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## **Short Research Article**

# Performance study of Bayer method red mud based controlled low strength alkali inspired concrete

### Abstract.

The controlled low-strength materials were prepared by using red mud, fly ash, and ground granulated blast furnace slag (GGBS) as raw materials, NaOH as an exciter and polycarboxylic acid water reducing agent as a dispersant, and the working and mechanical properties of the materials were investigated at different ratios of red mud and fly ash. When the mass ratio of red mud to fly ash was 2:1, the dosing of GGBS was 10% and the dosing of NaOH was 1.5%, the maximum flow rate of the mix was 236 mm and the maximum unconfined compressive strength of the specimens at 28 d was 6.5 MPa, which satisfied the specification requirements of controllable low-strength materials. XRD, FTIR and SEM were used to investigate the mineral composition and microstructure of the controllable low-strength material. It was found that the hydration of red mud, fly ash, and GGBS under the action of NaOH formed C-S-H, calcium alumina (AFt), and a small amount of hydrated calcium sulfate, C<sub>4</sub>AF; The strength development of materials is closely related to the flocculent, honeycomb, and lamellar structures of hydration products. The results of this study provide a new technological approach to industrial solid waste scaling in the field of controlled low-strength materials.

**Keywords: Compressive, Alkali-activated cements, Alkali-silica reaction, C-S-H**

### Introduction.

Controlled Low Strength Material (CLSM) is a non-compaction, self-leveling, self-consolidating cementitious material, usually with 28d unconfined compressive strength of less than 8.3 MPa, if CLSM is used as filler for future excavation, the 28d unconfined compressive strength requirement is no more than 2.1 MPa. It is widely used in engineering backfill, pavement base, pipeline bedding and other engineering fields because of its simple preparation process, easy construction and low re-excavation cost[1]. It is widely used in the areas of backfilling, pavement bases, pipe bedding, and other engineering applications. Similar to regular concrete, CLSM is mainly composed of cemented materials, aggregates, water and various additives. As the utilization of solid waste resources has been promoted, experts and scholars have conducted numerous studies on the preparation of CLSM from industrial solid waste, which provides an effective way to eliminate industrial solid waste in large quantities [2].

Red mud is an industrial solid waste discharged from alumina industry, and the current utilization rate is less than 10%. [3] The utilization rate is currently less than 10%. Due to the fine particle size, strong alkalinity and high Na<sub>2</sub>O content of red mud, it is difficult to be used in large quantities in building materials such as silicate cement and concrete. However, the main

components of red mud ( $\text{CaO}$ ,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , etc.) are necessary and beneficial for alkali-induced cementitious materials. Therefore, the development of advanced alkali-excited technologies for the preparation of controlled low strength materials using red mud as a raw material and improving the utilization of red mud are of great importance for the application of industrial solid waste reduction and environmental protection. TAN et al. [4] The results showed that the fluidity of CLSM decreased when the cement substitution rate increased from 0 to 30%, but the water secretion rate was improved and the hydration process was accelerated; the 28d compressive strength of CLSM was best when the cement substitution rate was 15%, and all the indexes of CLSM met the standard requirements under the test substitution rate. [5] used silicate cement as cementitious agent and mixed with industrial solid waste such as red mud, fly ash and phosphogypsum to evaluate the engineering properties of CLSM such as flowability, compressive strength, water secretion rate and durability under different matching ratio conditions, which confirmed the feasibility of red mud in the preparation of cement-based controllable low strength materials. The results of the available studies indicate that red mud is feasible as a partial replacement of silicate cement in the production of controlled low strength materials and has potential applications.

In this paper, controlled low strength materials were prepared with red mud and fly ash as the main raw materials, ground granulated blast furnace slag as auxiliary materials and NaOH as alkali exciter, and their working and mechanical properties were evaluated to investigate the microscopic mechanism of strength development of controlled low strength materials prepared from red mud-based total solid waste, in order to provide a technical reference for the preparation of controlled low strength materials from red mud-based total solid waste.

## 1. Test materials and methods

### 1.1 Test material

(1) Red mud (RM). The test red mud was taken from an aluminum industrial plant in Henan, which is Bayer method red mud with a density of  $3.2\text{g}/\text{cm}^3$ ,  $d_{50}$  of  $16.33\mu\text{m}$  and a red powder appearance, and its chemical composition and XRD analysis results are shown in Table 1 and Figure 1, respectively.

Table 1 Analysis results of the chemical composition of the red mud. %

Element	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{CaO}$	$\text{Fe}_2\text{O}_3$	$\text{Na}_2\text{O}$	$\text{TiO}_2$	$\text{K}_2\text{O}$	LOI
Content(%)	10.7	17.5	1.32	55.7	8.15	4.12	0.13	2.38

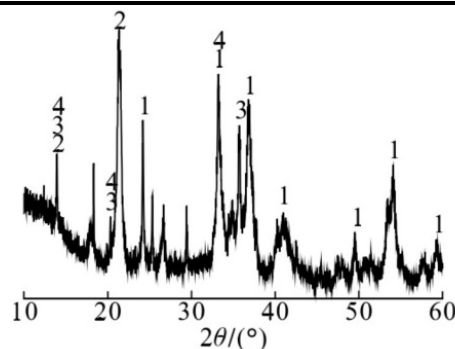


Fig. 1 XRD pattern of the RM.

1- $\text{Fe}_2\text{O}_3$ ; 2- $\text{SiO}_2$ ; 3-Titanium minerals; 4- $\text{AlNaO}_6\text{Si}_2$

From Table 1 and Figure 1, it can be seen that the main components of the Bayer RM are  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$  and also include a small amount of titanium minerals and sodium silica-aluminate

hydrate.

(2) Fly ash (FA). The test FA was taken from a power plant in Henan province and is a Class F FA. The chemical composition and XRD analysis results are shown in Table 2 and Figure 2, respectively.

Table 2 Analysis results of the chemical composition of the FA. %

Element	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>
Content(%)	45.6	34.6	4.54	10	1.73

Element	Na <sub>2</sub> O	TiO <sub>2</sub>	K <sub>2</sub> O	MgO	LOI
Content(%)	0.16	1.25	0.75	0.67	0.7

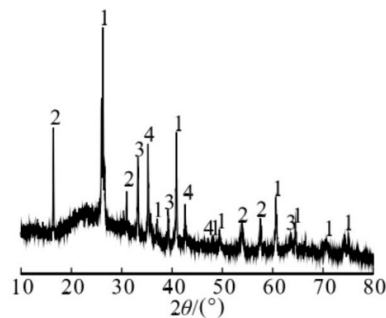


Fig. 2 XRD pattern of the FA.

1-alpha-quartz;2-mullite;3-hematite;4- calcite

As shown in Table 2 and Figure 2, the main components of FA are SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>; the main phases are α-quartz, mullite, hematite and calcite.

(3) Ground granulated blast furnace slag (GGBS). The GGBS was provided by Yongfeng Iron and Steel Co., Ltd. The chemical composition and XRD analysis results are shown in Table 3 and Figure 3, respectively.

Table 3 Analysis results of the chemical composition of the GGBS. %

Element	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>
Content(%)	29.75	14.24	41.45	1.7	2.75

Element	Na <sub>2</sub> O	MgO	MnO <sub>2</sub>	LOI
Content(%)	0.55	7.65	0.53	1.38

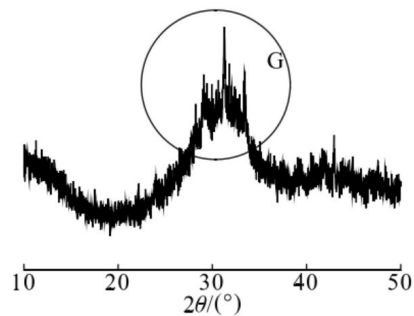


Fig. 3 XRD pattern of the GGBS.

G-Amorphous phase calcium-silica-aluminium minerals

As can be seen from Table 3 and Figure 3, the main components of the GGBS are CaO, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>; The main phase is an amorphous calcium-silica-aluminium mineral.

(4) Alkali exciter and admixture. The alkali exciter used in the test is NaOH with 96%; the admixture is water reducing agent polycarboxylic acid produced by Tianjin Weihe Science and Technology Development Co.

## 1.2 Test method

### 1.2.1 Proportional design and specimen preparation

The controlled low strength material was prepared by fixing the dosing of GGBS at 10%, adjusting the dosing of RM and FA, adding alkali exciter and 0.75% water reducing agent polycarboxylic acid by external doping method, and exploring the effect of NaOH dosing (1%, 1.5% and 2%) on the 28d unconfined compressive strength of the specimens, the specific ratio design is shown in Table 4.

Table 4 Design of test proportion(%)

Samples	RM	FA	GGBS
S1	45	45	10
S2	50	40	10
S3	55	35	10
S4	60	30	10
S5	65	25	10
S6	70	20	10

Weigh the RM, FA and GGBS according to the ratio design in Table 4, and then weigh the polycarboxylic acid water reducing agent of 0.75% of the total mass of the above materials, and mix well. Add NaOH to a certain amount of water and mix it fully until it is completely dissolved. After the NaOH solution is cooled to room temperature, pour the well-mixed material into the NaOH solution and then place it in NJ-160A mixer for mixing according to the standard. After the mixing is completed, it is poured into a 50.5 mm×50.5 mm×50.5 mm mold, and then put into a constant temperature and humidity maintenance box (maintenance temperature is 20±2℃, humidity≥50%) for maintenance.

### 1.2.2 Performance characterization

According to ASTM D6103 cylinder test method, the flow size of the mix was measured by a standard cylinder test mold of φ75mm×150mm; The 28d unconfined compressive strength of the cubic specimen was tested according to ASTM D4832, three times for each set of tests, and the average value was taken; The specimens maintained for 28d were crushed and placed in anhydrous ethanol to terminate hydration, and XRD, FTIR and SEM analyses were performed.

## 2. Experimental results and analysis

### 2.1 Effect of RM dosing on 28 d unconfined compressive strength of specimens at different NaOH dosing levels

Figure 4 shows the effect of RM dosing on the 28 d unconfined compressive strength of the specimens at different NaOH doses.

From Fig. 4, it can be seen that: (i) the 28d unconfined compressive strength of the specimen increases and then decreases with the increase of RM dosing under the same NaOH dosing; (ii) the 28d unconfined compressive strength of the specimen increases with the increase of NaOH dosing under the same RM dosing.

The 28-d unconfined compressive strength of the specimens reached the maximum value

when the mass ratio of RM to FA was 60:30, and the 28-d unconfined compressive strength of the specimens reached 4.2, 6.5, and 7.2 MPa when NaOH was dosed at 1%, 1.5%, and 2%, respectively, which was mainly due to the reduction of the active silica-alumina raw materials in the system due to the excessive dosing of RM, which was insufficient to stimulate the formation of an effective polymer structure, resulting in the reduction of the compressive strength. The 28-d lateral compressive strength of the specimens was less than 2.8 MPa when the Na OH dose was 1% and the RM dose was 45% and 50%, which did not meet the requirements of the CLSM specification; when the NaOH dose was 1.5% and 2%, the 28-d lateral compressive strength of the specimens was greater than 2.8 MPa and less than 8.3 MPa when the RM dose was tested, which met the requirements of the CLSM specification. In summary, under the condition of certain amount of GGBS doping, the highest reaction degree and the largest material utilization rate were achieved when the mass ratio of RM to FA was 2:1. In the case that the NaOH dosing of 1.5% and 2% meet the specification requirements, the subsequent test was conducted with the specimen of NaOH dosing of 1.5% as the object of study.

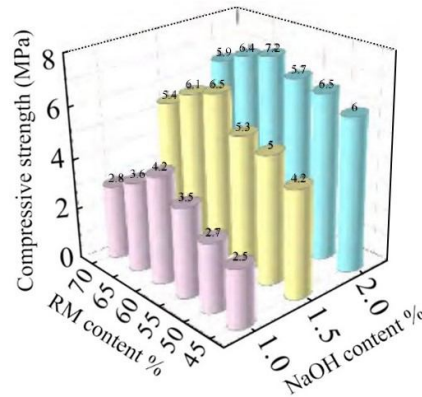


Fig. 4 Influence of the RM addition on 28 d unconfined compressive strength of the samples with different amount of NaOH.

## 2.2 Effect of water-solid ratio and RM admixture on the flow of CLSM

The effects of water-solid ratio and RM dosing on the CLSM flow rate were investigated under the condition of Na OH dosing of 1.5%, and the results are shown in Fig. 5.

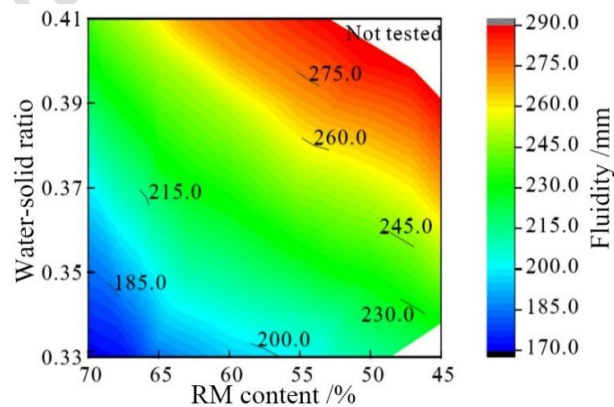


Fig. 5 Influence of water to solid ratio and RM addition on fluidity of CLSM.

From Fig. 5, it can be seen that CLSM with high water-solid ratio and low RM admixture has good flowability and the water requirement increases with the increase of RM admixture. The

corresponding water-solid ratios are 0.385, 0.370, and 0.353 when the flow degree is 230 mm and the RM admixture is 70%, 60%, and 50%, respectively. Since the requirement of flow degree for controllable low strength materials is not less than 180 mm[6, 7] the minimum water-solid ratio was chosen to be 0.35 when the RM admixture reached a maximum of 70%.

The effect of RM admixture on the CLSM flow rate under the conditions of 1.5% NaOH admixture and 0.35 water-solid ratio is shown in Figure 6.

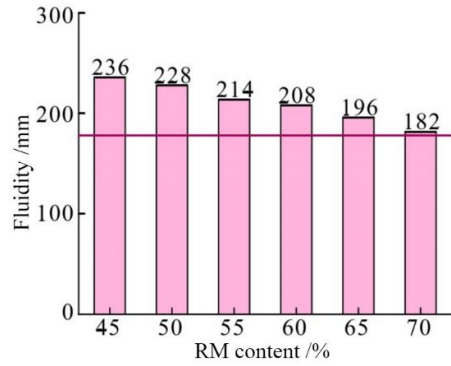


Fig. 6 Influence of RM addition on fluidity of CLSM.

From Fig. 6, it can be seen that the CLSM flowability is negatively correlated with the RM admixture. When the RM admixture is 45%, the flowability reaches 236 mm, while when the RM admixture increases to 70%, the flowability is only 182 mm. Analysis suggests that this is mainly due to the fact that the flowability size of the system is influenced by the specific surface area of RM[8]. The specific surface area of RM is relatively large, and under the condition that the water-solid ratio is determined, the amount of water adsorbed on the surface of RM increases with the increase of its admixture. In addition, due to the spherical shape of FA, the surface structure is round and dense, which can significantly improve the fluidity of the slurry [9]. However, the fluidity of CLSM decreases significantly as the amount of RM increases and the amount of FA decreases.

Therefore, for the RM - FA - GGBS system, to meet the flow performance requirements of CLSM, the water-solid ratio of 0.35 is the most economical and reasonable under the condition of constant GGBS admixture, and when the admixture of RM is less than 60%, the medium-high flow degree (200 mm) requirements of CLSM can be achieved.

### 2. 3 XRD analysis

Figure 7 shows the results of XRD analysis of the specimens after 28 d of curing.

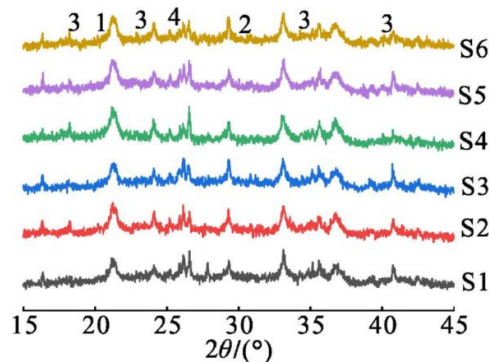
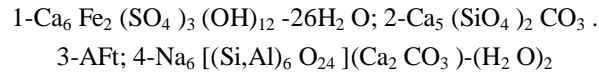


Fig. 7 XRD patterns of the CLSM specimens cured for 28 days.



As can be seen from Fig. 7, the hydration products of CLSM under different matching ratio conditions are basically the same, mainly calcium alumina, silica calcite, calcium chalcocite and hydrated calcium sulfate ferrate. After the addition of NaOH to the material, the reactive oxides in RM react with FA and GGBS under alkaline environment, and the silica-oxygen tetrahedra and alumina-oxygen tetrahedra decompose, and the silica-oxygen and alumina-oxygen bonds break and rebond to form Si-O-Al polymers as well as AFt [10, 11]. The reactive SiO<sub>2</sub> in RM dissolves under the excitation of NaOH and reacts with Ca<sup>2+</sup> and CO<sub>2</sub> in the air to finally form silica calcite[11] The hydrated calcium thiosulfate belongs to the AFm phase, and Fe<sub>2</sub>O<sub>3</sub> in the RM replaces part of Al<sub>2</sub>O<sub>3</sub> in the hydration reaction to produce hydrated calcium thiosulfate. In this system, part of C<sub>3</sub>A is hydrolyzed to form AFt [12] which indicates the formation of silica-alumina-oxygen polymer cemented AFt crystals as well as other hydration products in this system. Some of the AFt diffraction peaks in the figure became sharp, while the diffraction peaks of the other hydration products hardly changed with the different ratios, which indicates that no new phases appeared with the change of the raw material ratios.

#### 2. 4 FTIR analysis

Figure 8 shows the results of FTIR analysis after 28 d of specimen maintenance.

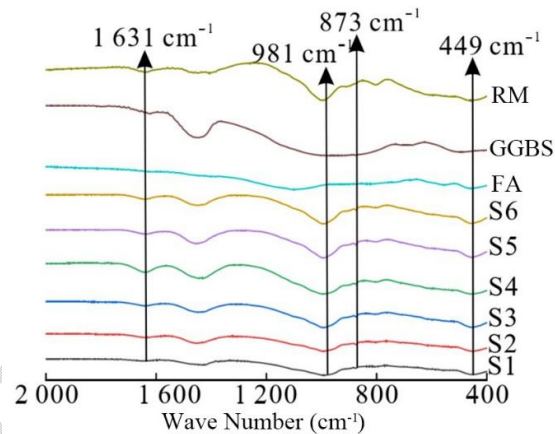


Fig. 8 FTIR patterns of the CLSM specimens cured for 28 days.

In a general sense, 900~1200 cm<sup>-1</sup> are the characteristic peaks of the main band of alkali-excited gelling materials, where the Si-O-Si stretching vibrations in [SiO<sub>4</sub>] at around 1200, 1100, 960, 900 cm<sup>-1</sup> are C-S-H [13, 14]. The absorption bands at 449 cm<sup>-1</sup> and 981 cm<sup>-1</sup> belong to the bending vibration absorption peaks of Si-O-Si (Al, Fe), which are the characteristic bands of tobermorite and C-S-H gels[15]. The bending vibration of H<sub>2</sub>O in the C-S-H gel was observed at 1631 cm<sup>-1</sup>[16]. In addition, the weak absorption peak at 873 cm<sup>-1</sup> is the result of the solid solution of Fe by the hydration product AFt, and the small substitution of Fe for Al leads to the decrease of the symmetry of AFt and the weak red shift of the band[17].

Combined with the results of XRD analysis, this suggests that some of the reactive Fe<sub>2</sub>O<sub>3</sub> in the RM will replace Al<sub>2</sub>O<sub>3</sub> in the hydration reaction to form hydrated calcium sulphate ferrate[18]. In addition, the slight shift of the spectral band at 873 cm<sup>-1</sup> may also be related to the formation of C<sub>4</sub>AF. In general, the characteristic peaks of CLSM did not change significantly under different matching conditions, indicating that the mineral composition and content of the hydration

products of each specimen were generally consistent.

### 2.5 SEM analysis

In order to further analyze the morphology, grain size and distribution of hydration products after 28d curing of the specimens under different ratio conditions, SEM analysis was performed and the results are shown in Fig. 9.

From Fig. 9(a), it can be seen that when the mass ratio of RM to FA is 45: 45, there are more clusters of unreacted RM, and a small amount of AFt is generated at the same time.

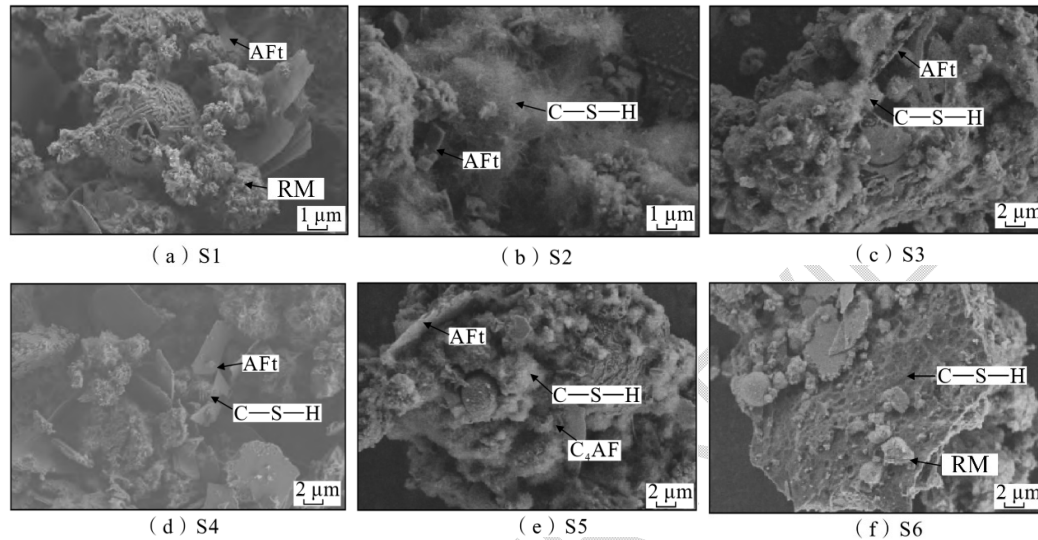


Fig. 9 SEM images of the CLSM specimens cured for 28 days.

From Fig. 9(b), it can be seen that with the increase of RM admixture, the alkalinity of the system gradually increased, and the honeycomb hydration products C-S-H with larger porosity and AFt and some uninvolved FA appeared, and the overall structure was looser, so the strength was also lower.

From Fig. 9(c) and (d), it can be seen that when the mass ratio of RM to FA is 55: 35, denser C-S-H and a small amount of AFt are produced, porosity decreases, and CLSM strength increases; when the mass ratio of RM to FA reaches 60: 30, the hydration products AFt increase, and are densely linked and cross-linked with silica and aluminum-oxygen gel polymers and C-S-H, as well as with other crystals. The hydration products fill and lap each other to form a composite double structure with strength [18-20].

From Fig. 9(e) and (f), it can be seen that when the amount of RM admixture is further increased, the amount of calcium alumina is relatively reduced and the CLSM strength decreases; when the amount of RM admixture is too much, the hydration products are relatively reduced with the reduction of active silica-alumina substances in the system and cannot provide sufficient strength for the structure, which echoes the trend of the compressive strength results.

In addition, the prismatic product of the white intermediate phase can be seen in Figure 9(e) (Figure 10), presumably as  $C_4AF$  [21]. It was found that the strength of the iron phase slurry is mainly dependent on the colloidal-crystalline cementation structure; in high alkalinity slurries, the iron phase is less gelatinous [22]. This explains the shift of the spectral band at  $873\text{ cm}^{-1}$  and the decrease in intensity due to the solid solution of Fe by AFt in FTIR.

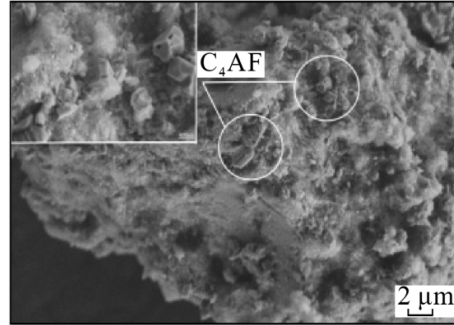


Fig. 10 Micromorphology of  $C_4AF$  in CLSM system.

### 3. Conclusion

(1) With the increase of RM dosing, the 28d unconfined compressive strength of the specimens first increased and then decreased, and the compressive strength of the specimens reached the maximum when the mass ratio of RM to FA was 60:30. In addition, the size of the flow rate is affected by both the specific surface area of RM and the amount of FA admixture, and the flow rate decreases when the amount of RM admixture increases and the amount of FA admixture decreases; when the mass ratio of RM to FA is 70:20, the flow rate is within the defined permissible range, and the flow rate is 182 mm.

(2) NaOH as an exciter can activate the system of RM, FA and GGBS, and the 28d unconfined compressive strength of the specimens can reach 4.2~6.5 MPa and the flow degree is 182~236 mm when the NaOH dosing is 1.5%.

(3) The hydration products of the NaOH-excited system mainly include C-S-H gel polymer and AFt; in addition, in AFt  $Fe_2O_3$  will replace part of  $Al_2O_3$  to participate in the hydration reaction to produce hydrated calcium sulfate and iron, and part of  $Fe^{3+}$  in the RM replaces  $Al^{3+}$  to possibly produce  $C_4AF$ . The gel and crystals in the microstructure are filled with each other and cross bonded to form a relatively dense structure, which provides a microstructural support for the formation of a certain strength of CLSM. CLSM provides microstructural support.

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