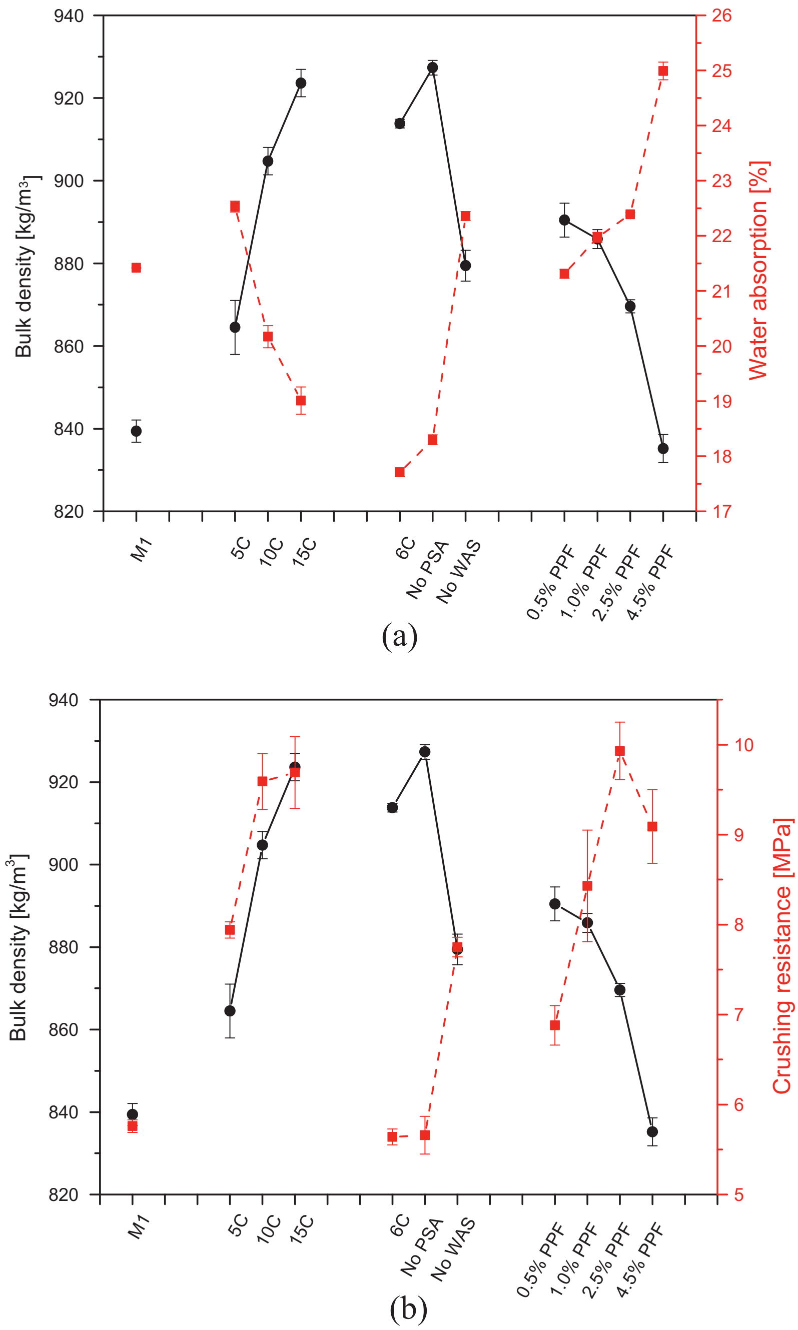



**Fig. 6.** The relationship between bulk density, water absorption (a) and crushing resistance (b) of the produced aggregates after 28 days curation.

and they all can be categorized as lightweight aggregates according to EN 13055-1 (2002). The lightweight aggregates produced in the present study have a water absorption of 17–25%, which is similar with the one produced in literature using powders (Colangelo et al., 2015; Cioffi et al., 2011; Ferone et al., 2013). Furthermore, it is shown that the bulk density and water absorption of these aggregates has an inverse relationship, a similar result is reported (Bernhardt et al., 2013).

Comparing samples 5C, 10C and 15C, it can be seen that the bulk density increases with the increase of binder amount, while water absorption has a decreasing trend. This can be attributed to the fact that the higher binder amount mostly contributes to a lower porosity (Manikandan and Ramamurthy, 2008) and denser microstructure of the pellets and cement have higher density than the other raw materials, which increases the total density when its amount is increased. Similar results were observed in a previous study (Thomas and Harilal, 2015). For samples with PPF, their bulk densities decrease with the increase of PPF content and the water absorption increase. This is due to the fact that the PPF is not very easy to be glued and compacted in the pellets, hence the microstructure of the pellets is not as dense as the other samples; in addition, the PPF has very low density compared with OPC or BAF (Table 1). Sample M1 has lower bulk density and higher water absorption than sample No PSA, which indicates that increasing BAF amount can result in lower porosity of the produced aggregates. The higher surface area of the powders than BAF results in a higher amount of water absorbed on their surface during pelletization, which may form a water layer force between particles. Subsequently, the compaction of the pellets is more difficult than the sample with BAF. Moreover, after the evaporation of the water,

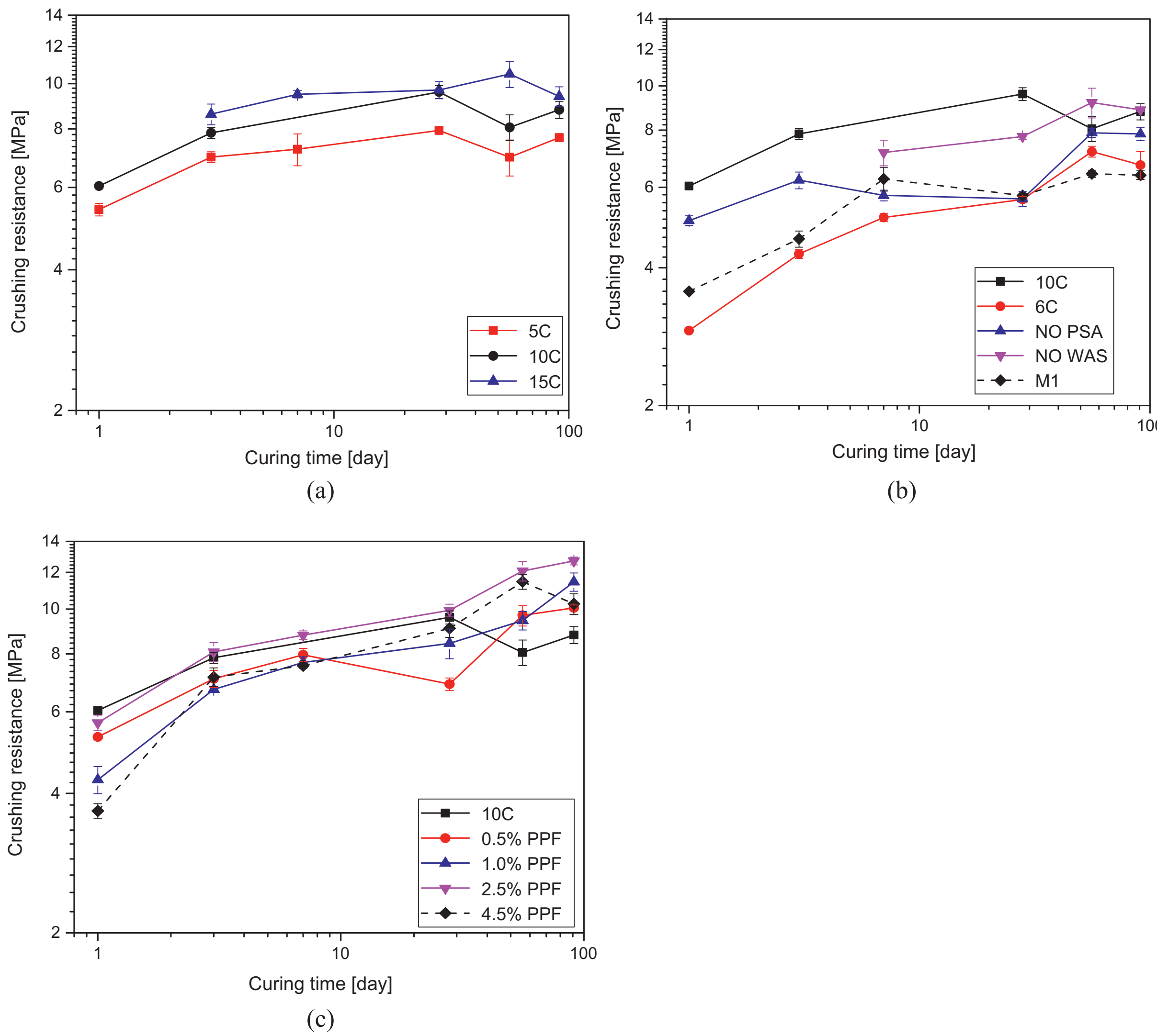



**Fig. 7.** The crushing resistance of the aggregates after different curing times.

higher amounts of capillary pores are left which results in a higher porosity. Thus, the sample with higher powder/BAF ratio has lower bulk density. Sample 6C shows an increased bulk density and decreased water absorption compared with sample 10C, while sample No WAS shows the opposite trend. The WAS can enhance the glue of the particles together, also its finer particle size may contribute to a slightly denser microstructure.

It can be briefly summarized that increasing binder amount (comparison of 5C, 10C and 15C), BAF content (M1 and No PSA) and PSA (10C and No PSA) amount can result in the decrease of the porosity of the produced lightweight aggregates while the increase of PPF content results in the increase of aggregate porosity.

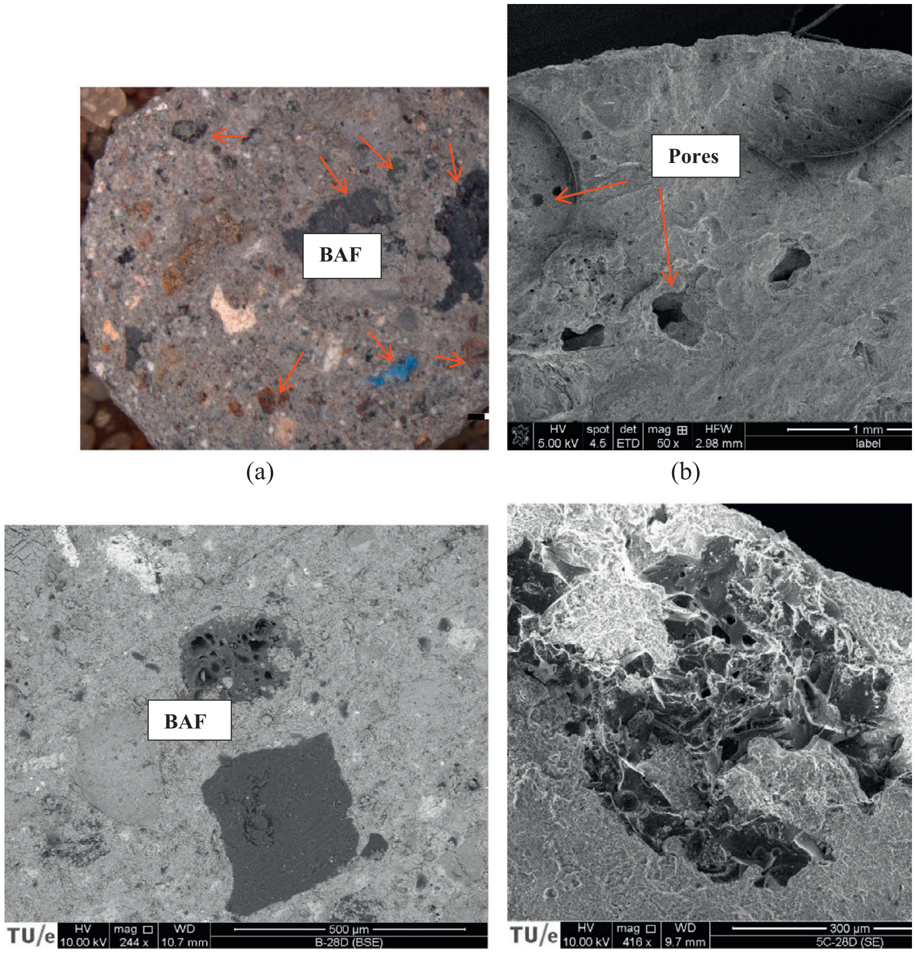
### 3.2.3. Crushing resistance of the produced lightweight aggregates

The 28-day crushing resistance (CR) of the samples produced in this study is around 5.64–9.93 MPa (Figs. 6 and 7). Fig. 7 shows the CR development along with curing time and the CR of all the samples is increasing with the curing time and this increase is more dramatic at the early age (around 70–85% can be reached).

Fig. 7(a) shows that the CR has an increasing trend with the order of sample 5C, 10C and 15C during the whole curing time. After 91 days, the CR of 10C and 15C is higher than 5C by 14.7% and 22.5%, respectively. The binder amount has an influence on the CR and higher amount of binder results in higher CR, and Fig. 6(b) shows that the bulk density of these samples has positive correlation with the binder amount. This is due to a denser microstructure and better bond strength between particles and matrix are obtained when the binder amount is increasing. Similar results were obtained in others' research regarding to artificial aggregates produced by only power materials (Thomas and Harilal, 2015; Colangelo et al., 2015). It can be seen in Fig. 9 (a) and (b) that the hydration products grow well around the particles, and the BAF particle is well embedded in the matrix and surrounded by the binder hydrates and fly ash particles, which contributes to the pellet strength.

The CR comparison of sample No PSA and 10C (Fig. 7(b)) shows that the addition of paper sludge ash (PSA) has a positive effect on the CR, sample 10C containing PSA obtains around 12% higher CR than sample No PSA after 91 days curing. In addition, Fig. 6(b) shows that sample No PSA has higher bulk density than 10C which indicates a good compaction of aggregate, while its CR is lower compared to sample 10C, this is due to the contribution of PSA.

Fig. 10 shows the influence of FA and PSA on the cement hydration by isothermal measurement. It can be seen that the addition of PSA contributes to an earlier heat peak compared with pure OPC




(a) (b)

(c) (d)

**Fig. 8.** The optical microscopy and scanning electron microscopy (SEM) graphs of the cross section of the pellets (a. and c: sample 10C; b: sample 2.5% PPF; d: sample 5C).

and its total heat released is higher than that of OPC, while addition of FA leads to the retardation of the heat peak and the total heat is lower than OPC. This is due to the fact that firstly PSA contains calcium silicate and portlandite (Fig. 2) which contributes to cement hydration (Ferrándiz-Mas et al., 2014), while FA is reported to has pozzolanic properties which takes long time to show up (Bentz et al., 2011). Therefore, early strength which is necessary for resisting the aggregates shape can be improved by adding PSA, while the longer time strength can be increased by FA. Hence, the combination of FA and PSA benefits the aggregates properties.

Sample No WAS and 10C has very similar CR (slightly higher after 91 days), and the addition of washing aggregate sludge (WAS) slightly decreases the CR of pellets. The WAS is an inert material which does not contribute to the cement hydration, however, the production of the pellet can be shortened due to its fine particle size and high water absorption. Sample 6C has lower CR compared with 10C and comparable to sample 5C and its CR is increasing steadily. It is reported that the ground granulated blast furnace slag (GGBS) can result in higher strength and lower porosity of concrete due to its pozzolanic property (Oner and Akyuz, 2007), however, this is not observed in the produced aggregates. The possible reasons can be that the pozzolanic property needs a longer time and higher pH.

Comparing sample M1 and No PSA, the amount of BAF is increased by around 25% wt. Sample M1 has a lower CR and after 91 days sample No PSA has a CR higher than M1 by 23%. Moreover, as addressed in Section 3.1, M1 has a higher CR than other cold bonded lightweight aggregates which were produced with only powders (Baykal and Doven, 2000). This demonstrates that the addition of BAF contributes to a higher CR. This can be comprehensively explained from two aspects: firstly, when more BAF is added to replace coal fly ash, the surface area of the BAF is lower than the powdered coal fly ash, which decreases the amount of binder needed to bond them. Hence, sample with more BAF has higher CR when the binder amount is the same. Secondly, the BAF can act as ‘aggregate’ in the pellet to support the pellet strength and the irregular shape of the BAF also increases the bonding strength between the matrix and BAF (as shown in Fig. 8), also the BAF is stronger than the matrix to support the loading. Therefore, the addition of BAF can improve the artificial aggregate strength, which is not reported before. Additionally, in this way the BAF can be used as a suitable materials for producing aggregates, instead of using as building material directly in which the BAF only has a detrimental influence (Tang et al., 2015; Yu et al., 2014; Müller and Rübner, 2006) or being disposed as waste material in landfill areas. Furthermore, the BAF particles mostly are very porous as Fig. 8(d) shows, hence the use of BAF instead of powders would not increase the bulk density of the pellets, or even lower bulk density of pellets can be achieved.

Hence, the combination of these solid wastes for producing lightweight aggregates is an efficient way to recycle them, they can be used to improve the pellet properties based on their special characteristics. In future studies, other waste fine fractions can also be added to modify the properties of the artificial aggregates, such as sludge, fine bottom ashes and recycled concrete fines.

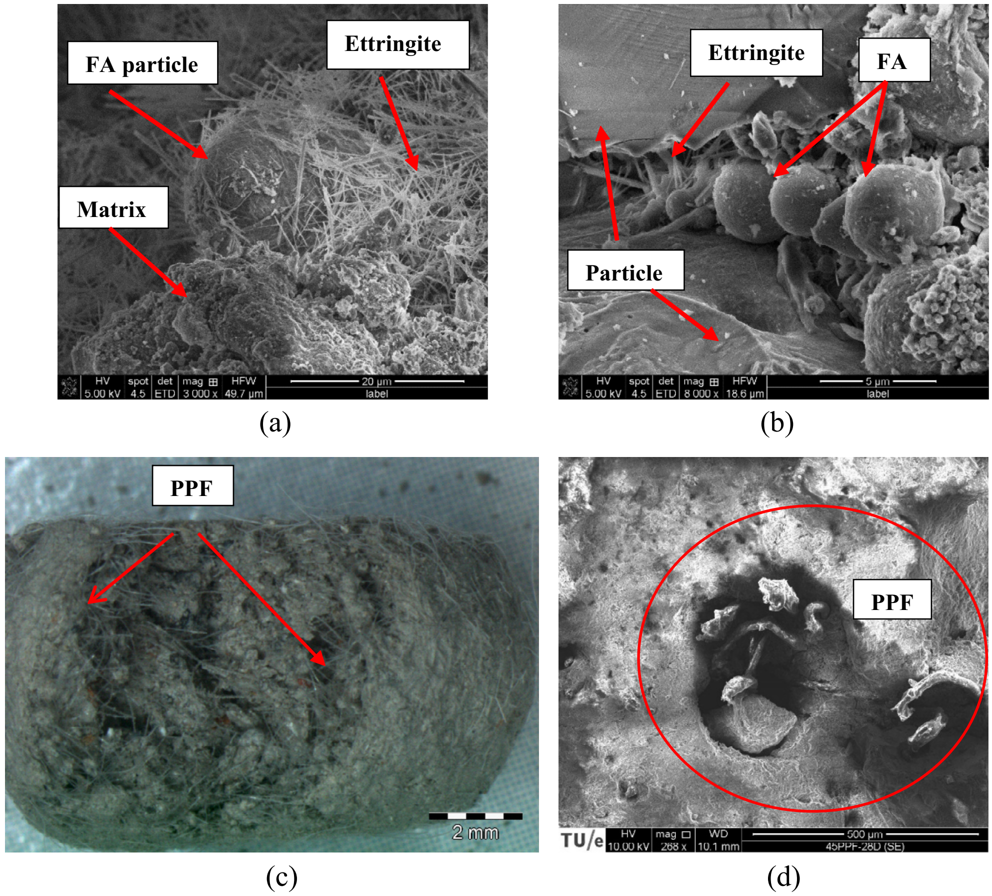
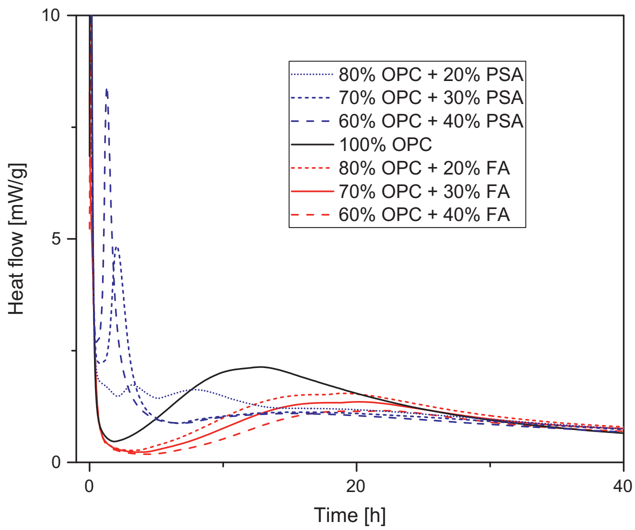



**Fig. 9.** The optical microscopy and scanning electron microscopy (SEM) graphs of the pellets (a: sample M1; b: sample 10C; c: sample 2.5% PPF; d: sample 4.5% PPF).

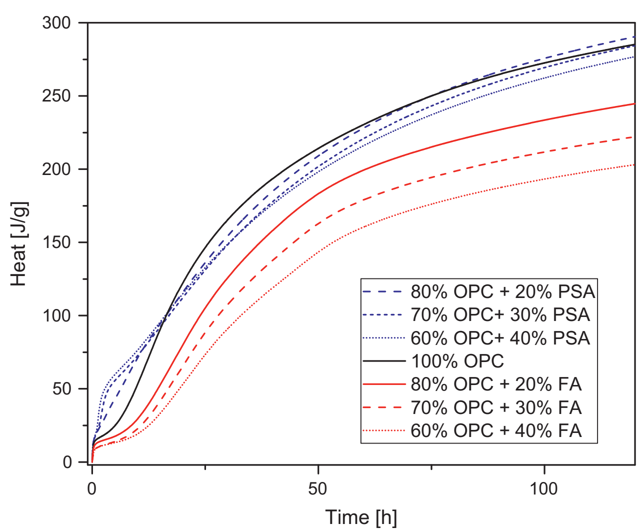
The comparison of samples 10C, 0.5–4.5% PPF demonstrates the influence of PPF on the CR. The addition of the PPF contributes to comparable CR to or even higher than 10C, while with lower bulk densities; and the more PPF is added, the higher CR can be reached. Hereby, the functions of the PPF is similar as the fibres used in the concrete (Lee and Kim, 2010; Bonakdar et al., 2013; Bernhardt et al., 2014). Under individual pellet test, the pellets without PPF are broken into two hemispheres, while the pellets with PPF were still held together after the crack almost penetrate the whole pellet (as shown in Fig. 9(c)); also the PPF embedded in the matrix goes through the voids (Fig. 9(d)), these voids in general are the weak part of the pellets. It is observed that along the crack after loading, some fibres were broken, some of them were pulled out, which means the PPF burdened the force during the loading procedure. However, the CR of samples with PPF at early stage is lower than 10C, and the higher amount of PPF the sample contains, the lower CR it will have at early age. This may attribute to the fact that these samples have high porosity and the hydration of the binders is still undergoing, which has a lower capacity to bind the fibre into the matrix. Hence, when the aggregates are under loading, most fibres are pulled out easily, which do not play a significant role as reinforcement. The CR increment of samples with 0.5%, 1.0%, 2.5% and 4.5% Vol. PPF after 91 days is about 14.4%, 30.2%, 44.4% and 16.6% compared with 10C, respectively. It can be seen that when the PPF amount is 4.5%, the CR is lower than the sample with 2.5% PPF. Firstly, when the amount of the PPF exceeds a certain level, the pellets are more difficult to compact during the pelletizing procedure which results in higher porosity of the pellets. Secondly, higher amounts of PPF need more binder to provide a good bonding strength between the PPF and the matrix. Hence, the amount of PPF should be controlled. It is worth to mention that regarding to the fibre that can be used to increase the pellet strength, also waste fibres can be considered, such as waste wood fibre and waste PPF (García et al., 2014) which cannot be used directly in the concrete and do not contribute too much to the concrete strength.

It is shown in Fig. 7 that some test values are lower than previous values, this may due to the inhomogeneity of samples and variation of testing operation. Additionally, BAF has a certain amount of metallic aluminium (around 0.4 wt.% Tang et al., 2015) which may lead to crack of some pellets when it reacts with alkalis, this could be also a reason why the pellet strength of some samples decreases. Further research is needed to investigate and quantify this influence.

In literature, there were several special methods used to increase the pellet strength, such as lime addition (Vasugi and Ramamurthy, 2014; Geetha and Ramamurthy, 2010b), milling of bottom ash fraction into powder (Cioffi et al., 2011), two-step pelletizing (Colangelo et al., 2015), and surface treatment (Gesoğlu et al., 2007). However, by milling the bottom ash into powder the cost is increased and more binder is needed to obtain similar strengths due to the increasing surface area of raw materials. In this study, the BAF is used as received without further treatment, and better results are obtained. Considering two-step pelletization and aggregate surface treatment, the increase of the pellet strength is not so substantial comparing with the binder amount added. This is due to the fact that the microstructure of the pellet which influences the pellet strength is not densified by these two methods (only the shell layer of the pellets is strengthened, but the core of the pellet is still weak), even though the binder amount is quite high (almost 14 wt.%). In the present study, the BAF and other industrial powders are mixed homogenously and the BAF is acting




(a)




(b)

**Fig. 10.** The influence of PSA and FA on cement hydration normalized to total powder content.

as 'aggregates' to support the pellet strength and this can decrease the amount of binder used and increase pellet strength, which is one of the benefits by combining the use of BAF with other wastes. Polypropylene fibre is introduced into the pelletizing process to produce cold bonded lightweight aggregates for the first time, and the aggregates produced have lower bulk density while higher crushing resistance compared with normal cold bonded lightweight aggregates.

It can be summarized that to increase the crushing strength of the cold-bonding lightweight aggregates, the binder amount, BAF addition, and applying appropriate wastes powder as additives can be considered. For decreasing the bulk density and increasing the crushing strength of the lightweight aggregates, the addition of PPF can be a choice.

### 3.3. The leaching behaviour of the produced aggregates

The leaching behaviour of the solid wastes and products containing solid wastes is of significance and has to be investigated before their application or disposal according to Dutch environmental legislation (Quality Decree, 2008). In this study, the column leaching tests were performed on the produced aggregates to study the leaching behaviour of several elements (which exceed the leaching limitation in the bottom ash used in current study (Tang et al., 2015). Moreover, according to our previous study on the monitor of MSWI BA properties for more than 7 year (Tang et al., 2015), it is found that the heavy metals and salts which always exceed their leaching limits defined in the Dutch legislation (Quality Decree, 2008) are Sb, Cu and Mo, chloride and sulphate. Hence, in this work we only focus on the leaching properties of these heavy metals and salts. The leaching results can be used as indicator for further research on combined use of solid wastes.

The amount of BAF used for aggregates production is much higher than the other materials (FA, PSA, WAS), and the total content of Sb, Cu and Mo in BAF is almost 2–10 times of that in the industrial powders in this study (as shown in the following part of this section). The leaching of the heavy metals in aggregates is supposed to be mainly influenced by the BAF properties. Hence, the leaching of the BAF is tested only. The municipal solid waste incineration (MSWI) bottom ash fines (0–2 mm, BAF) in this study has leaching concentrations of Sb, Cu, Mo, chloride and sulphate of 0.47 mg/kg d.m., 1.1 mg/kg d.m., 0.68 mg/kg d.m., 950 mg/kg d.m. and 11,000 mg/kg d.m., of which the limit leaching value according to Dutch legislation (Quality Decree, 2008) is 0.32 mg/kg d.m., 0.9 mg/kg d.m., 1.0 mg/kg d.m., 616 mg/kg d.m. and 2430 mg/kg d.m., respectively. Fig. 11 shows the leaching concentration of antimony (Sb), copper (Cu), molybdenum (Mo), chloride ($Cl^-$) and sulphate ($SO_4^{2-}$) of the aggregate samples after 28 days and 91 days curing.

#### 3.3.1. The leaching behaviour of antimony (Sb)

Fig. 11(a) shows that the Sb leaching concentrations of all the samples are well under the limit value (0.32 mg/kg d.m.) according to legislation (Quality Decree, 2008). Sample M1 has the highest leaching concentration of Sb among all the samples, it should be mentioned that the total amount of Sb in BAF and FA (about 110–190 mg/kg d.m.) is 2–4 times higher than that in the PSA and WAS (around 42–52 mg/kg d.m.). Hence, the change of BAF and FA proportions will have significant influence on the leaching of Sb from the aggregates samples.

Comparing samples 5C, 10C and 15C, the leaching concentrations of Sb are decreasing with the increase of the binder amount. The Sb leaching of sample 15C decreased by around 23% compared with sample 5C, which indicates higher amount of binder benefit the immobilization of Sb in the aggregates. In general, the immobilization of the contaminants is positively related to the binder dose, which produce more hydrates to absorb the contaminants on the surface, or provide precipitation surface, or for the ion-exchange during the hydration process (Batchelor, 2006). In this study, as shown in Fig. 11, the leaching of heavy metals (Sb, Mo and Cu), chloride and sulphate mostly decreases with the cement amount. Sample 6C has similar leaching concentration after 28 and 91 days curing and it has lower leaching of Sb after 91 days compared with sample 10C when the OPC amount is 40% less. It is reported that ground granulated blast furnace slag (GGBS) has an excellent immobilization capacity on contaminants (Müllauer et al., 2015; Laforest and Duchesne, 2005). It is also stated that the addition of GGBS contributes to lower hydration speed, less portlandite and generation of C—S—H with lower Ca/Si ratio compared with OPC, which results in lower pH and maybe a negative charge surface of calcium silicate hydrate (C—S—H). Eventually, the solubility of the metal-bearing phases is lower at resulted pH and they are more favourable to be situated on the surface of the C—S—H. Hence, the samples with GGBS has a lower pH (Fig. 11 (f)) and has lower leaching of contaminates as shown in this study compared with 10C, even though it has lower crushing resistance. Sample 10C has a higher leaching of Sb than sample No PSA after

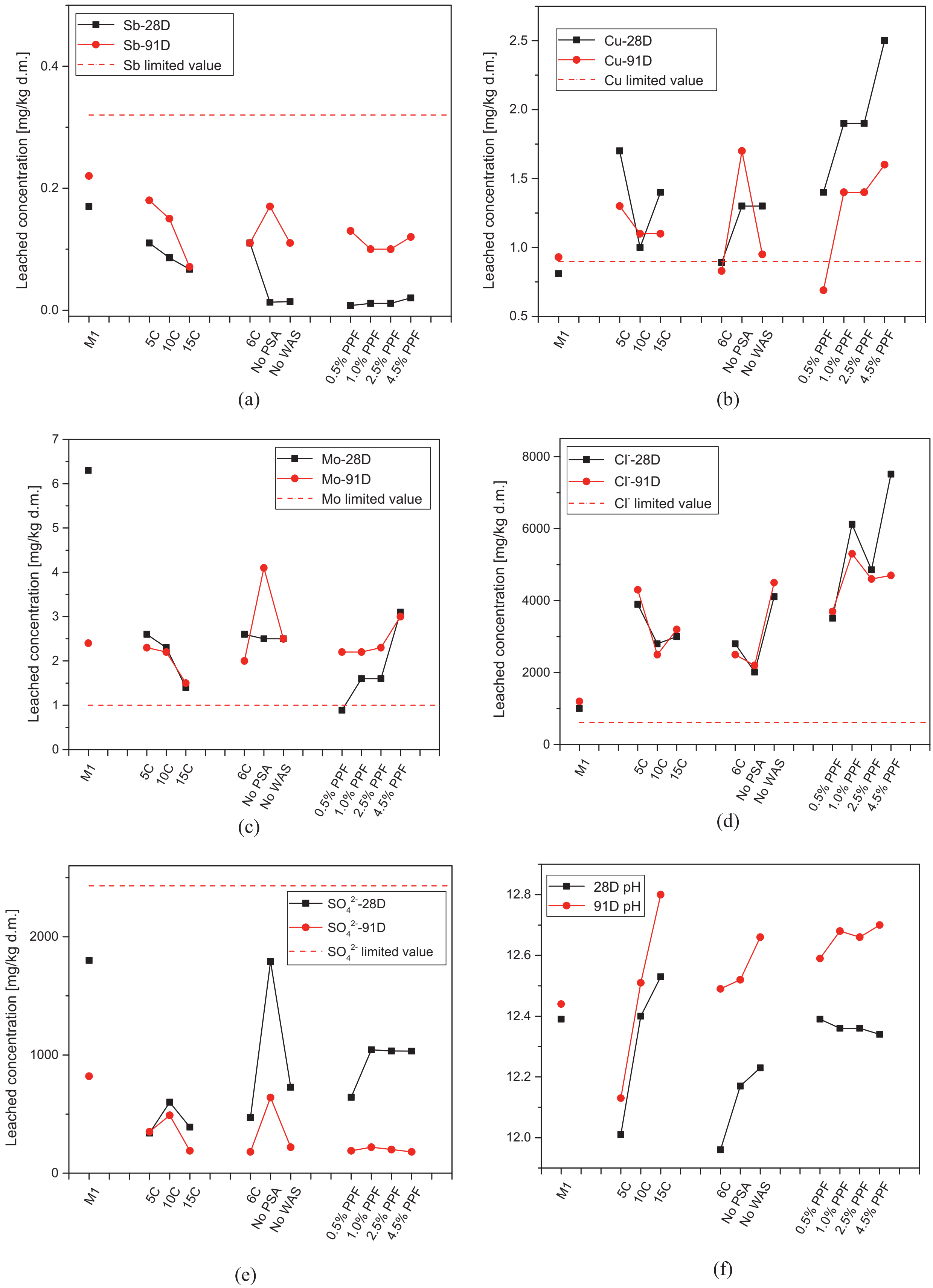



**Fig. 11.** The leaching concentration of Sb (a), Cu (b), Mo (c), chloride (d) and sulphate (e), and the pH (f) of all the aggregates samples (Table 2).

28 days curing, while it is lower than sample No PSA after 91 days curing. This can be explained that the addition of paper sludge ash (PSA) provides a calcium oxide resource which has a significant immobilization influence on Sb as reported (Van Caneghem et al., 2016). Moreover, the PSA contains calcium silicate which contributes to more hydrates for immobilization. Sample No WAS has a similar Sb leaching to sample 10C and the Sb leaching of samples with PPF did not show significant influence considering the amount of PPF added.

It is also noticed that the Sb leaching concentrations of samples after 91 days curing is higher than the samples which are cured for 28 days, and the pH of the samples has similar trend (Fig. 11(f)). Similar Sb leaching behaviour is reported by Cornelis et al. (2012). The cement hydrates can immobilize the Sb ions and then reduce its leaching, however, the Sb leaching of the BAF is influenced by pH (Cornelis et al., 2012) and the leaching of the Sb from BAF or FA can happen during the whole hydration procedure. Simultaneously, the released Sb ions are immobilized by the cement hydrates. Hence, it can be assumed that the hydration rate of binders after longer time curing is much slower than the leaching of the Sb from the waste solids, which attribute to the higher leaching of Sb after 91 days curing.

For Sb leaching in the produced aggregates in the present work, the addition of additives with calcium oxide which can contribute to hydrates, decreasing amounts of FA and BAF, addition of GGBS and increased binder amounts, will decrease the leaching concentration of produced aggregates in case the leaching of Sb exceed the limit value.

#### 3.3.2. The leaching behaviour of copper (Cu)

The total amount of Cu in the samples is strongly depends on the content of BAF (2300 mg/kg d.m.) used which is 5–10 times of the other solids (22–450 mg/kg d.m.) used in this study. Therefore, sample M1 which contains the lowest amount of BAF has the lowest leaching of Cu content among all the samples. Most likely, the leaching of copper decreases with the curing age (Fig. 11(b)).

The leaching of copper mostly exceeds the limit value, while sample M1 and 6C are very close to the limit value. Samples 5C, 10C and 15C after 91 days curing show that the leaching of copper has a roughly decreasing trend when the amount of binder increases and sample No PSA has a higher Cu leaching than sample 10C indicating the contribution of PSA on the Cu immobilization. According to Van Zomeren and Comans (2009), the leaching of copper in BAF is related to its dissolved organic carbon content and others' research shows that the copper leaching has an increasing trend when the pH is higher than 10 (Keulen et al., 2015). Therefore, during the cement hydration the leaching of the copper from BAF will be increased due to the high pH environment, meanwhile the Cu ions are bound by the cement hydrates, as also concluded by Li et al. (2001), the leaching of heavy metals in cement based system is controlled by pH and the solubility of the related metal hydroxides. This explains the low Cu leaching of sample 6C, which has lower pH and slower hydration speed compared with sample 10C. However, compared with the Sb leaching, the leaching of Cu did not show a clear trend related to the curing time.

The Cu leaching of samples with PPF shows an increasing trend when the PPF content is increased, and the leaching of Cu after 91 day is decreased compare with samples after 28 days curing. This might be attributed to the high porosity of pellets with PPF, which increases the ability of water to contract with cement hydrated during the leaching process, eventually more hydrates can be dissolved and release Cu ions.

Hence, decreasing the BAF amount, increasing the binder content and adding GGBS can lower the Cu leaching of the produced aggregates when the Cu leaching concentration is required during application.

#### 3.3.3. The leaching behaviour of molybdenum (Mo)

The total amount of Mo in FA is amount 2 times of that in BAF (15–28 mg/kg d.m.) and the amount of Mo in other solid wasted used in this study is around 1.0–5 mg/kg d.m., hence the leaching of Mo is contributed mostly by both FA and BAF amount in the samples. It can be seen that the Mo leaching of the samples mostly exceed the limit value, which is 1.0 mg/kg d.m. and there is no clear relation between Mo leaching and pH (Santos et al., 2013).

Samples 5C, 10C, and 15C has a decreasing leaching amount of Mo when the binder amount is increased. The replacement of cement by GGBS slightly decreased the Mo leachability when comparing sample 6C to 10C. Sample No PSA has the highest Mo leaching after 91 days and the Mo leaching concentration decreases when PSA is added (sample 10C). The sample with PPF has a lower leaching of Mo after 28 days compared with sample 10C, the leaching concentration is increasing with the PPF addition. While after 91 days curing the leaching of the Mo is similar to sample 10C and the samples with PPF have a very close leaching concentration of Mo regarding to the amount of PPF.

From the obtained leaching results, it can be seen that increasing the binder amount can reduce the Mo leaching significantly.

#### 3.3.4. The leaching behaviour of chloride ($Cl^-$)

It can be seen that the leaching of chloride exceeds the limit value and that is not significantly influenced by the curing time (Fig. 11(d)). The XRF results (Table 1) shown that the chloride content in the aggregates is mainly influenced by BAF and PSA amounts.

Sample M1 has the lowest chloride leaching due to the fact that the total chloride in this sample is mainly influences by the amount of the BAF, which exceeds the leaching limit slightly. Sample No PSA has a higher chloride leaching when the BAF amount is increased by 25%. The leaching of chloride decreased with the increasing amount of OPC, sample 6C features a lower chloride leaching than 10C, and very similar to sample 15C. Samples with PPF has an increasing chloride trend with the increase of PPF amount which is explained due to the high porosity of the pellets.

It follows that the chloride leaching after longer time curing did not shown big difference, which indicates that the immobilization of the chloride by hydration is limited and the chloride in the aggregates is highly soluble. Hence, the immobilization of chloride cannot be easily achieved by increasing the hydrates. Moreover, the amount of the produced aggregates should be controlled when the chloride leaching is restricted during the application.

#### 3.3.5. The leaching behaviour of sulphate ($SO_4^{2-}$)

The leaching of sulphate for all the samples decreased after longer time curing and is well under the limit. Samples 5C, 10C and 15C show that the leaching of sulphate is increased when binder is increasing, while when binder amount is 10C, the leaching of sulphate is higher than 5C and 15C. Sample 6C shows a lower leaching of sulphate than sample 10C, and similar to sample 15C. Sample No PSA and M1 have much higher amounts of sulphate leaching than other samples compared with sample 10C. The leaching of sulphate is not influenced by the amount of PPF. In the cement based system used here, the sulphate is mainly provided from OPC and BAF, which participates the hydration to form AFt/AFm (Tang et al., 2016), hence its leaching is significantly reduced along with the hydration time of cement.

To summarize shortly, the leaching of Cu, Mo and chloride of most of the aggregates produced, based on the current recipes exceed their leaching limit according to the Dutch legislation (Quality Decree, 2008). However, when they are used in concrete, the leaching of the concrete in general will comply with the legislation. In this case, the concrete with the addition of these aggregates can be deployed as a final product in market.

#### *3.3.6. Mechanism of the immobilization on the contaminants in produced aggregates*

It is summarized that the immobilization of contaminants by cementitious materials is mainly related to several parameters: the pH, the hydration products, the properties of the contaminants (stability, complexation, etc.), etc. (Batchelor, 2006). For the aggregates produced by BAF and industrial wastes in this study, the Sb can be immobilized successfully by increasing the binder amount, GGBS and PSA addition, which all results in more hydration by-products. The leaching of Cu is mainly influence by the behaviour of organic matter in BAF, which can be immobilized by binder addition. The binding of Mo shows a good correlation with binder content and its hydration. Sulphate leaching are well under the limit value owing to its participation of cement hydration. The chloride in the aggregates is highly soluble, and can partially be immobilized. Furthermore, the immobilization procedure in the pellets is a combination of ions leaching from the solid wastes and their stabilization by cement hydrates. Therefore, to modify the leaching of ions, especially heavy metals, their leaching behaviour at high pH, and the stabilization mechanism by cement is suggested to be determined individually before the combination of solid wastes.

In summary, the recycling of different industrial solid wastes is combined by using a pelletizing technique, and they have a good compatibility to be used for producing cold-bonding artificial lightweight aggregates. The aggregates properties, especially crushing resistance, which influence their application, are improved by both chemical (increasing binder amount) and physical methods (PPF addition). The leaching concentration of some contaminants (Mo and chloride), which exceed the limited values according to Dutch legislation (Quality Decree, 2008), can be reduced by adjusting the proportions of the raw materials. Moreover, these aggregates can be used in shaped materials (Quality Decree, 2008), such as concrete blocks, the leaching of which will be reduced significantly and will comply with the environmental legislation. Furthermore, the application of the aggregates can be adjusted according to the mechanical (crushing resistance, density), economic (binder amount, wastes proportions) and environmental (leaching properties) point of views.

Further research can be performed on the recycling of other solid wastes, such as recycled concrete fines, various industrial sludge and waste fibres. In addition, the durability and life cycle of these aggregates in concrete should still be studied as well.

## 4. Conclusions

- The cold bonded pelletizing technique can be used to recycle the municipal solid waste (MSWI) bottom ash fines (0–2 mm, BAF) and industrial powder wastes (coal fly ash, paper sludge ash and washing aggregate sludge). The BAF and industrial powder wastes have good compatibility during the pelletizing process, a type of cold bonded artificial lightweight aggregates is produced.
- To improve the properties of the cold bonded lightweight aggregates, several methods are investigated. It is concluded that the increase of binder amount results in the increase of crushing resistance (CR) and bulk density of the aggregates; the addition of paper sludge ash increases the CR due to its contribution to cement hydration; the addition of BAF increase the CR which attributes to the fact that BAF can act as 'aggregate' in the pellet to strengthen the skeleton of the pellets, as well as providing a smaller surface area; the addition of polypropylene fibre (PPF) increases the CR dramatically, and the amount of fibre should be controlled to have a maximum CR increase.
- For the produced lightweight aggregates, the leaching of the Sb and sulphate are all well under the limit value due to the immobilization of binder hydrates. The increasing of binder amount reduces the leaching of Sb, Cu, Mo, chloride and sulphate.
- The leaching of Cu is significantly influenced by the BAF amount, and the increase of binder amount and addition of GGBS can decrease the leaching of Cu. The leaching of Mo is mainly influenced by the amount of coal fly ash (FA) and BAF, and the increasing binder amount can decrease the Mo leaching slightly. The leaching of chloride is significantly influenced by the amount of paper sludge ash (PSA) and BAF. The addition of PSA and increase of binder amount decrease the leaching of chloride and the reduction of chloride leaching is not improved significantly by increase binder content and curing age.
- The produced cold-bonding lightweight aggregates can be used as aggregate replacement, and the amount of these aggregates can be controlled when the leaching properties of the concrete is specified. Further research can be focused on the integrated recycling of other wastes, durability and life cycle of the produced artificial aggregates.

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