Preparation and characterization of a new alkali-activated binder for superfine-tailings mine backfill

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Abstract
Recently, the increasing of ultrafine-tailings increases the amount of ordinary Portland cement (OPC) in cemented paste backfill (CPB), which leads to the rise of CPB cost and carbon emission. As a result, it is necessary to develop alternative binders. The present work focuses on the preparation of a new binder, which is activated by a mixture of calcined quarry dust (CQD) and NaOH at a mass ratio of 1:1. The results indicated that CQD/NaOH was more effective than using NaOH or CQD alone in activating blast furnace slag (BFS) and also showed better performance than OPC. The compressive strength of the CPB samples using 10% CQD/NaOH was around 3.78 MPa after curing for 90 days, around 42% higher than the OPC-based CPB samples. The reaction products of CQD/NaOH-activated BFS consisted mainly of C-(A)-S–H, hydrotalcite like phases (Ht), and M-S–H. The generation of Ht phases lowered the Al incorporation into the structure of C-S–H, resulting in lower average Al/Si ratio and mean chain length.

Keywords Alkali-activated BFS · CaO-MgO-NaOH · Cemented paste backfill · Superfine-tailings · Environmentally friendly

Introduction
Mining is considered the foundation of modernization, which has made remarkable contributions to the progress of society. However, it also leads to serious environmental problem (Zhao et al. 2022). During mining production, tailings are one of the most dangerous waste, which leads to serious environmental pollution, such as land occupancy, dam break, and heavy meal leaching (Barcelos et al. 2020). Cemented paste backfill (CPB) is one of the effective ways for tailings disposal (Guo et al. 2021; Zhao et al. 2022, 2021). In recent years, with the national regulation and control of sand and gravel aggregate policy, the production of sand and gravel aggregate is reduced. Thus, some mining enterprises classify tailings and sell coarse particles as aggregates, which results in the reduction of the grain size of the tailings used for CPB. In addition, with the increase of refractory ore year by year and the decrease of ore grade, the grinding fineness becomes finer and finer (superfine-tailings, particle size < 20 μm), which makes it difficult for the CPB to reach the ideal strength (Guo et al. 2021). Ordinary Portland cement (OPC) dosage has to be increased when using such superfine-tailings in CPB, resulting in the increase of CPB cost. On the other hand, carbon emission from the OPC production contributes to the greenhouse effect (Cao et al. 2018; Qiu et al. 2019). As a result, alternative binders need to be developed.

Alkali activated materials (AAMs) are one type eco-friendly cementitious composites and are widely investigated in the last decades (Provis and van Deventer 2014; Dutta and Ghosh 2019). In the production of AAMs, NaOH and Na2SiO3 are widely used alkali activators. Moreover, blast furnace slag (BFS) attracts more attention to be used as the raw precursors due to the abundant sources considering the rapid growth of demand for iron and steel with current rapid industrial development and high economic increase.
year-by-year (Hadji et al. 2021; Quedou et al. 2021). In comparison, the high-temperature treatment for the production of calcined clay can increase the cost of the manufacture of AAMs. The most extensively explored alkali activators in AAMs production are NaOH and sodium silicates. Being a strong alkali, NaOH is propitious to prepare alkali-activated slag (AAS) with competitive cost (Burciaga-Díaz and Betancourt-Castillo 2018), while it is difficult to manipulate the NaOH solution resulting from the highly corrosive nature (Kang et al. 2019). In particular, related safety training and personal protective equipment will be indispensable considering the serious threat to workers’ health and safety from alkali solutions (Provis 2018). These features make the large-scale utilization of AAS less appealing for construction companies and markets (Kang et al. 2019). Besides, it is common that AAS prepared from NaOH presents relatively low compressive strength due to the quick setting and coarse pore structure (Burciaga-Díaz and Betancourt-Castillo 2018). Sodium silicate (Na$_2$SiO$_3$) has been commonly used with NaOH to provide AAS composites with superior properties including satisfactory compressive strength and durability. Although the superior properties, Na$_2$SiO$_3$ is the most energy-intensive component, which is the major concern of the environmental footprint in AAS composites.

AAS composites can also be produced by alternative alkali activator activators with lower environmental implication. Lime (CaO) was explored for the AAS production due to its lower cost compared with NaOH (Park et al. 2016). Calcium silicate hydrates is the primary hydration product in CaO-activated BFS. Magnesium oxide (MgO) has also been examined as an alternative alkali activator. Shen et al. (2011) investigated the hydration of BFS-fly ash binders activated by MgO; the results showed that C–S–H, Mg(OH)$_2$, and MgCO$_3$ were the main reaction products. Jin et al. (2015) investigated the activation effects of reactive MgO on the properties of AAS composites. The results showed that the BFS activated by reactive MgO contributed to higher strength than that of hydrated lime activated BFS composites. Similar phenomenon was also found by Yi et al. (2014), who reported that BFS-based binders activated by MgO gave higher strength than the hydrated lime-based composites. Gu et al. (2014) studied the synergistic effect of CaO and MgO mixtures on the properties of AAS composites. The results indicated that MgO increased the strength at the later curing ages due to the formation of Ht phases, although CaO could promote the C–S–H generation.

Although lime and MgO activated binders continue to attract the attention of researchers due to their uniqueness in some respects, the lower compressive strength and the slower setting compared with that of NaOH based binders and OPC seem to restrict the large-scale applications. Therefore, as one solution for these disadvantages, the use of auxiliary activators to lime or MgO has been proposed. In previous work (Zhao et al. 2020c), we explored the effects of Na$_2$SO$_4$ dosage on the properties of CaO activated BFS considering the slow strength development. The incorporation of only 1% Na$_2$SO$_4$ increased the compressive strength of the AAS binders (Zhao et al. 2020c). The beneficial effects can be attributed to the accelerated reaction of BFS due to the increased pH value of the reaction environment and the additional ettringite (AFt) formation (Zhao et al. 2020c). A more previous work by Park et al. (2016) investigated the influence of gypsum with contents from 0 to 15 wt% on the CaO-activated BFS composites. The results indicated that the 10% of gypsum addition gave the best final compressive strength resulting from the pore-size refinement effect due to the formation of AFt. Oswaldo et al. (Burciaga-Díaz and Betancourt-Castillo 2018) reported the results of strength and hydration products of AAS binders prepared by a mixture of MgO and NaOH at a mass ratio of 1 added at 4–8%. The results showed that the AAS composites activated using only 4% MgO-NaOH presented higher strength than that of composites with 6% NaOH. This is attractive considering the lower dosage of NaOH used in the activator mixture (Burciaga-Díaz and Betancourt-Castillo 2018).

Although the endeavors illustrated above, no reports were found about the AAS production using a mixture of CaO-MgO-NaOH. Reactive CaO and MgO was provided from the quarry dust (mainly consists of dolomite) after calcination. In our previous work (Zhao Yingliang et al. 2020), we explored the properties of calcined quarry dust (CQD) activated slag composites, while relative early strength was obtained, only 3.8 MPa and 28.1 MPa at 1 day and 28 days. Strategies are necessary to further modify the properties of the AAS composites. Therefore, this paper studies the preparation of AAS binders activated using a mixture of CQD/NaOH, which was used for superfine-tailings CPB. The compressive strength of CPB samples was compared with OPC-based samples. The reaction kinetics was explored by the isothermal calorimeter, BSE/IA, and quantification of the chemically bound water content (CBW). Besides, the hydrates were studied via XRD and TG analysis. Finally, MIP, SEM combined with EDS, and $^{27}$Al and $^{29}$Si MAS NMR spectroscopy were used to investigate the microstructure and the elemental composition of the reaction products.

Materials and methods

Materials

Blast furnace slag (BFS) and quarry dust (QD) were collected from Shandong and Hebei, China, respectively, and their chemical composition is listed in Table 1. Sodium hydroxide (NaOH, 98% purity, powder) was bought from Kemeiou Chemical Reagent Co. LTD. Calcined quarry
dust (CQD) was produced using the method in (Zhao et al. 2020a), and the XRD patterns of QD and CQD are shown in Fig. 1. Artificial silica tailings (AST) with SiO$_2$ content higher than 98% was used to replace the natural tailings as aggregate in this study, as reported in previous studies (Guo et al. 2021). The particles size distribution of AST is shown in Fig. 2, and about 85% of AST particles are smaller than 20 μm.

Ordinary Portland cement (42.5R) bought from CR Cement Co. Ltd was used in this study. The chemical composition of the cement is listed in Table 1, measured via X-ray fluorescence (XRF).

### Sample preparation

Three AAS composites were prepared using a mixture of CQD-NaOH at a mass ratio of 1/1 incorporated at concentrations of 6%, 8%, and 10% by the mass of BFS content. Two reference AAS composites activated only by 8% NaOH and 8% CQD, respectively, were also elaborated as comparison. The details are given in Table 2. Solid NaOH particles were dissolved in water and cooled at 20 °C before mixed with BFS. To prepare AAS paste samples, CQD and BFS were dry mixed in a pan for 1 min. NaOH solution was then added into the mixture and stirred for 5 min. After that, the slurry was transferred into molds and cured at 20 °C and 95% R.H. until the designated aging.

For the CPB sample preparation, AST were dry blended with the binders. The binder/tailings ratio was 1/6 in the present work. The following methods were similar to the paste sample preparation, as illustrated above.

### Characterization methods

Unconfined compressive strength (UCS) of the CPB samples was tested after curing for 1, 3, 7, 28, and 90 days according to the standard ASTM C39. Before testing, the top and bottom surfaces of the cylindrical samples were ground flat for good contact with the probe. The ultimate stress was derived from the average values of at least three samples.

The pore structure of the samples was characterized via MIP using an Autopore IV 9500, which can analyze pores with diameters of 0.006–100 μm.

Reaction kinetics were measured using a I-Cal 8000 HPC. About 8 g of samples were placed in an ampoule, and the test was conducted at 20 °C for 72 h.

The hardened samples were crushed, ground to <74 μm and rinsed via immersion into isopropanol (A.R., 99.7%) and stirred for 1 h for the reaction stopping. Next, they were filtered and dried for 24 h. The mineralogical composition of the hydrated sample was tested by XRD using a Shimadzu XRD-7000 in the range of 5° to 50°; TG analysis

| Table 1 Chemical composition of the raw materials (wt%) |
|---------------------------------------------------------|
| Materials | CaO | MgO | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | Na$_2$O | K$_2$O | SO$_3$ |
| BFS      | 37.50 | 9.70 | 30.3 | 15.30 | 0.37 | 0.19 | 0.32 | 0.85 |
| QD       | 63.56 | 31.35 | 4.46 | 0.29 | 0.59 | 0.02 | 0.01 | 0.003 |
| OPC      | 62.14 | 2.85 | 20.33 | 5.73 | 1.54 | 0.13 | 0.32 | 3.15 |
| AST      | 0.02 | - | 98.73 | 0.01 | 0.01 | - | - | - |

Fig. 1 XRD patterns of QD and CQD (Zhao et al. 2020a): D-dolomite, C-calcite, L-lime, M-MgO

Fig. 2 Particle size distribution of AST
was conducted using a STA409PC from room temperature to 1000 °C. The chemically bound water (CBW) contents were derived from the TG results according to the methods described in Wu et al. (2019) as follows:

\[
CBW = \frac{M_{50} - M_{1000}}{M_{1000}} \times 100\%,
\]

where \(M_{50}\), \(M_{550}\), and \(M_{1000}\) are the residual masses at 50, 550, and 1000 °C, respectively.

A small piece was cut from the hardened sample and rinsed using isopropanol (A.R., 99.7%) for 3 days. Next, they were dried for 3 days in a vacuum oven. Zeiss Gemini 300 equipped with an energy dispersive spectrometer (EDS) was used to investigate the microstructure of the hardened samples. Backscattered electron images were taken in a high vacuum environment. An acceleration voltage of 15 kV and a working distance of 3–5 mm were used.

The amorphous component in BFS generally has a homogeneous grey level, which allows it to be identified by image analysis (Kocaba et al. 2012). Backscattered electron images analysis (BSE/IA) method has been widely used to measure the reaction degree of supplementary cementitious materials in blended cement (Mouret et al. 2001) and alkali-activated cement (Brough and Atkinson 2000). In this work, 50 fields were analyzed at 200 runs magnification to obtain the lowest reasonable standard error. Images were analyzed using the ImageJ software. The hydration degree of BFS (DoH) was investigated using BSE/IA following the method reported in Kocaba et al. (2012) and obtained according to the following equation:

\[
DoH(t) = \frac{V_{anhydrous\ slag(t=0)} - V_{anhydrous\ slag(t)}}{V_{anhydrous\ slag(t=0)}}
\]

where \(V_{anhydrous\ slag(t=0)}\) is the volume fraction of initial anhydrous BFS and \(V_{anhydrous\ slag(t)}\) is the remaining volume fraction of unreacted BFS after time \(t\).

A Brucker 400 M NMR spectrometer was used to obtain \(^{29}\text{Si}\) and \(^{27}\text{Al}\) MAS-NMR spectra. Mean chain length (MCL) and average molar Al/Si ratio is calculated following Eqs. (2) and (3) in (Myers et al. 2013):

\[
\text{Al/Si} = \frac{I_{Q_3(1Al)}}{I_{Q_1} + I_{Q_2(1Al)} + I_{Q_2} + 2I_{Q_3(1Al)} + I_{Q_3}}
\]

\[\text{MCL} = \frac{4[I_{Q_1} + I_{Q_2(1Al)} + I_{Q_2} + 2I_{Q_3(1Al)} + I_{Q_3}]}{I_{Q_1}}\]

where \(I_{Q_n}\) refers to the integrated intensity of \(Q^n\).

Results and discussion

Unconfined compressive strength (UCS) of the CPB samples

The effect of alkali activator on the UCS of the CPB samples up to 90 d is shown in Fig. 3. At 1 day, the UCS of CPB samples activated by 6%-CQD/Na was around 0.43 MPa, while those with 8% and 10%-CQD/Na developed UCS of 0.59 MPa and 0.67 MPa, respectively. The UCS rapidly increased until 28 days, reaching 2.82 MPa, 3.15 MPa, and

![Fig. 3 Compressive strength results of CPB samples](image-url)
3.56 MPa for 6%- , 8%- , and 10%-CQD/Na, respectively. After that, the CPB samples witnessed marginal strength gain.

Sample 8%-Na showed the highest UCS value in the first 7 days; however, CPB samples activated by CQD/Na clearly presented higher UCS value than sample 8%-Na at the later curing ages. At 28 days, the UCS for sample 8%-Na was only approximately 2.90 MPa. Previous reports also demonstrated that AAS composites with NaOH as an activator presented higher initial strength but lower strength development rate after curing for a long time. Besides, AAS with 8% CQD presented the lowest strength at the early curing ages and reached about 2.95 MPa at 28 days, which is comparable with that of the composites 8%-Na. Lower early strength in CaO or MgO activated BFS has also been reported elsewhere (Zhao et al. 2020b). The slow hydration of CaO or MgO activated BFS for AAS production is considered one of the main factors restricting its wide application as illustrated in the introduction section.

Figure 4 showed the total heat release through the first 72 h of reaction. Binder 8%-Na showed the highest cumulative heat release at the initial reaction periods, reaching around 162 J/g after 3 days. This corresponds to the highest UCS value in the early periods. A relative lower cumulative heat release was found in CQD/Na activated BFS, about 127 J/g and 91 J/g for 10%- and 6%-CQD/Na, respectively. Using single CQD as an activator reported the lowest total heat release (only around 87 J/g) due to the slow reaction. All these data agreed well with the early strength evolution of the CPB samples.

An isothermal calorimeter is a semi-quantitative method to estimate the reaction kinetics of the BFS hydration. Thus, BSEM/IA was subsequently conducted to quantize the initial wetting and dissolution of the raw materials (Yang and Jang 2020). This peak was followed by a sudden slowdown to the induction period. After that, a second exothermic peak was noticed at around four hours, mainly caused by the massive precipitation of the reaction gels (Zhao et al. 2020b). Variation of the alkali activator did not seem to change the occurrence of the second peak but the intensity. Using NaOH as an activator presented the highest heat flow rate in the second peak, resulting from the higher pH value of the pore solution (Fig. S1). By comparison, a lower heat flow rate was noticed in binder 10%-CQD/Na during this period. Binder 8%-CQD showed the lowest heat flow rate due to the slow reaction between BFS and CaO/MgO, agreeing with previous reports (Zhao et al. 2020c; Ruan and Unluer 2018).

The slow hydration of CaO or MgO activated BFS for AAS production is considered one of the main factors restricting its wide application as illustrated in the introduction section.

**Characterization of the binders**

**Reaction kinetics**

Figure 4 shows the heat flow rate and total heat release of the binder hydration during the first 3 days. A exothermic peak was noticed in the very first hours of the composites reaction, as shown in Fig. 4a, resulting from the initial wetting and dissolution of the raw materials (Yang and Jang 2020). This peak was followed by a sudden slowdown to the induction period. After that, a second exothermic peak was noticed at around four hours, mainly caused by the massive precipitation of the reaction gels (Zhao et al. 2020b). Variation of the alkali activator did not seem to change the occurrence of the second peak but the intensity. Using NaOH as an activator presented the highest heat flow rate in the second peak, resulting from the higher pH value of the pore solution (Fig. S1). By comparison, a lower heat flow rate was noticed in binder 10%-CQD/Na during this period. Binder 8%-CQD showed the lowest heat flow rate due to the slow reaction between BFS and CaO/MgO, agreeing with previous reports (Zhao et al. 2020c; Ruan and Unluer 2018). The slow hydration of CaO or MgO activated BFS for AAS production is considered one of the main factors restricting its wide application as illustrated in the introduction section.

Figure 4b showed the total heat release through the first 72 h of reaction. Binder 8%-Na showed the highest cumulative heat release at the initial reaction periods, reaching around 162 J/g after 3 days. This corresponds to the highest UCS value in the early periods. A relative lower cumulative heat release was found in CQD/Na activated BFS, about 127 J/g and 91 J/g for 10%- and 6%-CQD/Na, respectively. Using single CQD as an activator reported the lowest total heat release (only around 87 J/g) due to the slow reaction. All these data agreed well with the early strength evolution of the CPB samples.

An isothermal calorimeter is a semi-quantitative method to estimate the reaction kinetics of the BFS hydration. Thus, BSEM/IA was subsequently conducted to quantize the
hydration degree of slag (Kocaba et al. 2012). The results are shown in Fig. 5a. BFS hydration was greater in 8%-Na before 28 days. By 1 day, the DoH of BFS was around 25% for 8%-Na, while this number was about 19% for 10%-CQD/Na, while after 90 days, binder 10%-CQD/Na presented the highest DoH value, reaching approximately 56%. This is correlated well with the UCS development as shown in Fig. 3. More reacted BFS indicated higher content of hydration gels formation, favoring the UCS evolution. This could also be confirmed by the chemically bound water (CBW) content from thermal analysis, as shown in Fig. 5b. CBW content has also been used to examine the extent of hydration (Deboucha et al. 2017; Ukpata et al. 2019). Similar to the BSEM/IA method, binder 8%-Na presented the highest CBW content at early hydration periods, but no strong increase was noticed at later ages. The CBW content for binder 10% CQD-NaOH experienced a rapid increase after 3 days, reaching around 23% at 90 days, which is higher than that of paste 8%-Na (about 19%).

Even though the methods used here to study BFS reaction are characterized by the high measurement uncertainty, all of them indicate that a similar trend: BFS activated by NaOH presents faster reaction at the early stages (evidenced by the isothermal calorimeter results in Fig. 4), which contributes to the early strength development as shown in Fig. 3. While although CQD/Na-based binders presented slower reaction at the initial periods, the later reaction was intensive than that of NaOH based composites. This contributed to the increased BFS reaction in later ages, favoring the strength increase.

Figure 6 presents the relationship between UCS and DoH and CBW content. The linear relationship between the strength and DoH and CBW content can be described as $y = 8.84 + 11.84x$ ($R^2 = 0.9$) and $y = 7.36 + 3.52x$ ($R^2 = 0.8$), respectively. The increase in the DoH and CBW content is beneficial for the increase of the CPB strength due to additional hydration products formed in the matrix.
X-ray diffraction

Figure 7 shows the X-ray diffraction (XRD) patterns of the binders at 1 day and 90 days, and the raw BFS is also included as a reference. As shown in Fig. 7, BFS was mainly amorphous with a minor content of crystalline phases like akermanite and gehlenite. In NaOH-activated BFS (8%-Na), calcium silicate hydrate (C-(A)-S–H) was identified as the major reaction products. C-(A)-S–H has been reported to be the predominant contributor to the mechanical properties in AAS with various types of alkali activator and cement-based materials. Hydrotalcite like phase (Ht) was distinguished at around 11.6°, 23.1°, 36.7°, and 39.5° 2θ. Ht is a common reaction gel in AAS samples when sufficient magnesium is present in the raw materials or alkali activators (Haha et al. 2011). In previous work, the formation of additional Ht was found to contribute to a denser matrix, contributing to the strength development (Jin et al. 2014; Burciaga-Díaz and Betancourt-Castillo 2018). Therefore, the generation of more Ht could be related to the higher compressive strength developed in CQD/Na activated binder. The peak of hemicarboaluminate (Hc, C4Ac0.5H12) was at 10.7° 2θ as reported by (Baquerizo et al. 2015; Zajac et al. 2014). Slight carbonation seemed to take place with the presence of peaks located at about 29.4° and 43.2° 2θ. These peaks were assigned to calcite (CC), forming due to the reaction between the dissolved CO3^2− and Ca^2+ released from the BFS. Besides, the remaining akermanite and gehlenite were also present in the unreacted BFS, in line with other works about AAS composites (Burciaga-Díaz and Betancourt-Castillo 2018).

CQD/Na-based samples (6% and 10%) presented similar phase assembles to that of 8%-Na, while compared to 8%-Na, the peak for C-(A)-S–H seemed to be sharper with a lower full width at half maximum (FWHM), 0.648 vs 0.749, as shown in Fig. 7a. This indicates that the C-(A)-S–H gels formed in CQD/Na-based composites are slightly more ordered at the atomic level compared to that of 8%-Na (Gong and White 2016). In NaOH-activated BFS, more Na can be incorporated into the structure of C-(A)-S–H with the formation of more C-(N, A)-S–H, which is predominately amorphous compared with C-(A)-S–H (White et al. 2015). Besides, less Ht was detected in the sample activated by 6%-CQD/Na. Although reactive MgO was provided from the activator (CQD), the lower pH value of the pore solution (Fig. S1) did not favor the leaching of Al from BFS, which limits the formation of Ht. In this case, the pH value of the pore solution determined the phase assembles and formation kinetics. Furthermore, no brucite magnesium silicate hydrate gels (M-S–H) was detected from the XRD patterns, mainly attributing to its amorphous nature, and it is hard to detect by XRD (Jin and Al-Tabbaa 2013).

Lower peak intensity of C-(A)-S–H was noticed in CQD activated BFS (8%-CQD), mainly resulting from the lower pH value of the pore solution, as shown in Fig. S1. Lower pH value restricts the dissolution of BFS, leading to fewer hydration gels formation. This is correlated well with the
DoH of BFS and CBW content (Fig. 6). This could explain the lower UCS value in CQD based AAS composites.

Long time curing (90 days) did not strongly modify the phase assembles, but the intensity of the peaks was noticed to increase. This result is caused by the continual BFS reaction, which leads to the formation of more hydration gels. DoH of BFS and CBW content increased with the hydration time confirmed this result.

**Thermogravimetric analysis**

Figure 8 shows the TG/DTG results of the hydrated binders at 1 day and 90 days. The major humps and some tiny peaks were detected as illustrated below:

1. The dehydration of C-(A)-S–H gel occurred up to 200 °C according to (Kim et al. 2013; Ben Haha et al. 2011 et al. 2011).
2. Hydrotalcite-like phases [Ht, MgAl₂CO₃(OH)₁₆·4(H₂O)] commonly showed two steps of decomposition, around 220 °C and 380 °C, respectively (Machner et al. 2018).
3. The peak located at around 530 °C was assigned to dehydroxylation of silanol (Si–OH) groups in M–S–H structure, as reported previously (Jin et al. 2015; Bernard et al. 2019). The decomposition of M–S–H also occurs between 30 and 250 °C due to the loss of the physically bound water (Bernard et al. 2019).
4. The decomposition of carbonate-containing phases was evidenced by the peak from 600 to 800 °C (Jin et al. 2015).

The weight loss was divided into two main stages (50 °C and 200 °C, 200 °C and 400 °C) and denoted as Δm₁ and Δm₂, respectively. Table 3 summarizes the weight loss of the samples after hydration at 1 day and 90 days. The major humps and some tiny peaks were detected as illustrated below:

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The weight loss was divided into two main stages (50 °C and 200 °C, 200 °C and 400 °C) and denoted as Δm₁ and Δm₂, respectively. Table 3 summarizes the weight loss of the main phases. All the two variables for the AAS composites increased with the curing time, resulting from the continual reaction and generating more reaction products. Composite 8%-Na showed higher values for both Δm₁ and Δm₂ at 1 day due to the fast reaction of BFS under the activation of NaOH with higher pH value (Fig. S1). This was followed by 10%-CQD/Na, and sample 8%-CQD gave the lowest values. After curing for 90 days, composite 10%-CQD/Na witnessed the highest values for these two variables due to the continual hydration even after long time curing. This is confirmed by the DoH value of BFS, showing that sample 10%-CQD/Na presents the highest reaction degree after 7 days, as given in Fig. 5. All these data agrees well with the UCS test results and the hydration kinetics of the composite reaction.

One distinct hump detected on the DTG curve of CQD/Na-based composites at 90 days was the presence of M–S–H especially in sample 10%-CQD/Na. M–S–H is the main reaction product in Mg–Si binders, resulting from the reaction between the dissolved Mg and Si. Although more reactive MgO in sample 8%-CQD, the lower pH value of the pore solution limited the dissolution of BFS than the leaching of Si. The formation of M–S–H was then restricted. This was different from previous work (Burciaga-Díaz and Betancourt-Castillo 2018) about the AAS composites using a mixture of MgO and NaOH, which reported that no M–S–H was detected. The authors infer that this may be attributed to the variations on the characteristics of the BFS and dosage of the alkali activators, which can make differences in the solids solution and then the phase assembles.

**Pore structure**

The pore structure of samples after curing for 1 day and 90 days characterized by MIP are shown in Fig. 9. Figure 9a shows the porosity evolution over curing time. 8%-Na presented the lowest porosity at the early curing ages; this is due to the fast reaction of BFS activated by NaOH, leading to the more hydrates production. At 1 day, the porosity of 8%-Na was around 41%; then, it decreased rapidly and reached about 34% at 3 days. No significant changes were noticed after that, and approximately 30% of porosity for 8%-Na was noticed at 90 days.

By comparison, the porosity of CQD/Na activated CPB samples did not change significantly at the early stages. A decrease of only 3% was noticed for 10%-CQD/Na during the first 3 days, while this number was 15% for that of 8%-Na. This trend is consistent well with the results in Sect. 3.2, which shows the less intensive reaction occurred at the initial stages for CQD/Na-based AAS composites. The porosity for 10%-CQD/Na then decreased rapidly after 3 days, reaching about 27% after curing for 90 days. As reported in Sect. 3.3, more Ht formed in sample 10%-CQD/Na. The formation voluminous Ht has been reported to contribute to the matrix with lower porosity (Wu et al. 2018).

Using single CQD as an activator witnessed a slower evolution in the porosity with a marginal decrease during the first 3 days. This result is in line with the isothermal calorimeter results, which indicated a slow reaction in CQD based AAS composites. After curing for 90 days, the porosity for 10%-CQD/Na was around 33%. The higher porosity was caused by the lower pH value of the pore solution, leading to less hydration products. This result agrees well with the DoH value in Fig. 5, which showed higher content of un-reacted BFS remaining.

Figure 9b and c show the pore size distribution of CPB samples after curing for 1 day and 90 days, and the pore volume statistics are shown in Fig. 9e and f. It can be seen from Fig. 9b that all the samples presented the main peak at around 100 μm at 1 day, mainly as capillary pores, resulting from the low reaction extent of BFS at the initial stages. It can be confirmed by Fig. 9e that more than 70% of the pore was identified as capillary pores at 1 day, no matter
Fig. 8 Thermogravimetric analysis of AAS composites after curing for 1 days (a) and 90 days (b).
Table 3: $\Delta m_1$ and $\Delta m_2$ calculated from the TG analysis of AAS composites

| Weight loss (%) | 6%-CQD/Na | 10%-CQD/Na | 8%-Na | 8%-CQD |
|-----------------|------------|------------|-------|-------|
| 1 day | 90 days | 1 day | 90 days | 1 day | 90 days | 1 day | 90 days |
| $\Delta m_1$ | 3.22 | 5.76 | 4.85 | 11.19 | 6.40 | 8.95 | 2.89 | 6.21 |
| $\Delta m_2$ | 1.46 | 2.60 | 2.13 | 3.95 | 2.55 | 3.10 | 1.61 | 3.07 |

Fig. 9: The pore structure of the CPB samples: porosity evolution (a), pore size distribution at 1 day (b) and 90 days (c), and pore volume statistics at 1 days (e) and 90 days (f)
the binder composition. Besides, sample 8%-CQD clearly presented more capillary pores than other samples, resulting from the formation of lower amount hydration gels. It can be confirmed from TG analysis, which showed that sample 8%-CQD presented the lowest \( \Delta m_1 \) value. By contrast, the lowest capillary pores volume was found in sample 8%-Na, which could be attributed to the fast reaction of binder under NaOH activation. 

More than half of the pores in the AAS composites were identified as mesopores after curing for 90 days (Fig. 9c and f). The continual reaction of BFS contributed to the formation of more gels, which could fill the voids between BFS particles and lead to a more compact matrix. According to previous studies, among the pores in cement-based samples, the gel pore and capillary pore have little effect on the mechanical properties. However, macropores present a

Fig. 10 BSEM micrographs and EDS of the AAS composites at 90 days: a 10%-CQD/Na, b 8%-CQD, and c 8%-Na
negative impact on the mechanical properties (Yang et al. 2021). Different from the situation at 1 day, 8%-Na contained a higher number of capillary pores, being one of the factors to the lower strength after a long time curing. By comparison, it can be seen that the sample 10%-CQD/Na showed the lowest volume of capillary pores, contributing to its higher later strength.

**BSEM**

Figure 10 shows the BSEM of the AAS composites after curing for 90 days. Un-hydrated BFS particles were identified to be dispersed in the matrix with a light gray tone in all AAS composites, while the grey regions between the un-reacted BFS particles refer to the main binding phase (Kocaba et al. 2012). Among the three investigated composites, sample 10%-CQD/Na presented the most compact matrix with less un-reacted BFS particles remaining. This could be attributed to the higher content of hydration gels as reported in Fig. 6 and Fig. 7. A less homogenous matrix was noticed in composites 8%-Na although a higher content of NaOH was incorporated. Pore size distribution also confirmed larger pores and higher porosity in composites 8%-Na, as shown in Fig. 9. More cracks and pores were found in sample 8%-CQD, due to the lower reaction degree of BFS. MIP test results also demonstrated that composites 8%-CQD showed the highest porosity in all curing ages. The qualitative results from energy dispersive spectroscopy (EDS) indicated that the generation of C-(A)-S–H gel intermixed with Ht phases.

Figure 11 shows the Mg/Si vs. Al/Si ratios of the AAS composites after curing for 90 days. The presence of Ht phases could be confirmed by the liner slope (Haha et al. 2011). The Mg/Al ratio was noticed to decrease with the content of MgO in the raw precursors. Sample 8%-CQD presented the highest Mg/Al ratio due to the highest MgO content in the alkali activator. This was followed by composites 10%-CQD/Na, with a Mg/Al ratio of 1.94. The lowest Mg/Al ratio was noticed in sample 8%-Na, reaching 1.77. The positive abscissa indicated the presence of Al in the C-(A)-S–H gels. The Al incorporation in C-(A)-S–H decreased with increasing MgO content from Al/Si = 0.17 for 8%-Na to 0.11 for 10%-CQD/Na and 0.07 for 8%-CQD.

**Solid-state 27Al and 29Si MAS NMR spectroscopy**

Figure 12a shows the 27Al NMR spectra of the samples. The spectrum of the anhydrous BFS was observed as a hump located at around 60 ppm related to Al in AlO₄ in the BFS structure (Shimoda et al. 2008). The broad character of the peak indicated the disorder and heterogeneity of the local structure of BFS (Perez-Cortes and Escalante-Garcia 2020).

This result agrees with the amorphous character in XRD pattern (Fig. 7). In the activated binders, the peak related to AlO₄ shifted to a higher chemical shift value, and new signals appeared corresponding to AlO₆ centered at around 9 ppm.

The spectrum deconvolution of 27Al NMR indicated that Al was inserted into the structure of C-S–H with a sharp peak located at around 70 ppm (Myers et al. 2015a). This peak was more obvious in composite 10%-CQD/Na, agreeing with the results in TG/DTG analysis in Fig. 8, indicating that more hydration gel formation in sample 10%-CQD/Na. The resonance at chemical shift value around 9 ppm corresponded to AlO₆ in layered double hydroxide (LDH) structures including Mg–Al and AFm (Ke et al. 2016; Jones et al. 2003). Composite 8%-CQD presented a higher proportion of AlO₆ resulting from the abundant MgO content. Thus, more Al was incorporated into LDH structure, resulting in lower Al content in C-(A)-S–H gels. This led to a lower Al/Si ratio in composite 8%-CQD as evidenced in EDX results in Fig. 9.

The 29Si MAS NMR spectra of anhydrous BFS and AAS composites after curing for 90 days are reported in Fig. 12b. The BFS showed a broad hump from -60 to -100 ppm, including Q⁰ (-74 ppm), Q¹ (-78 ppm), and Q²(1Al) (-82 ppm) sites, respectively. The small shoulder at -85 ppm indicated the presence of Q² site. After alkali-activated, the spectra of all the investigated composites presented significant changes with the main peak shift toward more negative values. This implies the formation of reaction products with different chemical nature (Burciaga-Díaz and Betancourt-Castillo 2018).

The deconvolution results of 29Si MAS NMR spectra indicated that the anhydrous BFS still exists, while the decrease in the intensity of Q⁰ suggested the consumption of BFS to form C-S–H type hydration gels mainly containing...
four $Q^a$ signals. The peaks located at -78 ppm and -80 ppm are corresponded to $Q^1$ sites, which are mainly related to the chain-end tetrahedral Si units in the C-(A)-S–H structure (Myers et al. 2013). The signal at -83 ppm is related to $Q^2(1\text{Al})$, which is caused by the isomorphous replacement of Si by Al in the C-(A)-S–H chain (da Silva Andrade et al. 2019). The peak at -86 ppm is assigned to $Q^2$ sites, in which tetrahedral Si units are located at the middle groups in the C-(A)-S–H structure. A single peak at around -90 ppm is related to $Q^3(1\text{Al})$ (Myers et al. 2013).

Table 4 shows the quantitative deconvolution results. From Table 4, it demonstrated a higher extent of BFS in composite 10%-CQD/Na, as evidenced by the lower residual BFS fraction and higher C-S–H type reaction product content. By comparison, the higher content of un-reacted BFS was noticed in sample 8%-Na and 8%-CQD, agreeing well with the BSEM-IA results, as shown in Fig. 5. Compared to sample 8%-Na, the content of $Q^2(1\text{Al})$ was noticed to decrease in sample 10%-CQD/Na, indicating that less Al is incorporated into the C-S–H structure. This result is in line with the EDS results. This phenomenon is attributed to that more Al tends to form Ht phases in the presence of higher MgO content, confirmed by the lower Al/Si ratio. As has been reported that no cross-linked $Q^3$ and $Q^3(1\text{Al})$ signals existed in pure C-S–H and only very week signals of $Q^3$ have been detected in C-S–H with low Ca/Si ratio (Bernard et al. 2017; Myers et al. 2015b). A significant quantity of $Q^3(1\text{Al})$ was noticed in the present work, indicating the formation of M-S–H. Similar results have been reported elsewhere (Bernard et al. 2017). This phenomenon is in line well with the

![Fig. 12](image-url)
Conclusion

Using OPC in ultrafine-tailings CPB commonly leads to lower compressive strength. A new BFS-based binder was prepared in the present work; the following conclusions are drawn as follows:

1. The CPB sample using CQD/Na as an activator gave a higher compressive strength after long time curing although slower reaction was noticed in the early stages. After curing for 90 days, the strength of sample 10%-CQD/Na was around 3.78 MPa, which was higher than that of composite using 8% NaOH as a single alkali activator and OPC-based sample.

2. The binder activated by CQD/Na presented a slower reaction rate compared with NaOH-activated sample in the early stages, confirming by the lower heat release rate and DoH values. However, the reaction rate of CQD/Na activated BFS surpassed that of NaOH based samples after 7 days, reaching approximately 56% after 90 days.

3. The sample 10%-CQD/Na showed a compact structure after long time curing, which contributes to its higher compressive strength. The hydration gels contain C-(A)–S–H, Ht phases, and M-S–H.

4. CQD modified sample presented better environmental friendliness, considering its lower Eco2 value; thereby, it was expected to be used as an alternative eco-friendly cementitious composite.

5. Although satisfactory strength was noticed for composites 10%-CQD/Na at later curing ages, the lower early compressive strength compared with NaOH-activated composites may limit the utilization in some cases where high early strength is necessary. Therefore, future studies should focus on the strategies for increasing the early strength.

### Supplementary Information

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### Author contribution

Yong Sun: data curation, writing original draft. Yingliang Zhao: supervision. Jingping Qiu, Xiaogang Sun, and Shiyu Zhang: conceptualization, methodology. Xiaowei Gu: visualization, investigation.

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### Declarations

No competing interests.

### Ethics approval and consent to participate

Not applicable.

### Consent for publication

Not applicable.

### Competing interests

The authors declare competing interests.

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