



Effects of fly ash and lithium compounds on the water-soluble alkali and lithium content of cement specimens



Wei Chien Wang*

Department of Civil Engineering, Chung Yuan Christian University, Chung Li 32023, Taiwan, ROC

HIGHLIGHTS

- The alkali content of fly ash is originally non-water-soluble.
- The alkali content of fly ash gradually becomes water-soluble during the cement hydration process.
- Lithium compounds could increase the water-soluble alkali concentration in cement specimen.
- ASR inhibition performance of LiNO_3 is than that of LiOH addition.

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ABSTRACT

This study uses fly ash to replace partial cement. NaOH is used to increase alkali content of cement to 1.0% $\text{Na}_2\text{O}_{\text{eq}}$. LiOH and LiNO_3 are added separately to obtain several $\text{Li}/(\text{Na} + \text{K})$ molar ratios to fabricate cement specimens cured at 38 °C in a 100% relative humidity environment. This study aims to observe the diachronic changes of water-soluble alkali and lithium content in specimens to understand the potential damaging effect of alkali–silica reaction by adding fly ash and lithium compounds simultaneously. Results indicate that the alkali content of fly ash is originally non-water-soluble, but it gradually becomes water-soluble during the hydration process. When lithium compounds are not added, an increase in cement alkali content increases both the amount and the ratio of water-soluble alkali in the specimens. Given the same curing time, when lithium compounds are added to cement, the ratio of water-soluble lithium content is higher than that of water-soluble alkali content. Therefore, water-soluble $\text{Li}/(\text{Na} + \text{K})$ molar ratio becomes higher than the original value. Furthermore, LiNO_3 addition exhibits better performance in increasing water-soluble $\text{Li}/(\text{Na} + \text{K})$ molar ratio compared with LiOH addition.

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1. Introduction

When pozzolanic materials are added to concrete, these materials consume OH^- in the pore solution and react with $\text{Ca}(\text{OH})_2$ to generate C–S–H gels with low Ca/Si ratio. The specific surface area of this gel is larger, absorbs more alkali metal ions, and consumes the alkali content of concrete, which is the main mechanism of pozzolanic materials in inhibiting alkali–silica reaction (ASR) [1,2]. When a pozzolanic reaction consumes the readily soluble calcium hydroxide in concrete and generates C–S–H gel, the reaction reduces concrete porosity and achieves pore miniaturization, which can resist the intrusion of foreign alkali or moisture content [3]. Pozzolanic materials can also increase the strength of concrete in later stages and enhance the restraining force of concrete to reduce expansion [4]. These reasons are conducive to inhibiting ASR expansion.

Majority of previous studies that dealt with the effect of replacing a portion of cement with pozzolanic materials to inhibit ASR have used experiment results on expansion to prove effectiveness. However, studies on the mechanism of expansion inhibition have focused mostly on investigating the relationship between the amount of expansion and the material composition or alkali concentration in the pore solution. Methods for analyzing alkali concentration in the pore solution mostly involve curing the specimens in a sealed environment from 23 °C to 40 °C for no longer than 2 years. The specimens are removed at the appropriate age. The pore solution would then be extracted through high-pressure approaches to analyze the OH^- concentration of the solution; Na^+ and K^+ concentrations would also be examined in small parts by observing the influence of type, composition, and amount of material that replaces cement on the alkali concentration in the pore solution and on the amount of expansion to analyze the mechanism for the inhibition of ASR expansion [2,5–8]. For example, fly ash and blast-furnace slag contain a certain amount of alkali, some of which may even be higher than the alkali content of the cement

* Tel.: +886 921700501.

E-mail address: weichien@cycu.edu.tw

to be replaced. However, majority of the literature shows that the main mechanism in reducing ASR expansion involves alkali concentration reduction in the pore solution of concrete, where the amount of reduction generally increases with the increase in the amount of replacement. Research has confirmed the direct relationship between the expansion caused by ASR and the alkali concentration level in the pore solution. The OH^- concentration of cement can probably be balanced with $\text{Na}^+ + \text{K}^+$ concentration [5–10].

Lithium compounds can function as ASR inhibitors. Durand [11] proposed that the minimum effective $\text{Li}/(\text{Na} + \text{K})$ molar ratio for inhibition should be related to the types of lithium compounds to be added, which are 0.6, 0.8, and 0.9 for LiOH , LiNO_3 , and LiCl , respectively. Thomas et al. [12] integrated his own experimental data with those obtained by other researchers, and proposed that the effective $\text{Li}/(\text{Na} + \text{K})$ molar ratio is 0.74 for LiNO_3 ; in addition, a higher effective $\text{Li}/(\text{Na} + \text{K})$ molar ratio of 0.85 is necessary for $\text{LiOH}\cdot\text{H}_2\text{O}$. When lithium compounds are added to concrete, lithium ions and reactive aggregate with breaking bonds generate a non-expanded or slightly expanded lithium–silica gel on the surface of the reactive aggregate to prevent the binding reaction between the reactive aggregate and Na^+ and K^+ , as well as to mitigate ASR effectiveness [13–17].

When pozzolanic materials are used to replace a portion of cement, part of the alkali content is integrated into the hydration products during the hydration process; the others freely exist in the pore solution. If reactive aggregates are present, part of the free alkali content combines with those reactive aggregates. If the concrete does not contain reactive aggregates, the free alkali content is not consumed by the ASR, and the ratio of the alkali content in the pore solution or hydration products is affected by pozzolanic material composition [6]. Numerous studies have been conducted on the mechanism of ASR inhibition; for example, using fly ash and blast-furnace slag to replace a portion of cement. The focus has mostly been on the effect of cement replacement on alkali concentration in the pore solution [5–10]. However, few relevant studies on the addition of both pozzolanic materials and lithium compounds to specimens are available; the same holds true on the investigation of the effect of alkali metal ion concentration in the pore solution to inhibit ASR.

The current study uses water-soluble alkali content of cement as the object of analysis. This finding is in contrast to previous studies that extracted the pore solution to analyze alkali concentration. Adding lithium compounds while using fly ash to replace a portion of cement allows observation of the diachronic changes of water-soluble alkali and lithium content of specimens; this procedure is performed to understand the effect of adding fly ash and lithium compounds simultaneously on a potential amount of water-soluble alkali and lithium that is involved in the reactive aggregate reaction. Another objective is to further understand the effect of different types of lithium compounds on the amount of water-soluble alkali and lithium content in specimens.

2. Experiment

Series 1: Cement specimens do not increase alkalinity. Class F fly ash was used to replace 0%, 10%, 15%, and 20% of the cement weight. The amount of replacement was within the common range for practical applications.

Series 2: Class F fly ash was used to replace 0%, 10%, 15%, and 20% of the cement weight. NaOH was used to increase additional alkali content in the specimens to 1.0% $\text{Na}_2\text{O}_{\text{eq}}$.

Series 3: Class F fly ash was used to replace 0%, 10%, 15%, and 20% of the cement weight. NaOH was used to increase additional alkali content to 1.0% $\text{Na}_2\text{O}_{\text{eq}}$. LiOH was also added to 0.6, 0.9, or 1.2 $\text{Li}/(\text{Na} + \text{K})$ molar ratios.

Series 4: Class F fly ash was used to replace 0%, 10%, 15%, and 20% of the cement weight. NaOH was used to increase additional alkali content to 1.0% $\text{Na}_2\text{O}_{\text{eq}}$. LiNO_3 was also added to 0.6, 0.9, or 1.2 $\text{Li}/(\text{Na} + \text{K})$ molar ratios.

The water/cement ratios of the aforementioned cement specimens were all 0.4. The specimens were demolded 1 d after fabrication, and were cured for 1 d at 23 °C in a 100% relative humidity (RH) environment. The specimens continued to be cured at 38 °C and 100% RH curing room for 1, 14, 28, 56, and 90 d based on the curing environment in the ASTM C227 test. Ninety-day curing treatment was based on the ASTM C227 test, in which the ASR reactivity of aggregates can be assessed using the results within 90 d.

After curing, the specimens were processed through AASHTO T260 experimental process. The specimen was first ground into powder. Afterward, 3 g of the ground powder was passed through a #50 sieve into a stainless steel cup. De-ionized water was added, and a magnetic stirring hot plate was used to heat the water until boiling. The water was boiled for 5 min. The power supply was then turned off, and the solution was left to stand for 24 h. The vacuum filtration method was used the following day to obtain a clear solution. Ion chromatography (IC) was then used to analyze Li^+ , Na^+ , and K^+ contents of the solution. The derived values of the Na^+ and K^+ contents can be used to calculate the $\text{Na}_2\text{O}_{\text{eq}}$ value of water-soluble alkali. The percentage of water-soluble alkali and lithium in the total alkali and lithium content could also be calculated.

This study further aims to observe the alkali release in cement and fly ash during hydration process using different amounts of fly ash to replace cement while simultaneously adding lithium compounds to cement specimens. The alkali concentration in the pore solution was not calculated. The cement consumes water in the cement hydration process, and the hydration products account for the space taken by the original water volume. Therefore, the water content of specimens with different curing time needs to be considered in calculating alkali and lithium ion concentrations in the pore solution from the water-soluble alkali and lithium content. However, this process is not included in the scope of this study.

The composition analyses of cement and fly ash are shown in Table 1. The total alkali contents are 0.50% and 1.21% $\text{Na}_2\text{O}_{\text{eq}}$, respectively. During the experiments, water-soluble methods revealed that the total instantly water-soluble alkali contents are 0.290% and 0.021% $\text{Na}_2\text{O}_{\text{eq}}$, whereas the instantly soluble alkali content accounts for 58.0% and 1.7%, respectively, of the total amount.

3. Results and discussion

Table 2 lists the analysis results (in percentage) of the water-soluble alkali and lithium content of the experimental specimens. The water-soluble $\text{Li}/(\text{Na} + \text{K})$ molar ratios for different curing times are calculated. Table 2 lists the water-soluble $\text{Li}/(\text{Na} + \text{K})$ molar ratios after 90 d curing. Each value is an average of the results obtained from duplicate experiments.

3.1. Treatment without additional alkali

Fig. 1 shows the diachronic change in the percentage of water-soluble alkali content in specimens that used fly ash to replace cement without additional alkali. Calculation was performed according to the AASHTO T260 test. The amount of water-soluble alkali in the specimens cured for 1 d to 90 d was examined, and then divided by the total alkali content of the specimens.

For the pure cement paste, after curing for 1, 14, 28, 56, and 90 d, the water-soluble alkali contents of the specimens are 0.32%, 0.30%, 0.22%, 0.18%, and 0.10% $\text{Na}_2\text{O}_{\text{eq}}$, respectively (Table 3). The water-soluble alkali content decreases with increased curing time. Given that the total alkali contents of the cement used in the experiments are all 0.50% $\text{Na}_2\text{O}_{\text{eq}}$, the water-soluble alkali content accounts for 64.2%, 59.5%, 44.7%, 35.2%, and 20.3% of the total alkali content (Table 2) after curing for 1, 14, 28, 56, and 90 d. Of the total alkali contents, 35.8%, 40.5%, 55.3%, 64.8%, and 79.7% are consolidated into the cement hydration products. In this study, cement paste fabricated with low-alkali cement and contains 0.5% $\text{Na}_2\text{O}_{\text{eq}}$ total alkali content was used. The water-soluble alkali content of the cement paste becomes 0.10% $\text{Na}_2\text{O}_{\text{eq}}$ after 90 d curing at 38 °C, which accounts for 20.3% of the total alkali content. Given that low-alkali cement is commonly used in fresh concrete to inhibit the ASR problem, this amount is not sufficient to trigger reactive aggregates and induce the ASR problem.

For specimens that used fly ash to replace 10% of the cement weight, the water-soluble alkali content of specimens becomes 0.29%, 0.35%, 0.32%, 0.30%, and 0.20% $\text{Na}_2\text{O}_{\text{eq}}$ after curing for 1, 14, 28, 56, and 90 d, respectively (Table 3). The total alkali content

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