

Application of R/Al Ratio in Preparation of Alkali Activated Cementitious Materials in Chemical Industry Based on Industrial Waste Residue Treatment

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It is a concerned question that the Portland cement concrete has poor performance in durability and stability. So, to develop a new binder to replace Portland cement has become an urgent issue. Alkali-activated cementing material (ACM), based on industrial waste residue (slag, fly ash and oil shale wastes), is an alternative to Portland cement which had better durability and high strength. However, the management of the water soluble alkali content, the inhibition of the long-term strength reduction and how to avoid the alkali-aggregate reaction are still under study because of the high content alkali in the ACM production. In this paper, we calculate and regulate the R/Al ratio when the content of Ca, Mg, Si and Al in raw materials appeared major difference. Then, theoretically analyze the influence of R/Al ratio on ACM product, and give explanation to the long-term strength reduction of the ACM. In our study, the R/Al control index shows two applicable formulas, $(\text{Na}+\text{K})/\text{Al} \leq 1.0$ [when $(\text{Ca}+\text{Mg})/(\text{Si}-\text{Na}-\text{Al}) < 0.8$] and $(\text{Ca}+\text{Mg})/(\text{Si}-\text{Na}-\text{K}) \leq 1.5$ [when $(\text{Ca}+\text{Mg})/(\text{Si}-\text{Na}-\text{Al}) \geq 0.8$]. The less the R/Al ratio in the formulas, the better the controllability of the water soluble alkali content and the stability of ACM product.

1. Introduction

Alkali-activated cementing material is a kind of binding material produced by reaction at lower temperature (under 200°C) of aluminosilicate minerals from industrial waste residue and alkali-activator. Over the past decades, alkali-activated cement and concrete has attracted a great deal of attention, for compared with traditional concrete, the production of Alkali-activated cement and concrete are associated with lower energy consumption and lower CO₂ emission, as well as the potential to reach higher mechanical strength and higher stability (Susan and Bernal, 2001; Roy, 1999; Shi et al., 2006; Talling and Krivenko, 1997; Wang et al., 1995). Nonetheless, the physical property and chemical property of industrial waste residue prepared for Alkali-activated cementing material will change greatly according to different production process, production place and time, so was born the undesirable phenomenon like large strength fluctuation and frequent alkali-aggregate reaction when Alkali-activated cementing material was made, and of course it is a great challenge to make sure that the quality control is complete. In this paper, a whole new quality R/Al [(Na+K)/Al] control method was provided as a reply to the hydration mechanism of Alkali-activated cementing material. The effect of R/Al on the structural stability of reaction product was analyzed, then was given the calculation and control method of R/Al on materials with different content of Ca, Mg, Si and Al by experimental research.

2. Experimental program

2.1 The material types and chemical composition analysis of Alkali-activated cementing material

The main Alkali-activated cementing material (ACM) can be classified into two groups, one is silica-aluminum material, and the other is calcium-magnesia material or material with all of them. For the cause to obtain the R/Al calculation and make sure the effect of calculated value on ACM strength practically, in this paper oil

shale residue (OSR) was chosen as representative of silica-aluminum materials, slag (S) and Portland cement clinker (solidification adjustive agent) as representative of calcium-magnesia material.

2.2 Raw materials

The raw materials used to produce the binder are OSR from the Huadian thermal power plants and granulated blast furnace slag from Tonghua steel plant, Jilin Province, China. The BET surface area of slag and OSR was $400\text{m}^2/\text{kg}$ and $399\text{m}^2/\text{kg}$, respectively. The main crystal in OSR was Quartz, along with a small number of Albite, Anorthite, Gehlenite and Hematite, and the rest was amorphous phase. Compared with OSR, the XRD investigation of slag showed that no crystallization peak was observed except an amorphous peak at 20 to 40 degree, which was indentified as solid solution of Gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$) and Akermanite ($\text{Ca}_2\text{MgSi}_2\text{O}_7$).

A commercial Portland cement clinker (PCC) was adopted as adjusting setting agent of which Blaine fineness and specific gravity was $320\text{m}^2/\text{kg}$ and $3300\text{kg}/\text{m}^3$, respectively. The alkali-activator of the ACM was sodium silicate ($\text{Na}_2\text{O}\cdot r\text{SiO}_2\cdot n\text{H}_2\text{O}$) with the silica modulus of the solution 1.2 (Palacios and Puertas, 2007) in amount of 2.5, 5.0, 7.5wt % Na_2O relative to the binder

China standard sand was prepared as fine aggregates in the manufacture of AOC mortar, its density, fineness modulus, and absorption were $2400\text{kg}/\text{m}^3$, 2.4 and 3.75%, respectively.

2.3 Experimental program

The binder/sand ratio of binder mortars adopted here is of 1:3. These mixes were formulated to reach mortar consistency of 90mm and water/binder ratio of 0.5. The specimens of AOC mortars ($4\times 4\times 16\text{cm}$) were designed on the basis of standard procedure GB/T 17671-1999, idt ISO 679: 1989 (Method of testing cements-Determination of strength). The ACM mortar were placed in a high humidity chamber (95%RH) for 24h at $20\pm 2^\circ\text{C}$ to avoid leaching of the alkali-activator, and the mix details are shown in Table 2.

3. Results and discussion

3.1 Compressive and Flexural strength

The mechanical strength of ACM was shown in table 1, Figure 1 and 2. As shown in the figures, the ACM mechanical strength increased as alkali activation progresses, especially when the concentrations of alkali activator solutions increased. However, given the alkali concentration of 2.5% ($\text{Na}_2\text{O}=2.5\%$) in high OSR content samples (1-1, 1-2), lower early and long-term ACM strength can be observed.

This is determined by the low degree amorphous structure of the OSR, when raw materials in low alkaline solution, less breaking down of the Si-O-Si and Al-O-Al covalent bonds would result in less polymerization of low stable structural units, then caused the lower strength observed. When the content of slag increased, the mechanical properties were being improved gradually. As we can see in 120d, the compressive strength and flexural strength of sample 1-7(40%OSR, 60%Slag) are 40.1Mpa and 5Mpa, and in sample 1-9(20%OSR, 80%slag) are 88Mpa and 9.2Mpa, which are higher than the 82Mpa and 8.4Mpa of neat slag sample, so it can be concluded that the OSR/slag mixed material has better effect on alkali-activation than pure alkali one. The P.Duxson' research (Duxson et al., 2007) shows that, not only does the Si/Al ratio determine the geopolymer polymerization degree and the hydration products characteristics, but also influence the geopolymer mechanical strength, when $1.15\leq\text{Si}/\text{Al}\leq 1.90$, increased strength are shown in higher Si/Al ratio samples, and when $\text{Si}/\text{Al}=2.15$, the mechanical strength shows decreasing trend in all samples. In our research, when the alkali activator concentration was 2.5%, the Si/Al ratio of sample 1-1 to sample 1-11 are 3.3, 3.25, 3.2, 3.15, 3.09, 3.03, 2.96, 2.89, 2.82, 2.69 and 2.61 separately, the 28d mechanical strength increased whit the decrease of Si/Al ratio from which we come to the same conclusion with P.Duxson's. But as the curing period reach to 120d, the compressive strength of sample 1-9 ($\text{Si}/\text{Al}=3.16$) is 88MPa, higher than the 87.7MPa of sample 1-10($\text{Si}/\text{Al}=2.69$) and 82MPa of sample 1-11($\text{Si}/\text{Al}=2.6$), this indicates other than the Al/Na ratio and Si/Al ratio, the Ca and Mg content will also affect the result, and we will discuss it in following chapters.

It has been found that increased activator concentration of 5% accelerate the hydration reaction progress, particularly significant when content of slag in a high level, resulting in quick setting disadvantage for the pouring in practice. In this paper Portland cement clinker (PCC) was adopted as the adjustable solidification agent, which has different chemical components from OSR and slag. During the solidification process, PCC particles would react with the alkali-activated products, first forming the C-S-H gel with high Ca/Si ratio and $\text{Ca}(\text{OH})_2$, then the $\text{Ca}(\text{OH})_2$ reacted with active SiO_2 in raw materials forming C-S-H gel with low Ca/Si ratio, in this process water was consumed and the thin C-S-H layer formed would slow down the alkali-activation, hence the solidification time can be manageable.

Table 1: The mechanical strength of alkali-activated mortar

No.	PC C %	OS R %	Sla g %	Sand g	Flexural Strength (Mpa)				Compressive strength (Mpa)			
					3d	7d	28d	120 d	3d	7d	28d	120d
2.5% Na ₂ O												
1-1	0	100	0	1350	0.24	0.26	0.26	0.83	1.02	1.22	2.03	3.17
1-2	0	90	10	1350	0.19	0.24	0.55	0.72	0.99	1.2	2.8	4.17
1-3	0	80	20	1350	0.24	0.25	0.62	1.44	0.8	1.12	5.1	9.32
1-4	0	70	30	1350	0.23	0.30	0.89	2.12	0.89	1.18	11.38	13.58
1-5	0	60	40	1350	0.23	0.32	0.95	2.63	0.98	1.23	11.42	18.7
1-6	0	50	50	1350	0.26	0.36	0.96	2.5	0.96	1.21	11.5	18.69
1-7	0	40	60	1350	0.74	1.47	2.51	5	2.58	5.6	23.3	40.1
1-8	0	30	70	1350	1.29	2.54	4.9	8.6	4.6	16.6	50.6	74.6
1-9	0	20	80	1350	1.36	3.9	6.8	9.2	6.8	36.4	61.6	88
1-10	0	10	90	1350	2.55	4.8	7.2	11	24.1	38.7	59.7	87.7
1-11	0	0	100	1350	5.2	7	7.7	8.4	28.7	45	65.7	82
5.0% Na ₂ O												
2-1	4	100	0	1350	1.12	1.3	2.6	2.9	5.3	7.5	10.6	14.2
2-2	4	90	10	1350	1.5	1.9	2.9	3.5	7.9	9.7	13.7	21.9
2-3	4	80	20	1350	1.7	2.3	4.3	6.9	7.5	11	17.4	36.1
2-4	4	70	30	1350	1.64	2.5	7.3	11.8	8.4	12	49.6	78.1
2-5	4	60	40	1350	1.66	4.24	7.9	11.3	8	19.5	71.9	101.5
2-6	4	50	50	1350	3.75	6.35	8.1	9.6	23.3	57.4	81.5	93.1
2-7	4	40	60	1350	3.41	5.3	8.6	11.3	40.7	56.6	86.2	105.3
2-8	4	30	70	1350	3.63	5.9	8.7	12.2	46.2	79.7	80.7	123
2-9	4	20	80	1350	3.87	6.25	9.1	11.2	62.7	85.3	93.1	102
2-10	4	10	90	1350	5	6.8	9.4	9.1	63.1	86.5	98.7	94.05
2-11	4	0	100	1350	6.22	8.0	10.1	10.0	73.3	95.2	103	93.1
7.5% Na ₂ O												
3-1	4	100	0	1350	3	3.8	7.4	8.0	14.2	28.1	41.88	51.7
3-2	4	90	10	1350	4.17	5.6	7.8	8.9	21.8	38.5	43.61	55.5
3-3	4	80	20	1350	4.6	6.5	7.6	9.6	26	43	65.12	75.3
3-4	4	70	30	1350	5.2	6.4	9.8	9.7	31.6	51.2	81.2	90.6
3-5	4	60	40	1350	5.2	6.5	9.7	9.8	42	65	98.39	98.6
3-6	4	50	50	1350	5.5	6.4	10.1	9.8	59	80.4	106	108.3
3-7	4	40	60	1350	5.7	5.9	8.3	8.3	67.1	80.7	105	105.3
3-8	4	30	70	1350	6.4	6.1	8	12.2	73.8	87.6	103.2	133.35
3-9	4	20	80	1350	7.15	6.7	7.6	10.1	71.9	91.2	104.5	110
3-10	4	10	90	1350	7.2	7.95	9.8	9.3	81.7	93.6	108	103.6
3-11	4	0	100	1350	8.3	8.3	10.8	10.7	88.3	95.9	118	107.3

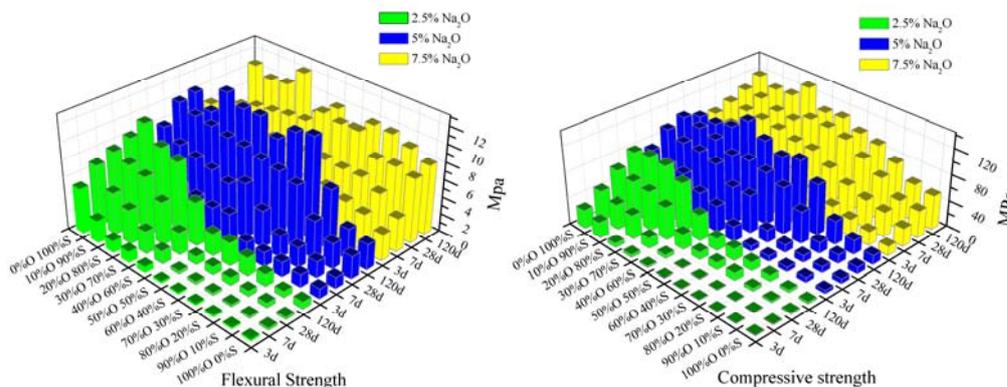


Figure 1: The mechanical strength of the ACM cure at (95%RH) and 20±2 °C

However the mechanical strength of high OSR content samples (2-1, 2-2) still can't meet engineering need for less polymerizing in materials, but with the increase of slag content, the mechanical properties showed evident improvement. When the slag content reached to 30%, the compressive strength of sample 2-4 in 120d were

78.1MPa, account for 216% of the sample 2-3 (20%slag), the Si/Al ratio of these two were 3.23 and 3.18. When the slag content reached to 70%, the early strength of sample 2-8 increased rapidly, the compressive strength of it in 3d was 46.2Mpa and in 120d was 123Mpa. With the further increase of slag content, samples (2-9, 2-10, 2-11), though still possessed relatively higher early and post strength, appeared noticeable strength reduction comparing 120d with 28d. In neat slag sample, the strength reduction rate reached to 9.6%. In addition to the variation of activator amount and soluble alkali amount, such performance was also affected by the raw material chemical composition.

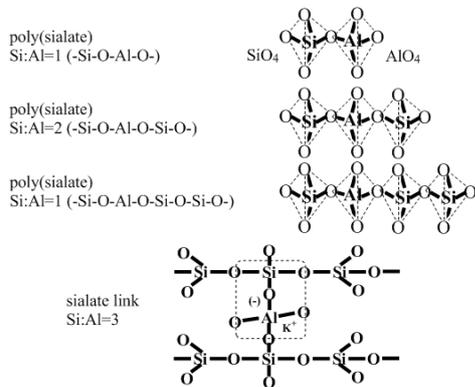


Figure 2: Poly (sialates) structures according to Davidovits

With activator concentration increasing, all ACM samples showed better mechanical strength. However the mechanical strength of high OSR content samples (2-1, 2-2) still can't meet engineering need for less polymerizing in materials, but with the increase of slag content, the mechanical strength showed evident improvement. When the slag content reached to 30%, the compressive strength of sample 2-4 in 120d were 78.1MPa, account for 216% of the sample 2-3 (20%slag), the Si/Al ratio of these two were 3.23 and 3.18. When the slag content reached to 70%, the early strength of sample 2-8 increased rapidly, the compressive strength of it in 3d was 46.2Mpa and in 120d was 123Mpa. With the further increase of slag content, samples (2-9, 2-10, 2-11), though still possessed relatively higher early and post strength, appeared noticeable strength reduction comparing 120d with 28d. In neat slag sample, the strength reduction rate reached to 9.6%. In addition to the variation of activator amount and soluble alkali amount, such performance was also affected by the raw material chemical composition.

As the activator content is of 7.5%, there was a big increase of the long-term stage strength in all samples including ones with high content of OSR. The compressive strength of neat OSR sample in 28d reached to 41.88Mpa and in 120d to 51.7Mpa. And with the increase of slag content, the growth of long-term strength in samples appeared more evident.

3.2 The establishment of the R/Al control method of Alkali-activated cementing materials

It is well-known that Alkali-activated cementing materials including "Alkali-slag cement" or "Alkali-activated slag cement" and geopolymer. Glukhovskiy (Glukhovskiy, 1959) had already made crucial investigations about the hydration products of Alkali-slag cement. He identified that the hydration products as being composed by calcium silicate hydrates and calcium and sodium aluminosilicate hydrates. When raw materials contained clay minerals, the hydration products are mainly of zeolite products. According to Davidovits (Davidovits, 1979; Davidovits, 1991), who developed and patented binders obtained from the alkali-activation of metakaolin, having named it after the term "geopolymer" in 1978. And he suggests use the name "polysialates" to understand geopolymer, in which Sialate is an abbreviation for aluminosilicate oxide. The sialate network is composed of tetrahedral anions $[\text{SiO}_4]^{4-}$ and $[\text{AlO}_4]^{5-}$ sharing the oxygen, which need positive ions such as (Na^+ , K^+ , Li^+ , Ca^{2+} , Mg^{2+}) to compensate the electric charge of Al^{3+} in tetrahedral coordination (after dehydroxilation the aluminium changes from coordination 6 (octahedral) to coordination 4 (tetrahedral)). In conclusion, as shown in figure 2 the reaction product of Alkali-activated cementing material had lots of polymers containing silicon-aluminum linking (Duxson et al., 2005).

For Alkali-activated cementing materials, the tetrahedral anions $[\text{AlO}_4]^{5-}$ existed in silicon-aluminum linking is very important. To become non-free ions in the network structure, the strong alkaline cations (K^+ , Na^+) in structure can only be restricted by nothing but the negative charge of tetra-coordinated Al^{3+} in silicon-aluminum linking. Otherwise, strong alkaline cations would continually dissolve in the water to erode and

destroy the network structure formed by aluminum-oxygen tetrahedron and silicon-oxygen tetrahedron. On the other hand, some aluminum-oxygen tetrahedron, having no direct polymerization with silicon-oxygen tetrahedron, would react with K^+ and Na^+ forming soluble sodium and sylvite, which could lower the solution alkalinity to relax the destructive power from K^+ and Na^+ . So the amount Na/Al ratio in structure, especially between K^+ , Na^+ and Al^{3+} , is a key point to the stability of aluminosilicate polymer structure. In aluminosilicate polymer, one aluminum-oxygen tetrahedron in the linking has only one spare negative charge, so it can reach charge balance with only one alkaline cation (K^+ or Na^+) and make one of them become non-free ion, the others and soluble salts would dissolve in the water to destroy the structure. Theoretically, to maintain the polymer stability, the $(Na+K)/Al$ ratio (atomic ratio) should be less than or equal to 1.0, and all Al^{3+} bonded to silicon-aluminum linking, as well as the Al/Si is less than or equal to 1.0 (for any two aluminum-oxygen tetrahedrons cannot share same one bridging oxygen).

As Ca^{2+} or Mg^{2+} were in aluminosilicate polymer, $[SiO_4]^{4-}$ with strong electronegativity would react with them forming products like C-S-H gel in the first place, after which the rest silicon-oxygen tetrahedron can link aluminum-oxygen tetrahedron into a chain. Therefore, the growth of Ca^{2+} in the system would increase the Ca/Si ratio in C-S-H gel, which would affect the product structure stability and reduce the polymerized silicon-oxygen tetrahedron amount leading to the growth of Al/Si ratio in silicon-aluminum chain, so much so that it could also increase the aluminum-oxygen tetrahedron amount unpolymerized with silicon-oxygen tetrahedron, consequently resulting in the lack of Na^+ fixation and rise of soluble alkali and soluble salt amount, and then furthermore weaken the structure stability.

As the Mg^{2+} in structure is of small atomic radius, generally it will not or less affect the product network structure, unlike the Ca^{2+} which would clearly affect the aluminosilicate polymer stability. But still it will affect the amount of formed silicon-aluminum chain as well as the Al/Si ratio in the chain, and then same as the Ca^{2+} result in the amount change of soluble alkali and soluble salt in polymer.

From the above, we can see that in the hardening system, the amount of silicon-aluminum chain is directly linked to the Ca/Si ratio and Mg/Si ratio. When the harden system has a fixed amount of Si and $(Ca+Mg)$, the higher $(Ca+Mg)/Si$ ration the less Si^{4+} and Al^{3+} polymerized into a chain. For most alkali-activated cementing materials, the hydrated calcium silicate gel is of C-S-H(I) which could coexist with Aluminosilicate polymer and silicate gel, and the Ca/Si ratio of it was in the range of (0.8~1.5). By this range, a simple division can be done as $Si-(Ca+Mg)/(0.8\sim 1.5)$ [if the S^{6+} is in a high level in the system, namely high SO_4^{2-} level, the number of $(Ca+Mg)$ should be subtracted in amount to certain S^{6+} according to Ca/S ratio of calcium sulfoaluminate hydrates]. The $Si-(Ca+Mg)/(0.8\sim 1.5)$ is to be considered the rest amount of Si^{4+} after Ca^{2+} and Mg^{2+} binding into C-S-H, which is also the amount available polymerize to Al^{3+} in to a chain. When Al^{3+} is greater than or equal to the quotient, Al/Si ratio in the silicon-aluminum chain would be 1:1, and this quotient is defined as Al^{3+} amount needed for Na^+ and K^+ fixation. As long as the Na^+ and K^+ amount is less than or equal to the quotient in the system, theoretically soluble alkali and soluble salt amount should be nil. Therefore, the formula can be put as follows:

$$\frac{Na+K}{Si-(Ca+Mg)/(0.8\sim 1.5)} \leq 1 \quad \text{equally} \quad \frac{(Ca+Mg)}{Si-Na-K} \leq (0.8\sim 1.5) \quad (1)$$

As we can see in the formula, the Na^+ and K^+ amount and $(Ca+Mg)/Si$ ratio is the key points to it. When $(Ca+Mg)/(Si-Na-K) \leq (0.8\sim 1.5)$, it indicated that no soluble alkali ion and soluble salt in the product. For lower $(Ca+Mg)/(Si-Na-K)$ value shows possibility of less $(Ca+Mg)/Si$, as well as higher polymerization degree of Si and Al coordination polyhedron in the product of general Alkali-activated cementing materials in which Ca^{2+} were much more than Mg^{2+} . When $(Ca+Mg)/(Si-Na-K) \geq (0.8\sim 1.5)$, it indicated the presence of soluble alkali ion and soluble salt in hydration products, and the soluble alkali will be overnumbered soluble salt by the ratio grows. In addition, when $(Ca+Mg)/(Si-Na-K) \geq (0.8\sim 1.5)$, theoretically there would be no silicon-aluminum tetrahedron chain but hydrated calcium aluminate, and all Na^+ and K^+ were to form soluble alkali and soluble salt. As the amount of Na^+ , K^+ , Ca^{2+} , Mg^{2+} and $(Ca+Mg)/Si$ ratio grows in the materials, with more soluble alkali and soluble salt and low silicon-aluminum tetrahedron polymerization degree, the hydration products would have no doubt to suffer the consequence of poor stability. Thus, this formula can give a relative correct expression to the product stability, with precondition that

$$Al \geq Si - \frac{Ca+Mg}{(0.8\sim 1.5)} \quad \text{equally} \quad \frac{Ca+Mg}{Si-Al} \geq (0.8\sim 1.5) \quad (2)$$

If $(Ca+Mg)/(Si-Al) < (0.8\sim 1.5)$, equally $Al^{3+} < Si-(Ca+Mg)/(0.8\sim 1.5)$, the Al/Si ratio would be less than 1 in silicon-aluminum chain, the Al^{3+} available for fixing Na^+ would be perfectly fit to the Al^{3+} amount existed in system. Under this precondition, as that $(Na+K)/Al \leq 1.0$, theoretically the soluble alkali and soluble salt in system would be nil. When $(Na+K)/Al > 1.0$, the presence of soluble alkali and soluble salt can be determined, and the

more the presence and difference between soluble alkali and soluble salt, the less stable the alkali-activated cementing material product structure.

4. Conclusion

It can be seen from the above analysis that in the view of hydration products, the (Na+K)/Al ratio is quite important to the quality control of alkali-activated cementing materials.

For ensuring the alkali-activated cementing material products stability, the R/Al calculation method, application range should be various due to great amount change of Ca, Mg, Si and Al in materials. Theoretically to make sure that the products contain no soluble alkali and soluble salt, and have higher polymerization degree, when $(Ca+Mg)/(Si-Al) < 0.8$, it is necessary to maintain $(Na+K)/Al \leq 1.0$, and when $(Ca+Mg)/(Si-Al) \geq 0.8$, to maintain $(Ca+Mg)/(Si-Na-K) \leq 1.5$ (no more than the application up limit). Provided the calculated R/Al is in or near the application range (if the S^{6+} is in a high level in system, the values in formula of Ca+Mg and Al should be subtracted in amount of the extra S^{6+} according to Ca/S ratio and Al/S ratio in calcium sulfoaluminate hydrates), the lower the R/Al ratio, the better the alkali-activated cementing materials product stability (less soluble alkali and higher polymerization degree). The experimental verification of R/Al calculation method would be expounded in part 2.

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