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# Re-cementation effects by carbonation and the pozzolanic reaction on LWAs produced by hydrated cement paste powder

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# ABSTRACT

This paper aims to evaluate lightweight aggregates produced with hydrated cement paste powder (HCP) using both carbonation and normal curing. The comparative study was conducted to figure out the effects of the portlandite amount in HCP I and HCP III on the aggregates during carbonation curing. The addition of up to 10 wt% silica fume to HCP I was attempted to make full use of the considerable amount of portlandite to prepare artificial aggregates under normal curing. The mechanical properties, reaction products and microstructure were analysed and the results show that the optimal carbonation periods for HCP I - and HCP III-type aggregates are different due to different amount of portlandite. HCP I-type aggregates can gain 3.14 MPa after 7-day carbonation and contain 35.60 wt% calcium carbonates. The remaining 13.95 wt% portlandite shows the enormous potential in elevating the strength and CO<sub>2</sub> capture capacity jointly via the optimized carbonation curing method. On the contrary, HCP III-type aggregates gained 2.97 MPa after 1-day carbonation and further carbonation decomposed C–S–H and lead to the formation of calcite and amorphous silica gel with significantly elevated specific surface area (from 10.69 m<sup>2</sup>/g to 42.96 m<sup>2</sup>/g). Additionally, the individual strength development of the prepared aggregates containing silica fume benefits from the sufficiently available portlandite due to the formation of secondary C–S–H, obtaining 2.39 MPa after 28-day normal curing.

## 1. Introduction

As a newly developed building material, lightweight concrete shows some extraordinary properties such as extremely low density, excellent sound absorption, and thermal insulation (Chandra and Berntsson, 2002). The main ingredients for a lightweight concrete design are cement and lightweight aggregates (LWAs) (Chen et al., 2020; Kan and Demirboğa, 2009; Spiesz et al., 2013; Yu et al., 2013). Generally, LWAs have a porous structure and low bulk density (less than 1200 kg/m<sup>3</sup>) and thus reduce the density of concrete structure effectively (Lo et al., 2007). However, the commercial LWAs, for instance, expanded clay, expanded glass and perlite, consume huge amounts of energy, as well as natural resource due to the production at high temperatures (700 °C–1400 °C) (Tajra et al., 2019). Consequently, some studies started focusing on the development of a low-carbon footprint and sustainable LWAs.

Cold-bonding technology has been proposed and developed for the

purpose of low energy consumption during LWAs manufacture (Tang et al., 2020; Tang and Brouwers, 2017, 2018). Combined with a pelletizing process, pellets in different sizes with porous structure were produced and then applied as LWAs (Yıldırım and Özturan, 2013). Some industrial solid wastes can be utilized in LWAs as well, for examples, bottom ash, concrete waste powder, and fly ash (Gesoğlu et al., 2012; Günevisi et al., 2015; Jiang et al., 2020; Shi et al., 2019; Tang et al., 2017). Since these solid wastes show a low or non-hydraulic reactivity, the incorporation of cement, additional curing strategies, or chemical additives in cold-bonded LWAs are commonly used to form extra hydration products and optimize pore structure in order to achieve adequate mechanical performance. Steam curing was applied to improve the properties of cold-bonded LWAs produced with cement waste slurry and bottom ash (Yıldırım and Özturan, 2013). Portlandite and alkali solution were also used to promote the reaction in blast furnace slag or fly ash-based LWAs (Geetha and Ramamurthy, 2013;

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Vasugi and Ramamurthy, 2014). However, the use of additional binders, alkali activators or other curing regimes can result in long curing period, high costs, high CO<sub>2</sub> emission and safety risks.

On the other hand, the recycling treatment of the aged concrete structure can recover most of the original wastes as high quality fine and coarse recycle concrete aggregates and thus leave 10%–20% of fine concrete waste powder (Lu et al., 2018). The main components in waste powder are hydrated cement paste (HCP) but the exact composition can depend on the crushing and heating methods used to recycle concrete (He et al., 2020; Serpell and Lopez, 2013; van de Wouw et al., 2020; Yang et al., 2019). The waste powder can have a high water demand due to the porous structure and high surface area, along with a very low reactivity when used as a supplementary cementitious materials (SCMs) (He et al., 2020; Shi et al., 2016a, 2016b), causing the difficulty in applying to normal concrete production without further treatment or activation (Kan and Demirboğa, 2009; Spiesz et al., 2013; Yu et al., 2013).

The HCP contains mainly calcium silicate hydrate (C-S-H) gels, calcium hydroxide, AFt, AFm and some unreacted clinker phases. Gassolid and aqueous carbonation were reported to be effective to utilize HCP powders as the SCMs (Jiang et al., 2022). The workability and mechanical property of cement paste blended with carbonated HCP powder can be enhanced via the formation of calcium carbonates which can act as fillers and then optimize the pore structure (Mehdizadeh et al., 2021, 2022). Carbonation can also lead to the formation of silica gel with pozzolanic properties due to the decomposition of calcium silicate phases (Silva et al., 2015; Zhan et al., 2019; Zhang et al., 2022). The reaction between portlandite and silica gel, has been widely applied to improve the bonding performance of recycled concrete aggregate in new concrete due to the formation of secondary C-S-H gel (Zanni et al., 1996). Furthermore, these re-cementation effects may make it possible to produce HCP powder based cold-bonded LWAs without cement and alkali activator addition, by using carbonation or the addition of silica fume only. The current research on the effects of carbonation and pozzolanic reactions on mechanical and microstructure development of cold-bonded HCP powder-based LWAs is still limited and needs to be further investigated.

For the purpose of investigating the mechanism of the mechanical and microstructure development of the artificial aggregates under carbonation or pozzolanic reaction, this study used the laboratory-made cement paste instead of a real demolished concrete to obtain HCP powder and then produce LWAs, which allows to better control the chemical composition of the waste powder by eliminating the variability of its properties when recovered from demolished materials. Additionally, researchers preferred to investigate the properties of HCP powder from CEM I which contains few SCMs, whereas CEM III is also widely used and deserves more attention in the production of LWAs. Therefore, both CEM I 52.5 R and CEM III/A 52.5 N were chosen as the starting materials. The amount of hydration products from CEM I and CEM III is different, especially the amount of portlandite. Therefor a comparative study has been conducted to figure out the effects of portlandite amount in HCP I and HCP III on the aggregates production under carbonation curing. The addition of up to 10 wt% silica fume into HCP I was also attempted to make full use of the considerable amount of portlandite to prepare artificial aggregates and evaluate the pozzolanic property under normal curing. The mechanical properties, reaction products and microstructure were investigated via XRD, FTIR and etc. to figure out the re-cementation effects of carbonation and pozzolanic reaction on LWAs produced with HCP powder. The results provide a low carbon footprint and sustainable method of HCP powder application and LWAs production.

## 2. Experiments and methodology

#### 2.1. Raw materials

Silica fume (SF) was used as a part of binder to produce aggregates, which was provided by Sika. The HCP powder was produced in the laboratory from the cement paste which was prepared by CEM I 52.5 R and CEM III/A 52.5 N (provided by ENCI, the Netherlands). The used CEM I 52.5 R and CEM III/A 52.5 N in this study contain 0 wt% and 36-65 wt% GGBFS according to European standard EN 197-1, respectively. All cement pastes were prepared with a water to cement ratio of 0.5 and then demolded 24 h after mixing, followed by the ambient curing in resealable bags for at least 3 months. The HCP was crushed manually and sieved to a size of below 5 mm. The sieved material was then oven dried at 105 °C until the weight was constant. A disc milling (Retsch, RS300XL) for 10 min was applied to obtain the waste cement paste powder for the tests. The milled HCP powder originating from CEM I and CEM III was labelled as HCP I and HCP III, respectively. The chemical composition of SF, HCP I and HCP III was analysed with X-ray fluorescence (XRF, Model Axios Advanced, PANalytical.B.V), as shown in Table 1. The mineral composition of HCP I and HCP III was determined using X-ray diffraction (XRD) analysis, which is shown in Fig. 1 (a) and (b), respectively. The particle size distribution of SF, HCP I and HCP III analysed with a laser particle size analyser (Model Malvern Mastersizer, 2000; Malvern PANalytical) is presented in Fig. 2.

#### 2.2. Pelletizing procedure

The disc pelletizer was used to produce the artificial aggregates. The model was D-7736, Maschinenfabrik Gustav Eirich, Germany. The size of the pelletizer was 40 cm in diameter and 10 cm in collar height. The vertical angle of the pan is  $75^{\circ}$  and the rotating speed was set as 60 rpm during the production process. 500 g of HCP were placed on the rotating pan. After about 3 min of rotating, 50 g of distilled water was sprayed slowly onto the mixed powder in the pan using a spray bottle over 10 min. After the aggregation was observed, the pan continued running for 5 min to form the pellets. The as-prepared aggregates fell out of the pan automatically once they reached sufficient size and were collected. The next batch was made with the addition of another 500 g of dry powder and followed the same procedure again. The freshly prepared LWAs were placed in a CO<sub>2</sub> chamber with a CO<sub>2</sub> concentration of 5% and a relative humidity of 75% and carbonated for 1, 3 and 7 days, respectively for carbonation curing. The temperature during curing was constant, around 20 °C. The prepared aggregates, which were originated from HCP I and HCP III, carbonated for 1, 3 and 7 days were labelled as C1A I, C3A I, C7A I, C1A III, C3A III and C7A III, respectively, as shown in Table 2.

To obtain aggregates containing SF, the mixture of SF and HCP I was first mixed via a Planetary mixer. Then around 500 g of mixed powder were placed on the rotating pan. The following pelletizing procedure was the same as mentioned above. The obtained fresh aggregates were then placed in sealed plastic bags for 28 days until further testing. The aggregates containing SF were labelled as SOA I, S5A 1 and S10A I, respectively, according to the different mix proportion that SF replaced partial HCP I with mass ratio of 0%, 5% and 10%, respectively, as shown in Table 2.

#### 2.3. Properties of prepared LWAs

## 2.3.1. Water absorption and loose bulk density

After curing, the prepared aggregates were dried in an oven at 105 °C until a constant mass. The loose bulk density test was carried out according to EN 1097-3. The water absorption test was carried out according to EN 1097-6.

#### Table 1

The chemical composition of raw materials for preparation of aggregates (silica fume (SF), milled hydrated cement paste powder I (HCP I) and milled hydrated cement paste powder III (HCP III)).

Materials (wt.%)	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	$SO_3$	MgO	MnO	TiO <sub>2</sub>	Others
SF	0.90	93.06	-	2.06	1.28	0.70	0.07	_	1.93
HCP I	65.96	17.42	7.57	3.51	3.08	1.43	0.08	0.44	0.51
HCP III	49.32	27.10	12.02	1.24	4.83	4.24	0.17	0.55	0.53



Fig. 1. XRD patterns of HCP I (a) and HCP III (b) (P = portlandite, A = alite, B = belite, C = calcite, Hc = hemicarbonate, D = dolomite, C-S-H = calcium silicate hydrates).



Fig. 2. Particle size distribution of silica fume (SF), milled hydrated cement paste powder I (HCP I) and milled hydrated cement paste powder III (HCP III).

#### 2.3.2. Mechanical property

The mechanical properties of the individual pellets with different diameters were tested in an MTS Criterion equipped with a load cell of 100 kN at a speed of 0.6 mm/min until collapse. The maximum compression load was used to calculate the individual crushing strength with Eq. (1) (Tang et al., 2019). 20 pellets were chosen as representatives for each group of prepared LWAs.

$$\sigma = 2.8P/\pi h^2 \tag{1}$$

where  $\sigma$  (MPa) is the crushing strength of each pellets tested, *P* (N) is the maximum compression load each pellet can withstand, *h* (mm) is the diameter of the round pellet produced.

 Table 2

 Mix proportion of aggregates prepared from different curing regimes.

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Curing regime	Sample	HCP I/ g	HCP III/g	SF/ g	Water/ g	Curing time/ day
CO <sub>2</sub> curing	C1A I	500	-	-	50	1
	C3A I	500	-	-	50	3
	C7A I	500	-	-	50	7
	C1A III	-	500	-	50	1
	C3A III	-	500	-	50	3
	C7A III	-	500	-	50	7
Normal	SOA I	500	_	-	50	28
curing	S5A I	475	-	25	50	28
	S10A I	450	-	50	50	28

# 2.3.3. Characterization of reaction products and microstructure

Different artificial aggregates were crushed manually and ground finely to pass a sieve of 68  $\mu$ m. Afterwards, samples were immersed in isopropanol for 30 min to eliminate hydration according to literature (Scrivener et al., 2018) and then dried in an oven at 40 °C until a constant mass. All samples were stored in desiccators, using a drying agent (CaCl<sub>2</sub> pellets) and sodium hydroxide pellets as a CO<sub>2</sub> trap until further test.

The crystalline phases of the prepared aggregates were detected by X-ray diffraction (XRD, Bruker D2 PHASER) analysis. The parameters chosen were as follows: Co tube, 40 KV, 30 mA,  $0.02^{\circ}$ /step,  $0.2^{\circ}$ /min, with variable divergence slits V20.

Fourier transform infrared spectroscopy (FTIR) test was conducted by using a Varian 3100 FTIR Spectrometer. All spectra were obtained with 30 scans per spectrum and a resolution of  $1 \text{ cm}^{-1}$ .

The microstructure was observed with scanning electron microscopy (SEM), by using a JOEL JSM-5600 instrument at an accelerating voltage of 15 kV.

Nitrogen sorption analysis was performed using a Brunauer-Emmett-Teller (BET) specific surface area and porosity instrument (TriStar II 3020, Micrometrics). The surface area was calculated with the Brunauer-Emmett-Teller method (Joyner et al., 1951) using the adsorption branch. The pore size distribution was determined with the Barrett–Joyner–Hallenda method (Brunauer et al., 1938) from the adsorption branch.

Thermogravimetric analyses (TGA) were conducted using a NETZSCH STA 449 F1. Ground powder samples (30–40 mg) were prepared, and a heating rate of 10 °C/min from 40 to 1000 °C in nitrogen atmosphere was used. The temperatures selected for the quantification of Ca(OH)<sub>2</sub>, CaCO<sub>3</sub> and chemically bound water were determined with derived thermogravimetric analysis (DTG), and are 400–500 °C, 600–800 °C and 75–550 °C respectively according to the literature (Liu et al., 2021; Scrivener et al., 2018; Zhu et al., 2020). The weight loss between 75 and 550 °C is equivalent to the amount of chemically bound water of C–S–H, ettringite, AFm phases, portlandite and etc. Therefore, Eq. (2) and Eq. (3) were used to quantify the amount of chemically bound water excluding portlandite that was quantified separately using Eq. (2) in the prepared samples before and after curing under different conditions (Zajac et al., 2020).

$$W_{Ca(OH)_2} = \frac{Water \ loss_{OH \ group \ in \ Ca(OH)_2}}{M_{H_2O} \times M_{800^{\circ}C}} \times M_{Ca(OH)_2} \times 100\%$$
(2)

Where *Water* loss<sub>OH group</sub> in  $_{Ca(OH)_2}$  corresponds to the weight loss from approximately 400 to 500 °C in the total mass fraction, M<sub>800°C</sub> is the weight loss at 800 °C, M<sub>H<sub>2</sub>O</sub> is molar mass of the water, equal to 18.02 g/ mol, M<sub>Ca(OH)<sub>2</sub></sub> is the molar mass of Ca(OH)<sub>2</sub>, equal to 74.09 g/mol.

$$W_{CaCO_3} = \frac{Mass \ loss_{CO_3} \ group \ in \ CaCO_3}{M_{CO_2} \times M_{800^\circ C}} \times M_{CaCO_3} \times 100\%$$
(3)

Where *Mass loss*<sub>CO3</sub> group in CaCO3</sub> corresponds to the weight loss from approximately 600 to 800 °C in the total mass fraction,  $M_{800^{\circ}C}$  is the weight loss at 800 °C,  $M_{CO2}$  is molar mass of CO2, equal to 44.01 g/mol,  $M_{CaCO3}$  is the molar mass of CaCO3, equal to 100.09 g/mol.

$$W_{Chemically bound water} = \frac{Water \ loss_{75-550 \ \circ C} - Water \ loss_{OH \ group \ in \ Ca(OH)_2}}{M_{800^{\circ}C}} \times 100\%$$
(4)

Where *Water* loss<sub>75-550 °C</sub> corresponds to the weight loss from approximately 75 to 550 °C in the total mass fraction,  $M_{800°C}$  is the weight loss at 800 °C, *Water* loss<sub>OH</sub> group in  $Ca(OH)_2$  corresponds to the weight loss approximately from 400 to 500 °C in the total mass fraction.

## 3. Results

#### 3.1. Loose bulk density and water absorption of the produced LWAs

The loose bulk density and water absorption are essential parameters for evaluating cold-bonding lightweight aggregates and have a significant influence on their application. Both parameters are shown in Table 3. It can be seen that all the samples have a loose bulk density of around 576.0–753.6 kg/m<sup>3</sup>, which is below 1200 kg/m<sup>3</sup> and thus satisfies the density requirement of lightweight aggregates according to EN 13055-1 (2002). The bulk density is highly related to the total mass of

Table 3				
Physical	properties	of the	produced	LWAs.

Curing regime	Sample	Bulk density (kg/m <sup>3</sup> )	Water absorption (%)	Average individual strength (MPa)
CO <sub>2</sub> curing	C1A I	$614.6\pm2.0$	$33.6\pm0.6$	$1.60\pm0.32$
	C3A I	$668.6\pm3.0$	$\textbf{32.2} \pm \textbf{0.4}$	$1.95\pm0.82$
	C7A I	$\textbf{738.4} \pm \textbf{1.8}$	$29.8 \pm 0.4$	$3.14\pm0.78$
	C1A III	$753.6\pm4.5$	$25.9\pm0.5$	$\textbf{2.97} \pm \textbf{0.98}$
	C3A III	$\textbf{715.8} \pm \textbf{3.6}$	$\textbf{26.3} \pm \textbf{0.8}$	$2.03\pm0.66$
	C7A III	$605.3 \pm 2.2$	$\textbf{30.2} \pm \textbf{0.1}$	$0.57 \pm 0.22$
Normal	SOA I	$\textbf{576.0} \pm \textbf{2.6}$	$39.5 \pm 0.2$	$0.83\pm0.16$
curing	S5A I	$637.5 \pm 1.2$	$33.7 \pm 0.2$	$2.39\pm0.60$
	S10A I	$637.7 \pm 2.0$	$\textbf{30.7} \pm \textbf{0.1}$	$2.22\pm0.31$

the carbonated aggregates, which was determined by the CO<sub>2</sub> uptake and water evaporation after drying. The carbonated samples show an increasing bulk density with increasing CO2 curing duration in the samples containing HCP I, indicating that more carbonation products were formed with increased CO<sub>2</sub> curing duration where portlandite was the main carbonated subject to form calcium carbonates to elevate total mass directly (Savija and Luković, 2016; Shi et al., 2019). However, the HCP III group shows the opposite trend because portlandite was consumed after 1-day carbonation and the longer carbonation resulted in the decomposition of C-S-H gel and other hydration products, producing silica or aluminium gel containing abundant evaporation water (Liang et al., 2020; Šavija and Luković, 2016). It can reduce the total mass during the drying process before bulk density test and cause more internal cracks and pores, then significantly decreasing the bulk density of the aggregates originated from HCP III after 7-day carbonation (Sisomphon and Franke, 2007). The density of the normal cured samples increases with the addition of SF, which can help promote the formation of C–S–H and modify the microstructure of the aggregates.

Additionally, LWAs produced with HCP I and HCP III show the different trends in the water absorption, which is related to the various microstructure evolution due to carbonation. It is discussed further in the following sections. The formation of various carbonation products fills the pores inside the artificial aggregates if the carbonation level is appropriate, effectively lowering the internal porosity (Shi et al., 2019; Yu et al., 1999). The water absorption of the studied artificial aggregates is higher than that of the other reported artificial lightweight aggregates, which is commonly around 15% (Shi et al., 2019; Tang et al., 2019). It might be attributed to not only the water absorption by the porosity but also water consumption by the rehydration of the unhydrated phases and reformation of ettringite in the 105 °C-dried aggregates (Chen et al., 2020; Taylor, 1997).

## 3.2. Mechanical properties

Fig. 3 shows the maximum compression load of each produced aggregate with different diameters. The results illustrated in Fig. 3 (a), (b) and (c) show the obvious trend that the compression load increases with the increasing diameter of the aggregates for both the CO<sub>2</sub> cured and normal cured samples, in agreement with the results reported in (Gesoğlu et al., 2013; Güneyisi et al., 2013). Table 3 shows the average individual strength of all the produced aggregates and Fig. 4 shows the strength of each individual aggregate. The average individual strength of the aggregates produced with HCP I increases with the increasing carbonation period while the aggregates produced with HCP III shows the opposite trend, which is illustrated in Fig. 4 (a) and Fig. 4 (b). The carbonation curing can contribute to the formation of calcium carbonates and then fill some pores to reduce the overall porosity and enhance the mechanical property. However, the aggregates obtained from HCP III may suffer carbonation shrinkage and internal cracking during carbonation process, leading to the reduction of individual strength. The addition of SF elevates the individual pellet strength of the produced aggregates as shown in Table 3 and Fig. 4 (c), which is attributed to the formation of C-S-H gel generated from the reaction between SF and portlandite produced by the hydration of Portland cement (Liu et al., 2021). The advantage of carbonation is the fast strength development of the LWAs produced with HCP I and HCP III, especially if the carbonation time is optimized. The SF can also benefit the strength development but the pozzolanic reaction takes much longer under normal curing to gain adequate strength.

The average crushing strength in different diameter ranges has been calculated and is shown in Fig. 5. The aggregates with a size from 5 mm to 7 mm show a dominant crushing strength compared to the aggregates with larger sizes. Similar results are reported by G. Perumal et al. (Perumal and Anandan, 2014). They show that crushing strength of individual pellets does not increase with the increasing size of the aggregates.



Fig. 3. Compression load of the produced aggregates.



Fig. 4. Individual pellet strength of the produced aggregates.



Fig. 5. Average crushing strength of the produced aggregates.

#### 3.3. Reaction products and CO<sub>2</sub> uptake

# 3.3.1. Phase identification

The main mineral phases of HCP I and HCP III include C–S–H gel, portlandite, calcite, hemicarbonate, and unhydrated alite, belite as shown in Fig. 1. Fig. 6 (a) and Fig. 6 (b) show XRD patterns of the carbonated aggregates of HCP I and HCP III, respectively. It can be seen that the intensity of diffraction peaks of portlandite decreases while calcite increases significantly with the increasing  $CO_2$  curing duration. This is due to the formation of calcite during the carbonation reaction, which consumed portlandite in hydrated cement. The content of portlandite in HCP III is less than that in HCP I due to the high volume of ground granulated blast furnace slag (GGBFS) contained in CEM III (Hager et al., 2019). The GGBFS also consumed the portlandite because it is pozzolanic, further reducing the portlandite content (Yang et al., 2021b).

The carbonation does not only consume portlandite but also affects ettringite, alite and C–S–H, which can account for the decreasing intensity of those peaks in the corresponding XRD curves (Borges et al., 2010; Šavija and Luković, 2016). The characteristic diffraction peak of ettringite around 10.51 °20 is not visible in HCP I and HCP III before pelletization but occurs in C1A I and C1A III in line with the reformation of ettringite due to the high moisture content (Chen et al., 2020; Yang et al., 2022). During the carbonation process, portlandite is able to delay ettringite carbonation because it acts like a buffer due to its higher reaction rate with  $CO_2$  (Pajares et al., 2003). Therefore, the diffraction peaks of ettringite can be detected in all samples obtained from HCP I but disappear in samples obtained from HCP III after 3-day carbonation, because the overall portlandite content is much lower.

Fig. 6 (c) shows XRD pattern of the aggregates produced with the mixtures of HCP I and SF. After another 28-day curing, the addition of SF preserves the original mineral composition of SOA I but changes the relative intensity of the peaks of portlandite in the corresponding XRD curves, indicating that SF consumed portlandite to generate C–S–H gel, which can effectively glue particles together and provide additional strength (Liu et al., 2021).



Fig. 6. XRD patterns of the produced aggregates (P = portlandite, E = ettringite, A = Alite, C = calcite, Hc = hemicarbonate).

# 3.3.2. Thermogravimetric analysis

Fig. 7 shows the TGA-DTG results of the produced aggregates. In general, the weight loss of the produced aggregates occurs in three stages: the loss of chemically bound water approximately between 75 °C and 550 °C; weight loss between 400 °C and 500 °C associated with the dehydroxylation of portlandite and a further weight loss approximately between 600 °C and 800 °C due to the decomposition of the carbonate groups in calcium carbonates (Liu et al., 2021; Scrivener et al., 2018; Zajac et al., 2020; Zhu et al., 2020).

Fig. 7 (a) and Fig. 7 (b) show the TGA-DTG curves of the carbonated aggregates produced with HCP I and HCP III, respectively. The intensity of the decomposition peaks of chemically bound water occurring around 75–300 °C and portlandite around 400–500 °C decreases with the increasing carbonation period, indicating the simultaneous carbonation of portlandite, C–S–H, AFt and AFm phases, etc. (Šavija and Luković, 2016; Yang et al., 2021a). The significant difference between HCP I and HCP III systems is that no weight loss is observed around 400–500 °C in the HCP III system, demonstrating that calcium hydroxide content is negligible due to the reaction with CO<sub>2</sub>, which is in line with the absence of portlandite peaks in the XRD data. The further weight loss peaks attributed to the decomposition of calcium carbonates become broader and sharper during the carbonation process. For instance, the

decomposition temperature of calcium carbonates starts at around 460 °C and continues till 810 °C as shown in C7A III. This could indicate the presence of amorphous or poorly crystalline calcium carbonate that can decompose at lower temperatures, as reported in the literature (Morandeau et al., 2014). The TGA-DTG curves of the aggregates under normal curing are similar and show the typical weight loss peaks of chemically bound water, portlandite and calcium carbonate. The intensity of the decomposition peak of chemically bound water occurring between 75 and 300 °C increases with the addition of SF due to the formation of secondary C–S–H gel via the reaction between SF and portlandite. The portlandite decomposition peak (400–500 °C) decreases because of the lower initial HCP I content and its consumption via the pozzolanic reaction, which is also in accordance with the above XRD results.

The amount of Ca(OH)<sub>2</sub>, CaCO<sub>3</sub> and chemically bound water is calculated from TGA data and summarized in Table 4. The amount of Ca (OH)<sub>2</sub> in carbonated aggregate samples decreases with the increased carbonation duration. Besides, an obvious increase of the amount of CaCO<sub>3</sub> is observed, from 5.20 wt% to 35.60 wt% in the HCP I system and from 2.55 wt% to 48.57 wt% in the HCP III system. The considerable change demonstrates that calcium hydroxide is not the only hydration product reacting with CO<sub>2</sub> during the carbonation process (Šavija and



Fig. 7. Weight loss determined with thermogravimetric analysis (TGA) and the first derivative of TGA (DTG) of the produced aggregates.

Table 4Weight loss calculated from TGA (wt%).

Sample	Ca(OH) <sub>2</sub>	CaCO <sub>3</sub>	Chemically bound water
HCP I	24.17	5.20	15.42
HCP III	2.53	2.55	8.63
C1A I	21.20	12.63	31.74
C3A I	19.14	14.86	24.55
C7A I	13.95	35.60	18.73
C1A III	-	22.30	28.83
C3A III	-	44.52	21.54
C7A III	-	48.87	18.17
SOA I	23.64	5.41	28.71
S5A I	21.03	4.49	32.26
S10A I	17.30	4.02	32.80

Luković, 2016). The addition of SF undoubtedly consumed portlandite and then contributed to the formation of C–S–H gel as illustrated in Table 4.

Table 4 shows the amount of chemically bound water in the cement hydration phases like C–S–H gel, AFt, hemicarbonate, etc. which benefit the mechanical property (Scrivener et al., 2018; Taylor, 1997). The amount of chemically bound water increases after 1-day carbonation from 15.42 wt% (HCP I) and 8.63 wt% (HCP III) to 31.74 wt% (C1A I) and 28.83 wt% (C1A III), respectively, which is in contrast to the results reported in (Shah et al., 2018; Skocek et al., 2020). This can be attributed in part to the reformation of ettringite (Chen et al., 2020; Ndiaye et al., 2017) according to XRD results. On the other hand, HCP I and HCP

III were treated at 105 °C while the prepared samples were dried at 40 °C, contributing to the higher amount of chemically bound water determined from 75 °C to 550 °C. After 3-day and 7-day carbonation, the chemically bound water decreases because further carbonation contributed to the decalcification or decomposition at a more extensive level of all hydration products (Šavija and Luković, 2016). The addition of SF elevates the amount of chemically bound water due to more C–S–H gel being formed.

# 3.3.3. FTIR analysis

The results of FTIR are presented in general terms by examining the infrared transmittance spectra in the region of 4000–400 cm<sup>-1</sup> as shown in Fig. 8. The broad bands at around 3410 cm<sup>-1</sup>, 1658 cm<sup>-1</sup>, 1640 cm<sup>-1</sup> are associated with the O–H groups in water (McNeil and Kang, 2013) as shown in all samples. Furthermore, the bands at 1107 cm<sup>-1</sup> shown in Fig. 8 (a), Fig. 8 (c) and the shoulder at 1129 cm<sup>-1</sup> detected in Fig. 8 (b) can be attributed to S–O stretching vibrations of the sulphate phases (Myneni et al., 1998). The absorption peak at 3640 cm<sup>-1</sup> originates from the O–H bond in Ca(OH)<sub>2</sub> in the samples containing HCP I (Skocek et al., 2020). This absorption peak disappears in the spectra of the aggregates out of HCP III, in accordance with the above XRD and TGA results that show portlandite was completely consumed after the 1-day carbonation treatment.

The broad bands detected in the range of  $1400-1500 \text{ cm}^{-1}$  and the peaks located at 870 cm<sup>-1</sup>, 711 cm<sup>-1</sup> are related to the CO<sub>3</sub> bending vibration (Li et al., 2017; Qin et al., 2019; Zhan et al., 2018). The



Fig. 8. FTIR spectra of the produced aggregates.

accelerated carbonation resulted in the formation of a large amount of calcium carbonates, visible in FTIR and also verified by the above TGA and XRD results.

The typical sharp peak located at around 958 cm<sup>-1</sup> is associated with the asymmetric stretching vibration of Si–O bonds in the C–S–H gel  $Q^2$ units (García Lodeiro et al., 2009; Yu et al., 1999). This peak shifts from 958 cm<sup>-1</sup> to 1026 cm<sup>-1</sup> after carbonation treatment, in line with the severe decomposition of C–S–H and formation of highly polymerized and elongated form of amorphous silica gel (Qin et al., 2019; Skocek et al., 2020). The absence of portlandite makes C–S–H gel the dominant carbonated subject during the carbonation process (Šavija and Luković, 2016). The formation of silica gel as seen with FTIR can explain the decreasing crushing strength of the aggregates containing HCP III after carbonation curing. The formation of silica gel ruined the structure of C–S–H which is commonly considered as a binding phase.

## 3.3.4. Nitrogen physisorption analysis

The BET specific surface area and pore size distribution were measured as shown in Fig. 9 in order to obtain more details about the pore structure of the produced aggregates on a nanoscale. The specific surface area of the carbonated aggregates containing HCP I decreases

with the carbonation duration as shown in Fig. 9 (a). C-S-H gel contains large amounts of gel pores with relatively higher specific surface area than other hydration products (Taylor, 1997). The carbonation leads to the partial decalcification of C-S-H gel and the reduction of gel pores (Johannesson and Utgenannt, 2001; Thomas et al., 1996). The carbonated aggregates obtained from HCP III instead shows a much higher specific surface area of C3A III and C7A III compared to C1A III. It is reasonable to assume that C3A III and C7A III are strongly affected by the severe decomposition of C-S-H gel as analysed in FTIR results and the subsequent formation of silica gel, which increases the porosity. The addition of SF shows little effects on the specific surface area of the normal cured aggregates as shown in Fig. 9 (c). The cumulative pore area slightly increases and then decreases with the addition of SF. 5% addition of SF undoubtedly contributed to the formation of C-S-H gel, in line with the results obtained from TGA. However, more addition of SF may not benefit the formation of more C-S-H gel as the chemically bound water only increases from 32.26 wt% to 32.80 wt% in TGA results. A SF powder content level of 10% might affect the optimal pore size distribution as reported in (Oltulu and Sahin, 2014).



(c)

Fig. 9. Cumluative pore area of the produced aggregates.

#### 3.3.5. SEM

Fig. 10 (a) and Fig. 10 (b) show the morphology of the produced aggregates originating from HCP I after 1-day carbonation and 7-day carbonation, respectively. The needle-like ettringite grows across C-S-H phases, arranged in a dense structure as shown in Fig. 10 (a) while after 7-day carbonation, ettringite phases seem to have disappeared almost entirely (Fig. 10 (b)). S5A I shows a similar morphology to C1A I, where the ettringite is denser and larger than in C1A I. Notably, the 7-day carbonated aggregates obtained from HCP III shows an entirely different morphology as shown in Fig. 10 (c). The plate-like calcium carbonates are covered by the tiny-needle-like phases which can be attributed to the crystallite calcium carbonates or silica gel, arranged in a loose structure due to the degeneration of C-S-H structure. The carbonation caused the decalcification of the interlayer calcium and then the degeneration of C-S-H dense network structure, in accordance with the FTIR results. Moreover, Fig. 10(e) and Fig. 10 (f) were obtained based on Backscattered Electrons (BSE) mode to illustrate the visible internal cracks in HCP III-type aggregates after 3-day and 7-day carbonation, further confirming carbonation shrinkage and cracking due to the severe decomposition of C-S-H.

## 4. Discussion

The individual strength of the carbonated aggregates originated from HCP I increases from 1.60 MPa to 3.14 MPa after 7-day carbonation, in line with the re-cementation effects of carbonation. Without carbonation, the normal cured aggregates (SOA I) only reach 0.83 MPa, indicating that carbonation can benefit the strength development. A reduction of porosity is observed most likely because the volume of the carbonates formed is 11–12% greater than the volume of portlandite as reported in (Chi et al., 2002; Šavija and Luković, 2016), which can optimize the pore structure and then enhance the mechanical property.

However, the HCP III system shows the opposite results, where the individual strength of the carbonated aggregates decreases from 2.97 MPa to 0.57 MPa, despite more carbonation products being formed over time. This is caused by the decomposition of C-S-H. It is suggested that carbonation of C-S-H is accompanied with a decalcification process. The decomposition of C-S-H gel can happen at a high calcification level (Chen et al., 2006). In HCP I system, the large amounts of portlandite (24.17 wt%) acted as a buffer and were carbonated preferentially, protecting C-S-H from decomposition while densifying the microstructure at the same time (Šavija and Luković, 2016). In HCP III system, however, the small amount of portlandite (2.55 wt%) was consumed rapidly resulting in the decomposition of C-S-H followed by severe carbonation shrinkage and cracking (Chen et al., 2006) and an increase in porosity because of it. This increased porosity also made further carbonation even easier. This is supported by TG results where the portlandite content in HPC I is still 13.95 wt% after 7-day carbonation, while no portlandite is detectable in HCP III after only 1-day carbonation. The peak shift from 958  $\text{cm}^{-1}$  to 1026  $\text{cm}^{-1}$  in FTIR and the significantly elevated specific surface area from 10.69  $m^2/g$  to 42.96  $m^2/g$  after carbonation treatment of HCP III also support this. On all account, the aggregates obtained from HCP III suffered carbonation shrinkage and internal cracking during carbonation process, leading to the reduction of individual strength and increasing water absorption (Borges et al., 2010) as shown in Figs. 11 and 12.

The addition of SF undoubtedly increases the individual strength of the produced aggregates from 0.83 MPa to 2.39 MPa due to the formation of new C–S–H gel to create a dense structure as shown in Fig. 11. The sufficient portlandite content plays an important role in the pozzolanic reaction with silica fume to promote the formation of C–S–H gel (Liu et al., 2021), in line with the increasing content of chemically bound water from 28.71 wt% to 32.80 wt%. Furthermore, the individual strength of S10A I is a little lower than S5A I. As can be seen in Table 4,



(e) C3A III

(f) C7A III

Fig. 10. SEM picture of the produced LWAs.

the amount of portlandite decreases from 21.03 wt% to 17.30 wt% and the amount of chemically bound water increases slightly from 32.26 wt % to 32.80 wt%, indicating the minor increasing content of C–S–H gel, AFt phases, etc. when the addition of SF is elevated from 5% to 10%. The slight increase of hydration phases seems to play little role in the enhancement of mechanical property. As reported in (Tang et al., 2019), the excess use of silica fume with a high surface area is not conducive to producing a compact structure during pelletization. It can be supposed that the strength development of the aggregates originated from HCP I and SF depends on the formation of C–S–H gel and the compaction between SF and HCP I powder.

Carbonation and pozzolanic reaction are both beneficial to the strength development while carbonation is faster and more sustainable. The addition of silica fume undoubtedly elevates the raw materials cost and extends the curing period, thus increasing the total manufacturing cost of LWAs. Meanwhile, using  $CO_2$  uptake to enhance the mechanical property is a proven emissions reduction solution, permanently removing  $CO_2$  from the atmosphere. However, taking the mechanical

performance into account, the carbonation curing period should be optimized to show the jointly positive effects on both CO2 capture amount and strength development. Hence, carbonation might be an inappropriate curing method for HCP III-type aggregates due to destruction of C–S–H and the resulting strength loss. HCP I, as a Ca-rich waste solid containing abundant portlandite, shows an enormous potential for producing high strength LWAs and  $\mathrm{CO}_2$  capture capacity at the same time via the optimized carbonation curing method. For instance, Jiang and Ling (2020) produced steel slag based artificial aggregates to achieve an optimal strength value of 5.24 MPa via post carbonation method, significantly increasing the strength compared to the synchronized carbonation during granulation. However, the optimal carbonation period should be emphasized in any case to avoid affecting negatively the resulting mechanical performance of the artificial aggregates even though CO2 uptake is continuously increasing (Neves Junior et al., 2014).



Fig. 11. Schematic of the re-cementation of the aggregates by carbonation and pozzolanic reaction.



Before carbonation

After carbonation

Fig. 12. Schematic of microsturcture of the produced aggregates before and after carbonation.

#### 5. Conclusions

The comparative study was conducted to figure out the effects of the portlandite amount in HCP I and HCP III on the aggregates under carbonation curing. The addition of up to 10 wt% silica fume into HCP I was attempted to make full use of the considerable amount of portlandite to prepare artificial aggregates under normal curing. The effects of re-cementation during carbonation and pozzolanic reactions on physical properties and mineral composition, microstructure of the produced aggregates have been investigated, which can be crucial and essential for the performance prediction of LWAs originated from the real demolished materials. Based on the findings from this study, the following conclusions can be drawn:

1. Carbonation reaction benefits the fast strength development because the formation of calcium carbonates decreases the porosity, optimizing the pore structure and then benefiting the mechanical property. However, carbonation curing is more appropriate for HCP Itype aggregates than HCP III-type aggregates due to the different amount of portlandite. HCP I-type aggregates show the enormous potential in elevating the strength along with  $CO_2$  capture capacity via the extended carbonation curing until the optimal curing period is reached. The optimal curing period for HCP III-type aggregates is short so that the individual strength and  $CO_2$  uptake are undoubtedly limited.

- 2. The sufficiently available portlandite acts like a buffer to prevent decomposition in C–S–H and other phases caused by carbonation. The lack of available portlandite makes C–S–H the dominant phase subjected to carbonation, accompanying with the decomposition of C–S–H at the extensive carbonation calcification. Consequently, shrinkage and internal cracking during carbonation can occur, leading to a significant reduction of individual strength.
- 3. Addition of silica fume elevates the individual strength of the aggregates originated from HCP I after normal curing due to the formation of C–S–H gel. 5% addition of silica fume is the optimal dosage and S5A I shows the highest individual strength among the normal cured aggregates in this study. 10% of silica fume is harmful to the

compaction of the mixed powder during pelletizing, which leads a reduction of strength performance.

4. Carbonation and pozzolanic reaction are both beneficial to the strength development while carbonation takes the advantages in sustainable development and sustainability in this study, which can reduce CO<sub>2</sub> emissions and manufacturing cost of LWAs.

## CRediT authorship contribution statement

Yanjie Tang: Conceptualization, Methodology, Writing – review & editing. Gang Liu: Conceptualization, Methodology, Validation, Writing – original draft. Katrin Schollbach: Writing – review & editing. Yuxuan Chen: Writing – review & editing. Wei Chen: Supervision, Writing – review & editing. H.J.H. Brouwers: Writing – review & editing, Supervision.

## Declaration of competing interest

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

## Data availability

No data was used for the research described in the article.

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