Experimental Study on Sludge Solidification by Multi-Source Solid Waste

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Abstract. Cement solidification is one of the most widely used methods of sludge improvement, but the cement production process generates high CO_2 emissions, thus a low-carbon curing agent is required. The purpose of this research is to explore the feasibility of sludge solidification by cementitious materials composed of multi-source solid waste, including calcium carbide slag (CCR), ground granulated blast slag (GGBS) and uncalcined completely decomposed granite (CDG). The effect of curing age, composition of precursor, CCR content, CDG particle gradation, initial moisture content of sludge on the unconfined compressive strength (UCS) was investigated by a series of tests. Mercury intrusion experiment (MIP), scanning electron microscopy (SEM) and X-ray diffraction analyses (XRD) were performed to gain additional insight into the mechanism of strength development. The experiment results indicate that, under the admixture proportion of GGBS to CDG in 3:1, the 28-day UCS of solidified sludge by multi-source solid waste is close to that of cement. An increasing CCR content will inhabit the formation of short-term UCS while promote the acquisition of long-term strength. The results also show that the incorporation of fine grains and gravel of CDG is beneficial in the short-term and long-term UCS respectively. In addition, the microstructural analysis verified the formation of hydration products leading to a less-porous and denser inter-structure. Consequently, it was confirmed that cementitious materials composed of multi-source solid waste can be a sustainable and effective curing agent as a substitute for cement.

Keywords: Sludge solidification, Alkali-activated cementitious materials, Solid waste

1 Background

A large amount of sludge with high moisture content and low strength in engineering construction. Solidification is a widely used disposal method for its high efficiency [1]. Currently, ordinary Portland cement (OPC) is the main curing agent in solidification process, however, it poses the problem of high cost and CO_2 emission

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during production is inevitable, which conflicts with the “Carbon peaking and carbon neutrality” strategy of China [2]. Therefore, various types of raw materials were adopted to substitute OPC to achieve satisfactory mechanical properties and sustainable development [3]. For example, calcium carbide slag (CCR), which is a waste residue produced in chemical industry, is feasible to be used as alkali activator for cementitious materials for its high content of calcium hydroxide [4]. Ground granulated blast slag (GGBS) works well as a precursor of cementitious materials [5]. However, these industrial solid wastes have attracted increasing attention and the related economic costs are rising. As a novel solution, the main component of waste soils in Shenzhen, completely decomposed granite (CDG), was recently used as precursor after grinding and calcination [6]. Nevertheless, the calcination process also causes a lot of energy consumption and carbon emission, and low-carbon utilization methods need to be explored.

In this study, solidification tests were conducted to investigate the utilization approaches of solid waste. Cementitious materials were synthesized from multi-source solid waste to solidify sludge, CCR was used as an alkali activator, GGBS was used as the main precursor and uncalcined CDG was used as a complementary precursor. A set of sludge cured by OPC was set as a contrast. Unconfined compressive strength (UCS) of solidified sludge was recorded over curing age, mix proportions, CDG particle types and initial moisture content of sludge. Series of microscopic test such as mercury intrusion porosimetry (MIP), X-ray diffraction (XRD) and scanning electron microscope (SEM) were carried out, aiming to obtain the patterns and mechanisms of each factor.

2 Experimental

2.1 Materials

The sludge and CDG in this study were collected separately from local projects in Shenzhen, China. CCR, GGBS and OPC were obtained from industry suppliers. The basic physical properties are shown in Table 1.

Table 1. Physical properties of sludge and CDG.

Property	Nature moisture content / %	Dry density/ g/cm^3	d_{10}/ mm	d_{30}/ mm	d_{60}/ mm	Liquid limit/ %	Plastic limit/ %
Sludge	60.4	1.38	0.006	0.018	0.055	57.5	28.0
CDG	28.7	1.54	0.112	0.483	1.041	40.6	27.5

The chemical composition of the cementitious materials and sludge are shown in Table 2. To investigate the influence of particle size, CDG particles are further divided into different ranges: fine grain ($d \leq 0.075$ mm), sand grain (0.075 mm $< d \leq$ 2 mm), gravel particles (2 mm $< d \leq 20$ mm), and original CDG particles according to the *Standard for Classification of Engineering Soil* (GB/T 50145-2007). The particle size of GGBS and CCR is 500 mesh (d < 0.03 mm) and 200 mesh (d < 0.074 mm) respectively. All the cementitious materials need to be oven dried to remove moisture before experiment and the sludge was dried at 105 ℃ for 12 hours, crushed and sieved with 2 mm for testing use.

Table 2. Main chemical composition of sludge and cementitious materials. /%.

Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Others
Sludge	51.9	23.5	6.4	2.1	2.2	1.4	12.5
CDG	60.4	33.7	3.4	0.3	0.3	0.9	1.0
GGBS	32.3	17.2	0.2	35.3	9.8	2.3	2.9
CCR	3.0	1.6	0.5	93.5	0.2	0.6	0.6
OPC	27.5	11.1	3.5	46.9	4.0	4.4	2.6

2.2 Testing Program

The main factors considered in the sludge cured by multi-source solid waste materials were as follows: the mixing ratio of GGBS content and CDG content in the precursor; the ratio of alkali activator content to precursor content (alkali activator content / precursor content, A/P ratio); particle size of CDG in the precursors; initial moisture content of sludge. The precursor content was fixed at 15% of the dry soil weight in order to limit a total dosage and achieve the stabilization effect. The levels of each factor are given in Table 3. The samples in each test group were set for curing ages of 7 d, 14 d, and 28 d, except that the No.2 group was tested for an additional 3 d curing age. Additionaly, a 15% dosage of OPC cured sludge group was conducted as a reference. Three replicate samples were tested for each series.

Table 3. Summary of the testing program.

NO.	Moisture Content/ %	GGBS:CDG	CDG Particle Gradation	A/P Ratio
1	60	0:1, 1:1, 2:1, 3:1, 1:0	Original	0.25
2	60	3:1	Original	0, 0.25, 0.5, 0.75, 1.0
3	60	3:1	Fine grain, Sand grain, Gravel	0.25
4	50, 60, 70, 80, 90	3:1	Original	0.25

2.3 Sample Preparation and Testing Method

Sample Preparation. The dry sludge was mixed with water to the specified moisture content. According to the pre-determined proportion, the curing material with specified quality was weighed and mixed into the sludge and stirred for 5 min. The mixed sludge was poured into the mould in three layers and compacted to form cubic specimens with an edge of 70.7 mm. After 2-3 days, the solidified sludge specimens were demoulded and continued for curing until the target age.

Testing Method. The UCS was obtained as the average of three duplicates, using a 10 kN compression machine and a loading rate of 1.25 mm/min which was according to *Standard for soil test method* (GB/T 50123–2019). After compression tests, the specimens were grinded and sieved through a 75-µm sieve for XRD test. Dried blocks are chosen and prepared for MIP testing. Prior to SEM testing, small pieces of samples were prepared with the fractured surfaces coated with gold dust.

3 Results and Analysis

3.1 Composition of precursor

Unconfined compressive strength. The effect of the mixing ratio of GGBS and CDG on the UCS of solidified sludge is shown in Fig. 1. The increase of GGBS content slightly improves the early strength while largely enhance the final strength, suggesting that more hydration products appear in the late stage. The 28 d UCS with a GGBS to CDG ratio of 3:1 is basically equivalent to that with OPC. The UCS of solidified sludge decreases with the increase of CDG content in the precursor, and obviously the activity of GGBS is much higher than that of CDG, indicating that the UCS of solidified sludge is mainly derived from the hydration reaction of GGBS with CCR to generate hydration products, which act as filling and cementation. However, when the precursor is entirely CDG, the solidified sludge still has a low UCS, indicating that uncalcined CDG can play a role in solidification, the reasons are as follows: firstly, the content of SiO_2 and Al_2O_3 in CDG are higher than that in general soil, and correspondingly the content of active SiO_2 and Al_2O_3 are higher than that in general soil; secondly, there are a large number of hydrophobic minerals with large particle size such as kaolinite in the fine grains of CDG, with a small specific surface area, the gel formed by hydration products is easier to completely enclose the soil particles.

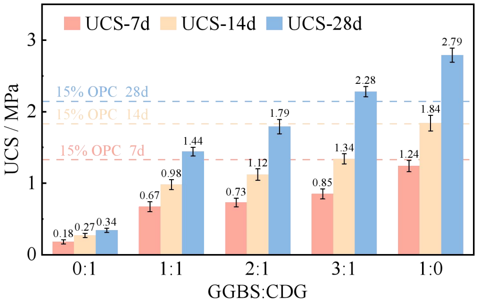

Fig. 1. The effect of the ratio of GGBS and CDG on the UCS.

Scanning electron microscope. Images of solidified sludge samples the age of 7 d and 28 d are shown in Fig. 2(a) and Fig. 2(b) when the ratio of GGBS and CDG is 3:1. The sludge is mainly composed of irregular layered particles. At 7 d, it can be seen that some needle-like or rod-like ettringite crystals are formed in the solidified sludge samples, acting as filling, but there are still many pores in the solidified sludge, which leads to the solidified sludge not being able to form sufficient UCS at this age [7]. Whereas at 28 d, there is a large amount of reticular gel products C-(A)-S-H in the solidified sludge samples, which not only fills the pores, but also wraps the sludge particles to form dense aggregates besides the ettringite, so that only a few tiny pores remain in the solidified sludge, which is the role of filling and cementing of the hydration products. This explains that the UCS of the solidified sludge grows faster in the late stage, and shows that as the curing age increases, the cementation and filling functions provided by the hydration products were the primary source of UCS in the solidified sludge.

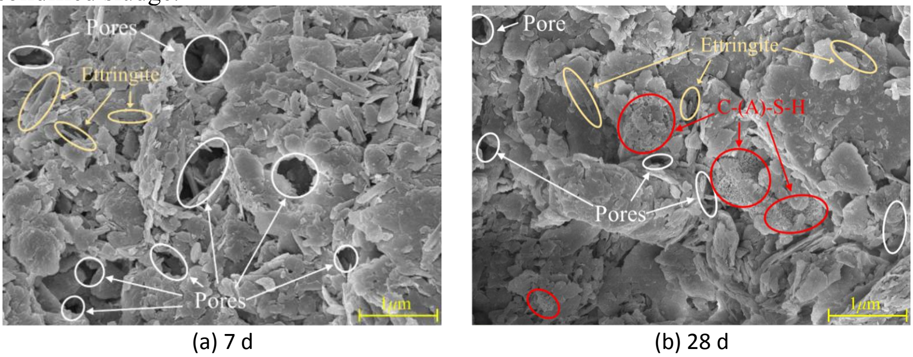

Fig. 2. SEM images of the solidified sludge at different ages.

3.2 The ratio of alkali activator content to precursor content

Unconfined compressive strength. The influence of the A/P ratio on the strength of the solidified sludge is shown in Fig. 3. The results show that the hydration reaction is negligible in the absence of CCR after 3-day curing, and the UCS is markedly enhanced after the incorporation of CCR. For the early stage (3 d, 7 d, 14 d), the UCS exhibited a decreasing initially, followed by an increasing with the increase of A/P ratio This can be explained as follow: The space network structure of GGBS glass body is composed of $[SiO4]^{4-}$ tetrahedron and $[AlO4]^{5-}$ tetrahedron, while CCR can provide OH^- and Ca^{2+} to break the glass body structure of GGBS, release $[SiO4]^{4-}$ and $[AlO4]^{5-}$, generate cementing materials such as C-(A)-S-H and ettringite, and results in a denser structure [8]. The CCR dosage largely determines the alkaline environment, but Ca^{2+} mainly exists in solutions with lower alkalinity. As the CCR dosage increase, the solution alkalinity is enhanced, the dissolution of $Ca(OH)_2$ is inhibited, leading to fewer hydration products and lower strength in the early stage of solidification [9]. However, the addition of increasing CCR reduces the moisture content of solidified sludge, and its particle size is much smaller than that of sludge and CDG, which will lead to a denser structure, so the UCS of solidified sludge increase slightly at the early stage as the A/P ratio continues to increase. As the reaction proceeds, OH^- is continuously consumed and the alkali concentration decreases, weakening the dissolution of $Ca(OH)_2$ and involving more Ca^{2+} to participate in the reaction, so the 28 d UCS is continuously improved.

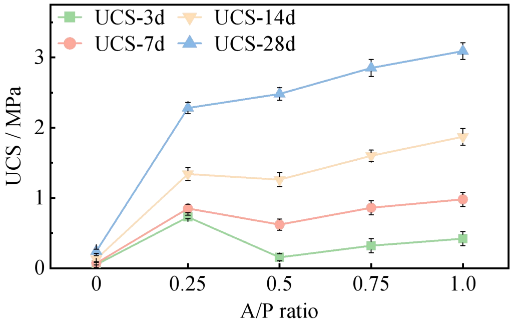

Fig. 3. The effect of the A/P ratio on the UCS.

X-Ray Diffraction. The XRD of solidified sludge with different A/P ratio are shown in Fig. 4(a) and 4(b). Diffraction peaks of portlandite ($Ca(OH)_2$) and calcite ($CaCO_3$) existed due to the incorporation of CCR, while the amorphous diffraction peaks of C-(A)-S-H gel between 20° and 40° and ettringite crystal appeared, implying that new products were generated by hydration reaction. At 7 d, an increase CCR content causes the diffraction peaks of portlandite and calcite to become more pronounced, but there is no significant change in the hydration products, suggesting that higher alkali content does not lead to an increase in the early hydration products, and the trend in the UCS affected by the A/P ratio is due to other factors. At 28 d, although higher CCR content means higher concentration of $Ca(OH)_2$, the phase of portlandite crystal is obviously reduced and the diffraction peaks of hydration products become more obvious, indicating that with the extension of curing age, higher CCR content leads to more hydration products, which is consistent with the trend of 28 day UCS. And kaolinite, the main component of CDG and the part with gelling activity, shows a slight weakening of the diffraction peaks with the addition of alkali activator and the extension of the curing ages, suggesting that a weak reaction occurs in uncalcined CDG under alkali conditions.

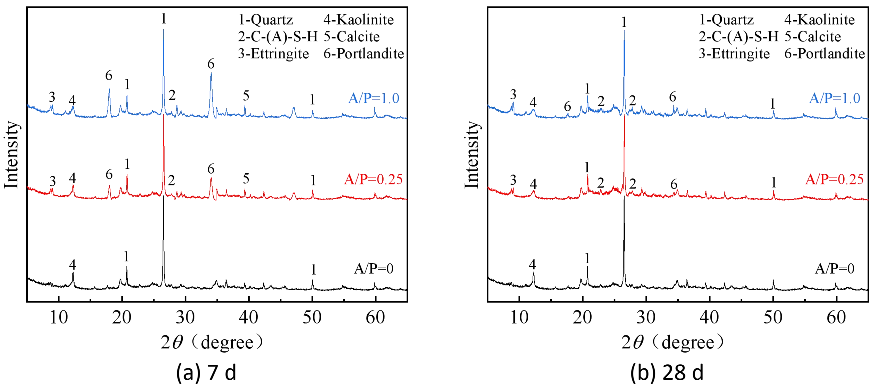

Fig. 4. The XRD of solidified sludge at different A/P ratio.

3.3 Particle types of CDG in precursors

Unconfined compressive strength. The effect of CDG particle gradation on the UCS of solidified sludge is shown in Fig. 5. Among the three types of CDG particles, the highest UCS of solidified sludge at different ages is not consistent. At 7 d, the UCS improves as the particle size decrease, with the solidified sludge mixed with fine particles achieving the highest UCS. Conversely, the UCS strengthen with the increase of particle size, solidified sludge mixed with fine grains obtaining the highest UCS at 14 d and 28 d. This inconsistency may be due to the fact that in the early stage of curing, there is not sufficient hydration product generated, fine grains with smaller size can act as the filling together with the hydration products, thus the solidified sludge incorporating fine grains has a higher UCS at 7 d; while in the late stage of curing, with the increase of hydration products, the contribution of fine grains in filling effect is limited, but gravel and sand particles with larger particle size and higher strength can act as the soil skeleton, thus the solidified sludge with sand and gravel particles has a higher UCS at 14 d and 28 d. Nevertheless, the UCS of solidified sludge with single CDG particles is lower than that with original CDG at all curing ages.

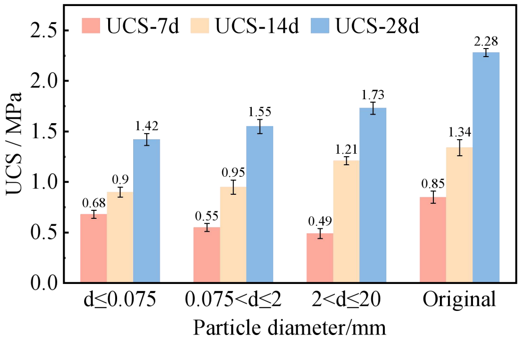

Fig. 5. The effect of CDG particle gradation on the UCS.

Mercury intrusion porosimetry. The cumulative pore volume of solidified sludge with different CDG particle sizes at 7 d and 28 d is shown in Fig. 6(a) and Fig. 6(b), and the volume density distribution is shown in Fig. 7(a) and Fig. 7(b). As the curing age increases, the hydration products continuously fill the pores of the solidified sludge, thereby reducing the total volume of the pores and decreasing the pore size, thereby enhancing the UCS [10, 11]. The solidified sludge will be brought more pores when incorporated with larger size CDG particle. The solidified sludge mixed with fine particles has the minimum pore volume, and the particle diameter corresponding to the peak of the density curve is also the smallest, but this is not consistent with the trend of UCS.

The strength of the solidified sludge depends mainly on the cementation and filling of hydration products [12]. At 7 d, few hydration products are generated, which are unable to cause soil particles to form aggregates. Filling plays a leading role in the formation of strength, and the pores of the solidified sludge are fewer when incorporated with fine particles, which can be effectively filled and lead to high UCS. At 14 d and 28 d, more hydration products are formed and the cementation can cause soil particles to form aggregates, which is the dominant source of strength. Larger aggregates are formed by larger particles, and the strength of gravel and sand grains is higher, hence a larger aggregate can provide higher strength. As for the original CDG, the highest UCS may be a complex superposition of two effects, further research is needed.

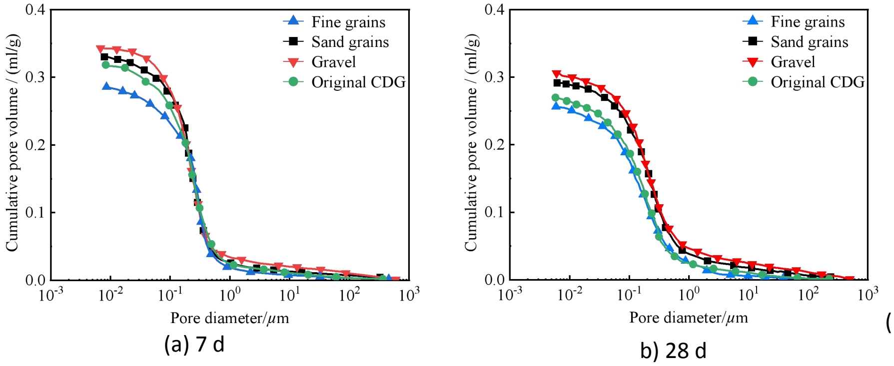

Fig. 6. Cumulative pore volume of cured sludge different CDG particles.

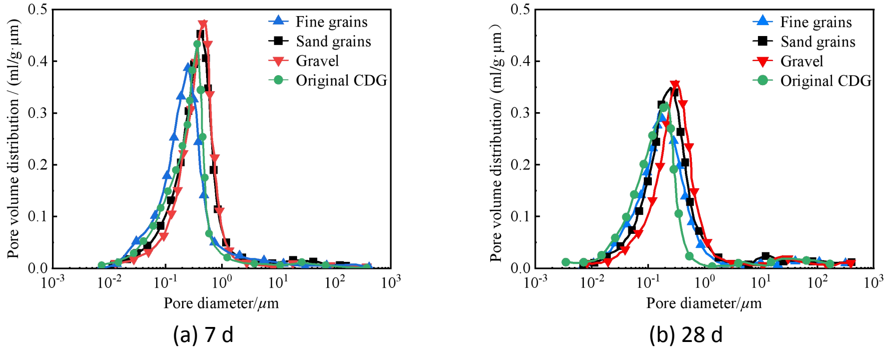

Fig. 7. Pore size distribution of cured sludge different CDG particles.

3.4 The initial moisture content of sludge

Unconfined compressive strength. The influence of the initial moisture content on the UCS is shown in Fig. 8. It demonstrates that the UCS of solidified sludge specimens decreases significantly with an increase in moisture content. As the initial moisture content increases, the proportion of hydration products in unit volume decreases, while the proportion of pore water increases. This weakens the bonding between particles, reduces the density, weakens the tensile force between particles, and disperses the particle arrangement, resulting in a decrease in UCS [13]. In addition, the slope of the UCS line of solidified sludge at all curing ages decreases sharply after the initial moisture content of sludge is 70%, because the actual moisture content of solidified sludge decreases immediately after the incorporation of dry curing materials. In the case that the initial moisture content of sludge is less than 70%, the actual moisture content of the solidified sludge mixed with dry materials is still below the liquid limit, while if the initial moisture content of the sludge is higher than 70%, and the actual moisture content of the solidified sludge mixed with dry materials is above the liquid limit.

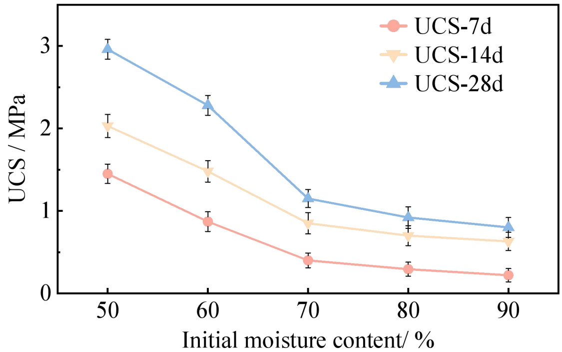

Fig. 8. The effect of the initial moisture content of sludge on the UCS.

4 Conclusions

In this paper, a series of sludge solidification tests were carried out using a multi-source solid waste material consisting of GGBS, uncalcined CDG and CCR, in order to explore the sustainable methods for solid waste recycling, and the conclusions are as follows.

(1) It is feasible to solidify sludge with multi-source solid waste cementitious materials composed of GGBS, CDG and CCR, and the UCS of the solidified sludge with reasonable proportion is close to that of cement solidification.

(2) As the alkali activator, CCR reacts well with GGBS-CDG precursor, a high alkali concentration will slow the hydration reaction in the early stage, but it can still improve the strength at 28-day.

(3) The incorporation of fine particles improves 7-day UCS, the gravel and sand grains enhance the 14-day and 28-day UCS, while the original CDG exhibits the highest UCS at all curing ages.

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