

Experimental investigation on alkali activity and prevention measures of Low-grade metamorphic rock aggregates

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Abstract

The potential alkali–aggregate reaction (AAR) is a barrier to the application of low-grade metamorphic rock (LMR). In this paper, a series of experimental investigations was conducted to study the potential of LMR for the alkali–aggregate reaction and examine the factors influencing the AAR of low-grade metamorphic rock; correspondingly, some preventive measures were proposed. The results indicated that the uniaxial saturated strength of the LMR was more discrete, and all the LMR samples contained a certain amount of microcrystalline quartz and were silicate rocks. In the accelerated mortar bar experiments, the 14-day expansion ratios of all the untreated concrete samples containing different LMR aggregates were larger than 0.1%, which revealed the potential of the low-grade metamorphic rock for the alkali–silica reaction (ASR). The effect of fly ash on restraining the AAR was obvious, and the 14-day expansion ratios of some specimens containing the fly ash were smaller than 0.03%. Moreover, the total alkali content of the concrete had a significant impact on the AAR; in fact, a higher alkali content of the concrete resulted in a higher alkali–silica reaction rate, a longer reaction cycle, and a larger expansion ratio. Thus, the optimum alkali content of the concrete should be controlled to be lower than 1.0% to prevent the ASR. The results of the scanning electron microscopy (SEM)–energy dispersive X-ray spectroscopy (EDS) revealed alkali–silica gel and cracks in the interfacial transition zone (ITZ) of the mortar sustaining the AAR and confirmed an increase in the Si and Na content of the ITZ. On the whole, it is suggested that the alkali reactivity of the LMR aggregate, the total alkali content of the concrete, the mineral admixtures, the stone powder content of the concrete, and the service conditions of the concrete should be examined when the LMR aggregate is used in actual engineering projects.

Keywords: aggregates; alkali activity; low-grade metamorphic rocks; micro-structure; prevention measures

1. INTRODUCTION

Alkali-Aggregate reaction (AAR) is an expansive reaction between alkali-active mineral components and the soluble alkaline solution from cement and admixtures in concrete pores [1]. The AAR process can be generally characterized in several successive steps. Initial attack from hydroxyl ions on SiO₂ firstly occurs at the aggregate-cement paste boundary, which leads to the formation of silanol groups at the SiO₂ surface. Siloxane groups are then formed, accompanied with polymerization. After adsorbing alkaline and Ca²⁺ ions, alkali–silica gels are eventually formed [2-4]. Since the alkali–silica gels have a strong water swelling capability, the expansion stress generated in the swelling process may exceed concrete strength, which therefore can further cause cracking damage to the concrete structures [5].

Since the problems of the alkali–aggregate reaction (AAR) were first identified in the early 1940s in the United States [6], it has received great attention. It is well known that the alkali–aggregate reaction has two forms: the alkali–silica reaction (ASR) and the alkali–carbonate reaction (ACR) [7]. Three basic elements, including certain humidity, reactive aggregate, and free alkali are required to trigger the alkali–aggregate reaction in concrete. The AAR generally occurs after the concrete is hardened for a

few years, and it consequently causes the volume expansion of the concrete, deteriorating its microstructure and durability. Since the reaction sites are widely distributed in concrete components, once the AAR occurs, it is difficult to prevent or repair the damage caused by it, which consequently affects the quality and service life of the concrete. Therefore, the methods for detecting the potential of concrete aggregate for the alkali–silica reaction are particularly important [8-10].

So far combining petrographic analysis and the accelerated mortar bar test has been considered to be the most effective way to assess the ASR potential of the aggregate used for concrete preparation [11,12]. Petrographic analysis, employing a polarizing microscope, is generally based on the mineral composition of aggregate, and the alkali reactivity of aggregate is qualitatively evaluated by the active minerals and their content [13,14]. The accelerated mortar bar test quantifies the ASR potential of aggregate based on the expansion ratios of the mortar bars prepared therefrom [15]. The methodical testing techniques satisfy most of the requirements of concrete engineering which demands rapid and accurate test results.

Low-grade metamorphic rock (LMR) is a resource of manufactured sand which is widely distributed in China with large reserves. Using low-grade metamorphic rock as the aggregate for concrete can reduce costs and alleviate the crisis of the shortage of river sand resources. However, LMR aggregate has been found to have the certain potential for alkali reaction [1,16-19]. Thus, if it is directly used without any treatment, it causes the concrete to expand and crack, posing a serious threat to the safe operation of structures. As a result, it is very important and crucial to detect and prevent the alkali–aggregate reactivity of LMR aggregate. Currently, there are severe application problems with LMR, so it is necessary to quickly evaluate its alkali reactivity and effectively prevent and control its harmful reactions when it is used as the concrete aggregate. In this paper, using a series of experimental investigations, the ASR potential of the LMR and the related preventive measures were analyzed; the factors affecting the alkali–silica reaction were also evaluated.

2. EXPERIMENTAL PROGRAM

2.1 Materials

The 42.5 grade commercial Portland Ordinary cement (P.O 42.5) (from Guizhou Huishui Southwest Co., Ltd.) was used in this investigation. The properties of the cement are listed in Table 2.1, and its chemical composition is listed in Table 2.2. Fly ash from Qiandong thermal power plant has an alkali content of 1.37%. The chemical compositions are shown in Table 2.2. The LMR material was selected from different areas in southeast area of Guizhou Province, China. The source of the rock sample is listed in Table 2.3.

2.2 Experiment methods

The basic physical and mechanical properties of LMR aggregates were tested according to the relevant provisions and procedures in GB/T 14685-2011 [20]. A polarizing microscope was used for petrographic analysis to study the main mineral composition of LMRs, the rock is cut and ground into thin slices with a thickness of 0.03mm and the process was carried out according to the steps in JTG E42-2005 [21]. The accelerated mortar bar method was adopted to analyze the potential alkali activity of LMR aggregates. The alkali activity can be characterized by the expansion ratio of LMR specimens [22].

A scanning electron microscopy (SEM) was employed to observe the microstructure of the SMR mortar specimens. The LMR specimens were broken into a sample with a size of 5 mm in length, 5 mm in width and 2 mm in height. First, a conductive film was formed on the surface of the sample. Then, use conductive glue to stick it on the sample stage. Finally, place it in the scanning electron microscope for observation. The chemical composition of the specimens in micro-area was analyzed by Energy Dispersive Spectrometer (EDS), aiming to analyze whether the LMR specimen has an alkali aggregate reaction.

Table 2.1: The properties of cements.

Cement type	P.O 42.5
Normal consistency (%)	27.3
Alkali content (%)	0.44
Initial setting time (min)	130
Final setting time (min)	195
3 d strength (flexural/compressive)(MPa)	6.5/26.4
28 d strength (flexural/compressive) (MPa)	9.3/50.6

Table 2.2: The chemical composition of cement and fly ash (wt:%)

Chemical composition	P-O 42.5	Fly ash
SiO ₂	22.35	58.33
Al ₂ O ₃	5.76	30.61
Fe ₂ O ₃	4.10	3.77
Na ₂ O	0.14	1.04
K ₂ O	0.46	0.49
CaO	59.52	3.22
MgO	3.15	0.74
SO ₃	1.92	0.62
LOI	2.36	1.13
Alkali content (Na ₂ O and K ₂ O)	0.44	1.36

Note: Na₂O and K₂O is calculated as (Na₂O wt% + 0.658xK₂Owt%).

Table 2.3: The LMR sample from the southeast ares of Guizhou Province

Number	Place of production
S1	Changzhai Quarry
S2	Pingyong Quarry
S3	Tashi Quarry
S4	Wuniaohu Quarry
S5	Yongle Quarry
S6	Kaidunya Quarry
S7	DaTang Town
S8	Nanpin Village
S9	Wuji Quarry
S10	Paipo Quarry
S11	Zhangao Village

3. RESULTS

3.1 Mechanical properties of LMR aggregates

To assess the influence of the compressive strength of the LMR on the mechanical properties of the aggregate, the mechanical properties of the low-grade metamorphic rocks from different southeast areas of Guizhou province were analyzed; the results are presented in Figures 3.1 and 3.2.

Figure 3.1 demonstrates that the uniaxial saturated compressive strength of the rocks from the different regions was quite discrete, and the saturated compressive strength of the rock in the vertical direction was very different from that of the rock in the horizontal direction. Moreover, the minimum saturated compressive strength of the rock was observed in group S1 (35.2 MPa) in the vertical direction. The rock of group S3 had the maximum saturated compressive strength of 179.6 MPa in the horizontal direction, while its saturated compressive strength in the vertical direction was only 70.9 MPa. Standards JTG/T F50-2011[23] and GB/T14685-2011 [20] stipulate that the compressive strength of rock aggregate in a saturated state should not be lower than 60 MPa both in the direction perpendicular to the rock bedding and in the direction parallel to the rock bedding. Therefore, apart from the specimens of groups S1 and S11 which do not fulfill the requirements in the horizontal direction of the outer bedding, all the other groups can satisfy the compressive strength requirements; thus, they can be used as concrete aggregate.

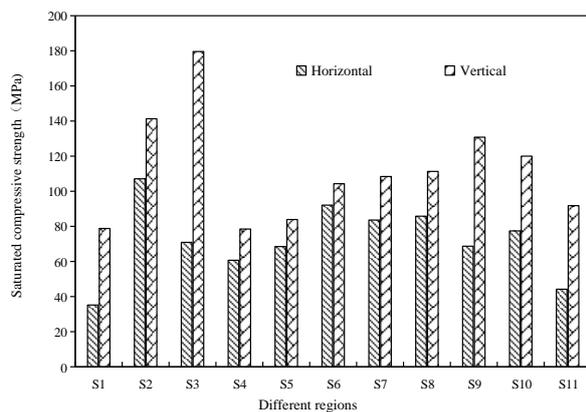


Figure 3.1: The saturated compressive strength of LMR rocks.

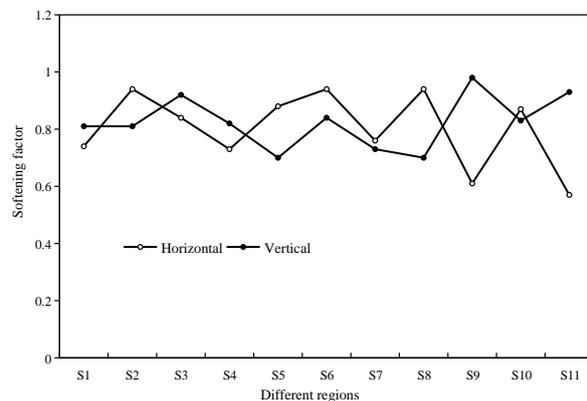


Figure 3.2: The softening coefficient test results of LMR rocks.

3.2 Lithofacies analysis

Lithofacies analysis is generally used for visually observing the mineral composition of aggregates and screening them in actual engineering projects [24]. Thus, using various optical microscopes, lithofacies analysis was conducted on the rock aggregate to determine whether it has the potential for the alkali–aggregate reaction.

Table 3.1 presents the lithology of the rocks in the different regions. All the metamorphic rocks in the various regions contained quartz acting as a binder [25]. The rocks in group S2 were largely clay minerals with a muddy crystalline structure, which contains partially recrystallized sericite and dolomite that is distributed in strips or clumps. Terrigenous debris dissolution was observed in group S5, and it was intermingled with the sericite and was partially distributed in agglomerates. The rocks in group S8 contained recrystallized sericite and terrigenous debris, and calcite filled the voids of the rocks. Therefore, it can be seen that all the rocks in the different regions are the siliceous slate and metamorphic sandstone, and they are largely composed of clay minerals, sericite, quartz, feldspar, dolomite, and calcite. The alkali-carbonate reactive minerals were not found in all the rock samples, but microcrystalline quartz was observed in all the rocks [17]. Therefore, these rocks have the potential for the alkali–silica reaction and are identified as the rock aggregate with a slow expansion reaction.

Table 3.1: The petrographic analysis results of LMR rocks.

Number	Lithofacies analysis			Lithology
	Alkali active component	Mineral grain size (μm)	Content (vol%)	
S1	Microcrystalline quartz	/	50	Metamorphic mudstone - sandstone
S2	Dolomite	<30	<5	Siliceous silty clay slates
	Hidden-microcrystalline quartz	/	10	
S3	Dolomite	>30	<5	Silty clay slates
	Microcrystalline quartz	/	15	
S4	Hidden-microcrystalline quartz	/	15	Sandy sericite clay slates
S5	Dolomite	>50	<10	Sandy sericite clay slates(Siliceous powder)
	Hidden-microcrystalline quartz	/	20	
S6	Hidden-microcrystalline quartz	/	30	Silty siliceous clay slates
	Dolomite	>30	<1	
S7	Hidden-microcrystalline quartz	/	50	Metamorphic quartz siltstones
S8	Microcrystalline quartz	/	30	Powder-Sandy clay slates
S9	Microcrystalline quartz	/	20	Silty clay slates
S10	Microcrystalline quartz	/	5	Powder-Sandy clay slates
	Dolomite	<100	<5	
S11	Microcrystalline quartz		<5	Sericite clay slates

3.3 Alkali activity of LMR aggregates

The accelerated mortar bar test is an important method for determining the alkali reactivity of aggregate [15]; also, it has a short test cycle and is highly sensitive to characterizing the ARS. Therefore, the alkali-aggregate reactivity of the LMR aggregates from the different regions was analyzed by using the accelerated mortar bar test.

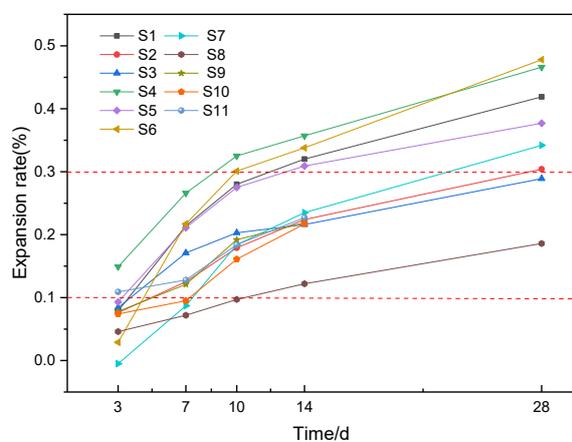


Figure 3.3: The alkali activity test results of LMR aggregates.

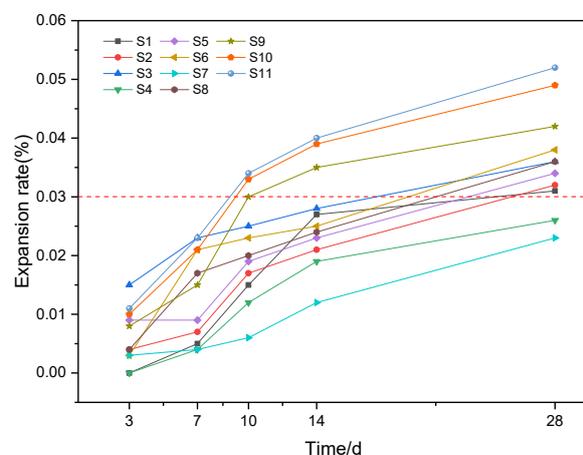


Figure 3.4: The inhibition results of LMR aggregates by fly ash.

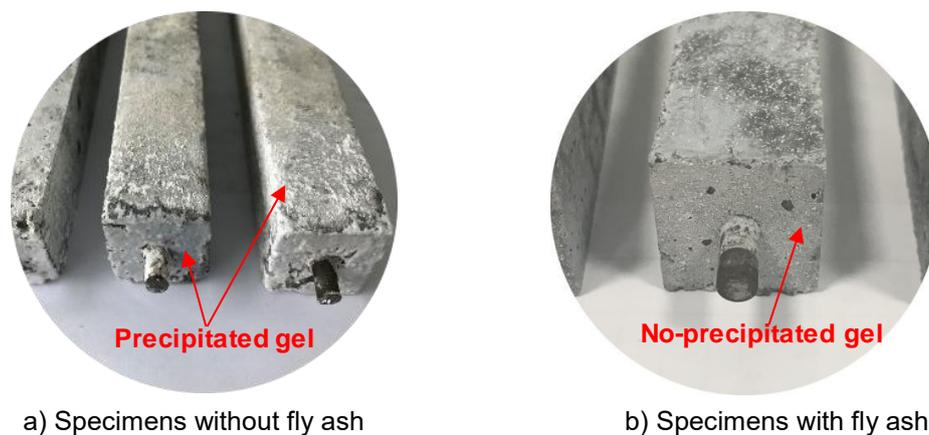


Figure 3.5: The surface morphology of different mortar specimens.

Figure 3.3 illustrates the alkali–aggregate reactivity of the LMR aggregate samples from the different regions using the accelerated mortar bar test. It can be seen that the 14-day expansion ratios of the untreated LMR aggregate samples obtained from the different southeast areas of Guizhou province were all higher than 0.1%, indicating that all the LMR aggregate samples in these regions obviously have the potential for the ASR.

The curing period of concrete had a significant influence on the expansion ratio of the rock aggregate samples, and the expansion ratio of the rocks in all the groups increased as the curing period extended. However, there was no obvious linear relationship between the expansion ratio and the curing period. The expansion ratio of the rocks of all the groups grew faster during the curing period of 3-7 days, while the expansion ratio of the LMR aggregate samples showed slower growth during the curing period of 10-14 days. This revealed that the accelerated mortar bar method could expedite the reaction of the alkali with the aggregate, so it could scientifically evaluate the alkali reactivity of the aggregate with lower reactivity.

Figure 3.4 presents the inhibitory effect of the fly ash on the ASR of different LMR aggregate samples. The alkali reactivity of all the rock aggregate samples mixed with the fly ash was significantly reduced, and the restraining effect of adding 30% of the fly ash was very obvious. The 14-day expansion ratios of the specimens prepared with the rocks of groups S1–S8 did not exceed 0.03%. The concentration of the alkali ions of the slurry pore solution was reduced when the fly ash was incorporated into the aggregate, which is attributed to the fact that the active component in the fly ash can react with the alkali ions, thereby consuming them [26,27]; therefore, fly ash can effectively reduce the effective alkali content of the aggregate required for the AAR and can play a suppressing role in the alkali–aggregate reaction [28]. However, the restraining effect of the fly ash on the AAR of the different LMR aggregate samples was obviously different. The fly ash had the highest restraining effect on the rocks of group S7 and the least preventive effect on the rocks of group S11 since the alkali active compositions and content of each group of the rock aggregates were different. Compared with the specimens without the fly ash (see Figure 3.4), the expansion ratio of the specimens mixed with the fly ash rapidly increased during the curing period of 3-7 days; however, it rose insignificantly during the curing period of 7-14 days. The results showed that the restraining effect of the fly ash on the alkali–aggregate reaction was chiefly concentrated in a curing period of longer than 7 days, which was closely related to the reactivity of fly ash. The main reason is that fly ash can react with $\text{Ca}(\text{OH})_2$ in the concrete specimens at a later stage, which consumes the alkali required for the AAR and reduces the concentration of the alkali ions in the slurry, thereby playing a suppressing role in the alkali–aggregate reaction.

The surface morphology of the mortar specimens with and without the fly ash is illustrated in Figure 3.5. The surface of the mortar specimens without the fly ash is partially covered with precipitated gel as shown in Figure 3.5 (a); moreover, the gel precipitation continued as the curing period of the concrete extended, and microcracks appeared in the specimen, which indicated that the specimen sustained the AAR. However, the gel was not significantly observed on the surface of the mortar

sample mixed with the fly ash as shown in Figure 3.5 (b), and no cracks were formed in the specimen due to the restraining effect of the fly ash.

3.4 Alkali activity of LMR aggregates

The main components of the concrete were cement, mineral admixtures, aggregate, and water; also, the cement and the mineral admixtures with a certain amount of free alkali have an important impact on the AAR of concrete [29-31]. Therefore, the influence of the alkali content of the cement and the mineral admixtures should be investigated comprehensively when the experiment on the alkali-aggregate reaction is carried out. In order to explore the effects of the alkali content of the cement, the fly ash, and the aggregate on the AAR, the effect of the total alkali content on the alkali-aggregate reaction of the concrete with the fly ash or without the fly ash was investigated using the accelerated mortar bar method. The design mix proportions are listed in Table 3.2, and the results are depicted in Figures 3.6 and 3.7.

Table 3.2: The influence of total alkali content on the AAR.

Number	Cement (wt%)	Fly ash (%)	Alkali content (%)	Fine aggregates
H1	100	0	0.6	S8
H2	100	0	0.8	S8
H3	100	0	1.0	S8
H4	100	0	1.2	S8
H5	100	0	1.4	S8
H6	70	30	0.6	S8
H7	70	30	0.8	S8
H8	70	30	1.0	S8
H9	70	30	1.2	S8
H10	70	30	1.4	S8

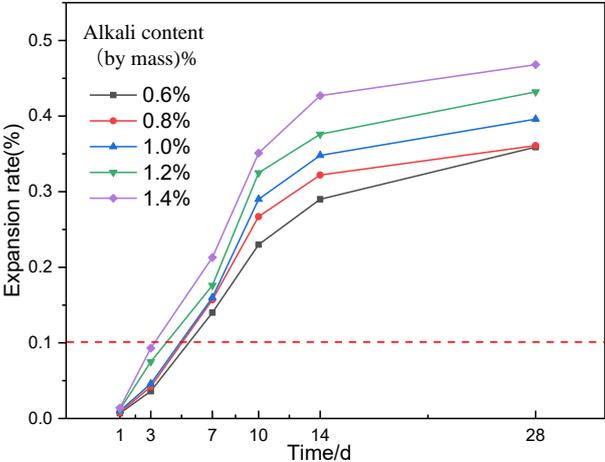


Figure 3.6: The influence of the total alkali contents on AAR.

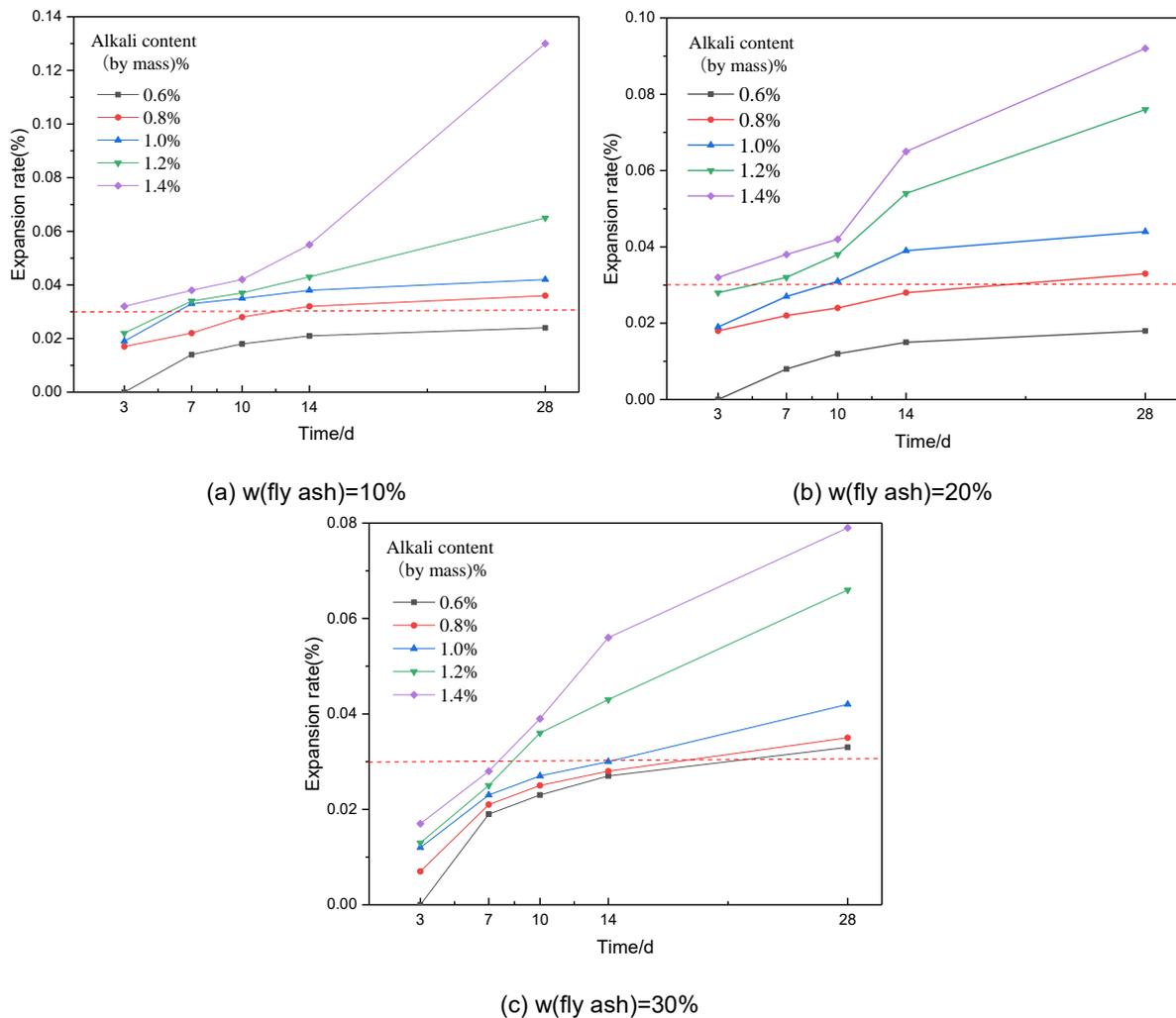


Figure 3.7: Influence of fly ash on specimens with different total alkali contents.

According to Figure 3.6, the expansion ratio of the mortar groups with different amounts of alkali increased as the curing period of the concrete extended. The 14-day expansion ratios of all the specimens were larger than 0.1%, which did not fulfill the requirements of the standard. The expansion ratios of the specimens containing different amounts of alkali increased rapidly during the early curing period of 1-10 days; nonetheless, it continued to rise at a much slower pace as the curing period further extended. When the total alkali content of the specimen was 1.4%, the expansion ratio of the concrete was almost proportional to its curing period, which revealed that the larger the alkali content of the concrete is, the faster the alkali-aggregate reaction, the longer the reaction cycle, and the larger the expansion ratio become.

Figure 3.7 demonstrates that the expansion ratios of the groups with the fly ash were significantly reduced. As can be seen in Figure 3.7 (a), when the fly ash content of the concrete was 10%, the 14-day expansion ratio of the specimen with an alkali content of less than 0.6% was smaller than 0.03%, which implies that the ASR is effectively inhibited. However, the 14-day expansion ratio of the specimen with an alkali content of higher than 0.6% was larger than 0.03%, which indicates that the ASR is not well inhibited. When 20% fly ash was added to the concrete (see Figure 3.7 (b)), the effective inhibition of the ASR raised the limit on the alkali content of the concrete to 0.8%. Figure 3.7 (c) reveals that enlarging the fly ash content of the concrete to 30% caused the 14-day expansion ratio of the specimen with an alkali content of 1.0% to be smaller than 0.03%, which indicates that the ASR is effectively inhibited for the specimens with an alkali content of lower than 1.0%.

When the alkali content of the concrete was less than 1.0%, the expansion of the specimens largely occurred during the curing period of 3-10 days. The growth of the expansion ratio of the specimens

slowed down significantly at curing periods longer than 10 days, and the 14-day expansion ratio of the specimens was less than 0.03%. To effectively inhibit the ASR, the optimal total alkali content of the concrete should be controlled to be lower than 1.0%.

3.5 Microstructure

Different alkali-reactive minerals in the aggregate can lead to various types of AAR, which are chiefly divided into the ASR and the ACR. The alkali-aggregate reaction of the low-grade metamorphic rock was mostly the ASR due to containing a large amount of SiO_2 . In essence, the ASR is the expansion reaction of active SiO_2 in aggregate with the alkali in the concrete pore solution, which forms honeycomb alkali-silica gel with a great water-swelling capability. As the stress generated by the expansion exceeds the strength of the concrete, it causes cracking damage to the concrete structure.

In this paper, scanning electron microscopy (SEM) was employed to analyze the morphology of the interfacial transition zone (ITZ) of the mortar specimen with the LMR aggregate, and the energy dispersive spectroscopy (EDS) was used to examine the composition of the ITZ so as to evaluate whether the specimen sustained the AAR and to identify its reaction state. In order to intuitively analyze and compare the AAR, the specimens of group S4 with the largest expansion ratio and those of group S8 with the smallest expansion ratio were selected as the test subjects.

Figure 3.8 presents the SEM-EDS analysis results of the specimens of group S8. The aggregate and the cement paste were relatively compact in the mortar specimens of group S8, and the aggregate surface was clean; also, no honeycomb alkali-silica gel was formed, as can be seen in Figures 3.8 (a) and 3.8 (b). It is obvious that there was no element enrichment in the ITZ (Figure 3.10 (a)), which indicated that no AAR occurred in the mortars of group S8.

However, alkali-silica gel was formed on the aggregate surface of the mortar of group S4 (Figure 3.9 (a)). Moreover, cracks were formed in the mortar of group S4 due to the water swelling of the alkali-silica gel, and its structure was destroyed (Figure 3.9 (b)). We also found that the Si and Na content of the ITZ increased and was significantly higher than the Ca content, so there was element enrichment in the ITZ (Figure 3.10 (b)). The results showed that the AAR occurred in the concrete of group S4, and it could be visually analyzed whether the sample had an alkali-aggregate reaction; the reaction state could also be examined by the SEM-EDS analysis.

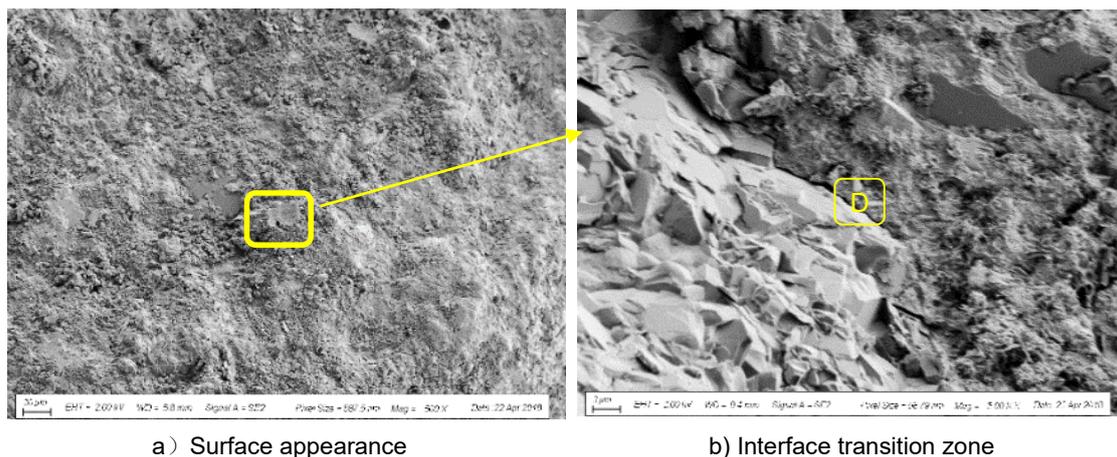


Figure 3.8: Sample microscopic analysis of S8 mortars

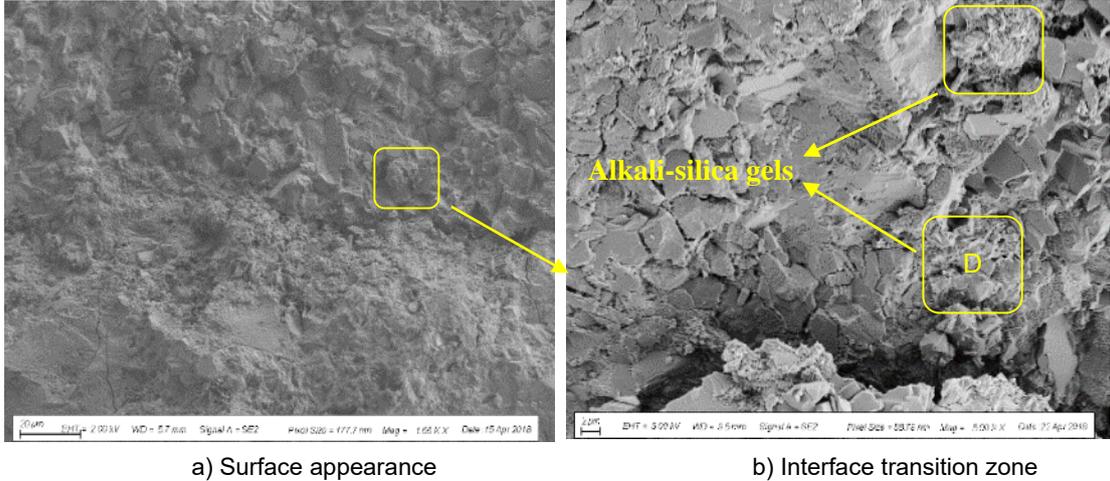


Figure 3.9: Sample microscopic analysis of S4 mortars

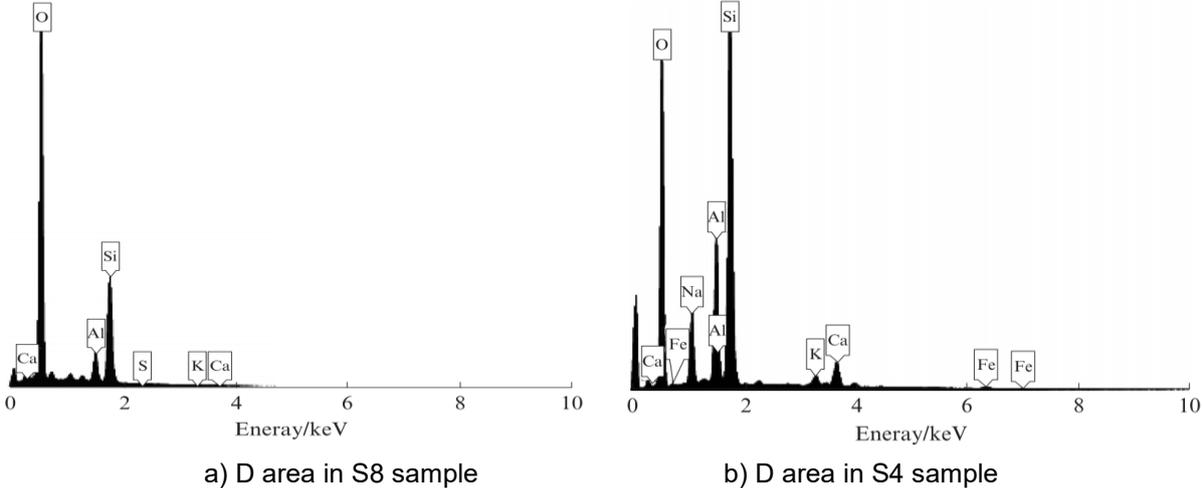


Figure 3.10: The EDS analysis of mortar specimens

3.6 Prevention measures of concretes with LMR aggregates

In practical engineering, it is necessary to take preventative measures to restrain the AAR from damaging the concrete structure containing the LMR aggregate [17,32,33]. Therefore, it is suggested that restraining measures should be taken by analyzing the alkali reactivity of the metamorphic rock aggregate, the total alkali content of the concrete, the mineral admixtures, the stone powder content of the concrete, and the service conditions of the concrete. The lithofacies analysis and the accelerated mortar bar method should be employed to comprehensively investigate the alkali reactivity of the low-grade metamorphic rock aggregate so that the uncertainty of a single test method is avoided. A significant restraining effect can be produced by incorporating mineral blends such as fly ash (at a recommended content of higher than 30%) into the concrete containing the LMR aggregate. In order to prevent the AAR from impacting on the service life of the concrete, it is necessary to strictly control the total alkali content of the concrete containing LMR to be lower than 1.0%. In addition, controlling the stone powder content of the LMR can reduce the probability of the occurrence of the AAR. The engineering applications of the LMR aggregate should also be selected according to the service conditions of the concrete. Furthermore, it is essential to observe and analyze the long-term influence of the alkali-aggregate reaction on the concrete containing LMR aggregate when it is applied in the actual engineering projects.

4. CONCLUSIONS

From the findings of the current paper, the following conclusions can be drawn:

- 1) The uniaxial saturated compressive strength of the low-grade metamorphic rock in the southeast area of Guizhou province was quite discrete, and all the rocks contained a certain amount of microcrystalline quartz; thus, they were identified as silicate rock with the potential for the alkali–silica reaction.
- 2) In the accelerated mortar bar tests, the 14-day expansion ratios of the untreated concrete specimens were all larger than 0.1%, so they all had the potential for the alkali–aggregate reaction. The restraining effect of the fly ash on the AAR was obvious, and the 14-day expansion ratios of some specimens containing the fly ash did not exceed 0.03%.
- 3) The total alkali content of the concrete had an important influence on the AAR, and the optimum alkali content of the concrete should be controlled to be lower than 1.0% so as to prevent the alkali–aggregate reaction.
- 4) The SEM–EDS results revealed alkali–silica gel and cracks in the interfacial transition zone of mortar S8 with the alkali–aggregate reaction; moreover, it was found that the Si and Na content of the ITZ increased and was significantly higher than the Ca content, indicating that the interfacial transition zone was enriched with these elements.
- 5) When the LMR aggregate is applied in actual engineering, it is suggested that preventive measures should be taken by analyzing the alkali reactivity of the LMR aggregate, the total alkali content of the concrete, the mineral admixtures, the stone powder content of the concrete, and the service conditions of the concrete.

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