Experimental Identification of Alkali-Aggregate Reaction in Concrete

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ABSTRACT

Several advanced and time-consuming methodologies have been developed to detect Alkali-Silica Reaction (ASR) in suspected structures. The main objective of this research study was to identify a reliable experimental procedure for detecting ASR in existing concrete. A simple staining solution is used here to detect ASR in concrete specimens. The staining reagent employed here is Sodium Cobaltinitrite, which is used in the Los Alamos staining method to detect ASR. Sodium Cobaltinitrite can identify potassium-rich ASR gel by staining it yellow for rapid field screening purposes. Reactive and control concrete specimens were cast to get some experience with this test and to verify whether this test can be used in a suspected concrete structure. Waste white soda-lime glass aggregate was used to cast reactive concrete specimens, whereas natural coarse aggregates were used to cast non-reactive concrete specimens. Testing was carried out in two batches. Each batch consisted of six reactive and six control concrete specimens which were cured in the above-mentioned solutions. The first batch was examined after 44-days and the second batch was tested after 60-days of casting. Results of this test showed that reactive concrete specimens cast using glass displayed yellow stains as expected, demonstrating the presence of potassium-rich ASR gel on the concrete surface. Employing NaOH as a curing medium had accelerated ASR. There is a limitation in the method when utilizing KOH as a curing agent. It is concluded that Sodium Cobaltinitrite can be used as a method for rapid identification of ASR in the preliminary stages of experimental identification of the alkali-aggregate reaction in an existing concrete structure.

KEYWORDS: *Alkali-Silica Reaction, Sodium Cobaltinitrite, Aggregates, Waste glass, Cement, Potassium hydroxide, Sodium hydroxide*

1 INTRODUCTION

The first alleged case of Alkali-Silica Reaction (ASR) was discovered when the premature decay of many concrete structures in California, USA were investigated (Stanton, 1940). This investigation was done by testing the aggregates and cement used in the concrete in a laboratory. Stark (1991) discussed the macroscopic symptoms of Alkali-Silica Reaction. Often the first noticeable indication of ASR is randomly directed cracking at the surface, also known as map cracking due to the irregular pattern of the cracks (Stark, 1991). These cracks progress over time by interconnecting and expanding the cracked surface. This is a slow process that can take many years to reach noticeable levels.

The main factors that influence ASR are water, temperature, reactive aggregate forms, aggregate size, alkali content, stress level, and also, air entrainment and porosity. Alkalis react with the silica in susceptible aggregates to form a gel. This silica gel swells only when moisture is present. Swelling of the gel is the cause of cracking which can lead to degradation of concrete quality and properties. Moisture content plays a major role in the progress of ASR.

A similar process involving carbonates in aggregates has also been identified. It is called an alkali carbonate reaction (ACR). Together, ASR and ACR are referred to as alkali-aggregate reactions (AAR). Of these two ASR is by far the more common form.

One of the most important issues related to AAR is the detection of Alkali Aggregate Reaction in existing concrete structures. Several techniques for detecting the presence of products of the alkali-aggregate reaction in concrete have been introduced. The majority of these techniques are based on the microscopic examination of affected concrete. Because of the complexities of these microscopic examination methodologies, two simpler staining test methods, viz. Uranyl Acetate test and Los Alamos test, also have been proposed to identify potential AAR by examining the staining pattern produced by certain chemicals on the surface of suspected concrete. These staining tests have the advantage of being simple procedures that can be implemented with basic facilities even in the field.

In Sri Lanka, like in many other countries, certain important concrete structures are suspected to be suffering from ASR. The objective of the present work was to explore the feasibility of using one of these staining tests – the Los Alamos test – as a rapid screening procedure for the detection of possible ASR in the Sri Lankan context.

2 LITERATURE REVIEW

All alkali gels are hygroscopic, which means they absorb moisture. As they absorb moisture, they tend to expand. This can cause damage to the surrounding concrete or mortar if the gel exerts pressure while expanding (Nilsson, 1983). There are two possible mechanisms for concrete in a structure to get exposed to moisture. The first is the structure's exposure to the ambient humid atmosphere, which does not directly involve liquid water. The second involves structures that are actively exposed to liquid water. This refers to the vulnerability of structures to rainfall or river streams with cyclic fluctuations of water levels (Steffens, Li, & Coussy, 2003).

Environmental factors such as temperature have a significant impact on the Alkali-Silica Reaction (ASR), which induces the expansion of concrete and harms long-term durability. An exposure experiment was conducted by Fournier et al. (2009) by using similar types of concrete blocks taken from various exposure locations. It was observed that the rate of ASR expansion after 3–4 years of exposure increased in warmer regions than under cooler temperature conditions (Fournier et al., 2009). Regarding the effect of temperature on the expansion rate and the final expansion, previous works have demonstrated contradictory evidence. According to a study done by Larive (1997), the temperature does not affect the final expansion but has a significant impact on expansion kinetics. Furthermore, the temperature dependence of the kinetics of ASR expansion follows Arrhenius' rule (Larive, 1997).

As not all aggregates are susceptible to ASR, the severity of the symptoms is frequently dependent on the type of aggregate present. Though many rock types have silica as a mineral, ASR is generated only by some siliceous aggregates. Mineral quartz does not display ASR signs as it is stable but Opal is a reactive mineral. Opal, volcanic glass, cristobalite, strained quartz, and various types of cryptocrystalline, microcrystalline, and tridymite are examples of reactive minerals (Thomas et al., 2011). Many variables, such as the modulus and silicate and alkali content, must be investigated concurrently to comprehend the activity of the materials under consideration. The majority of prior experiments for alkali reactive aggregates employed quartz glass and opal (Shi et al. 2015). Waste glass can be utilized as an aggregate in concrete in the form of fine or coarse aggregates. Earlier studies about the use of glass aggregates in mortars indicate that the reactivity of glass is affected by the size of the particles, with finer particles of glass aggregate resulting in lower ASR expansions. Increasing the surface area of glass particles can accelerate the ASR (Rajabipour, Maraghechi, & Fischer, 2010).

The size of the aggregates influences the rate of reaction as well as the expansion of concrete. Hobbs & Gutteridge (1979) investigated the effect of the size of aggregates by using opaline rocks as the aggregate type with fractions ranging from 150 μm to 4.72 mm. They concluded that expansion increases as opaline aggregate particle size decreases (Hobbs & Gutteridge, 1979).

Multon and Toutlemonde (2006) performed and analysed tests on concrete samples exposed to various forms of stress along three different directions. The calculated volumetric expansions of ASR samples that are damaged under 3D phases of stress are minimized, however, the classical analysis cannot conclude actual ASR-induced strains. They carried out this experiment to analyse the actual ASR-imposed strains. Deformations for these ASR-induced expansions are more representative of the ASR imposed expansions on weaker and mortar aged structures and enable volume expansions to be evaluated for the nine various states of stresses (1D and/or 3D). The quantity of axial ASR imposed strain added to twice the radial-imposed strain is equivalent to the volumetric applied strain for the cylinders. The volumetric applied strain was computed on the 400th day to assess the results depending on the level of stress (Multon & Toutlemonde, 2006).

When the concrete matrix contains well-distributed pores, the ASR-induced expansion is substantially reduced. Even though the reaction rate may be unaffected, the pores relieve the swelling of the gel, resulting in lower concrete expansion (Hobbs, 1988). The expansion of both air-entrained and plain mortar bars was measured by Jensen et al. (1984). A decrease of 40% in expansion was found when 4% air was added into the mortar specimen (Jensen et al. 1984).

ASR exhibits similar microscopic behaviour to other deterioration mechanisms including D-Cracking and freeze-thaw. A detailed examination of microscopic characteristics can be used to identify the basic degradation process and to evaluate the existence of ASR gel in a concrete structure. Some smooth, lapped concrete surfaces with reaction rims over coarse aggregate particles, microcracks across particles, and white in colour products of ASR gel could be seen. Verified gel deposits can prove that ASR has occurred (Stark, 1991).

Staining fractured concrete surfaces using Uranyl Acetate solution is another approach for detecting ASR. According to Stark (1991), this technique could be used to identify ASR gel on any concrete surface. Using this approach on formed and sawed concrete surfaces that have been exposed for years has revealed that this does not always produce satisfactory results with such concretes. It is better to use this method on fresh and newly formed concrete surfaces. Some instances for newly formed concrete surfaces include fresh fractures, cores, and sawed surfaces. The existence of ASR gel will be confirmed by a yellowish-green fluorescent glow under ultraviolet (UV) light (Stark, 1991).

Another potential staining technique is the geochemical method for the identification of ASR Gel, which was developed in the Los Alamos National Laboratory and often referred to as the dual staining process or Los Alamos Staining Method (Guthrie & Carey, 1997). It relies on the composition of ASR gel and one of its properties which is the capacity of a fluid to exchange cations. The stained concrete surface can be viewed in normal lighting conditions. In the treatment procedure, two different types of reagents are used. The first is a saturated aqueous solution of sodium cobaltinitrite [$Na_3Co\ (NO_2)_6$] also known as Sodium hexanitrocobaltate(III), that reacts with soluble potassium and forms a yellowish precipitate staining potassium-rich ASR gel. The other aqueous solution used in this procedure is saturated Rhodamine B solution. It can react with ASR components other than potassium-rich ASR and stain the concrete in a pinkish colour. Calcium ions contained in the ASR gel are identified using this. This dual staining method enables researchers to test concrete for ASR in-laboratory and also in the field like the uranyl acetate approach. However, unlike the uranyl acetate approach, this does not involve the use of Ultraviolet light in a light-tight setting. More significantly, it does not involve the use of variants of uranium which is a radioactive element. The dual staining method is preferable to the uranyl acetate approach not only because it provides more detail, but also as it can improve standard petrographic analysis of the concrete (Guthrie & Carey, 1997).

Field case studies have been done employing the Los Alamos staining method. Tests have also been carried out using different Rhodamine dyes such as rhodamine B base, Rhodamine B, and Rhodamine 6G solutions. Sodium Cobaltinitrite and Rhodamine have been applied on different concrete structures and chips to detect the presence of ASR. Those concrete structures which gave positive results to Los Alamos dual staining technique were subsequently examined via microscopic petrography to verify the existence of ASR gel (Guthrie & Carey, 1998).

In 2019 a research study was conducted to analyse the alkali-aggregate reaction in concrete dams in India. They recommend some extensive investigations to understand the distress mechanism due to ASR. This includes a combination of methodologies such as visual inspection of the interior concrete core, the staining approach proposed by Guthrie & Carey (1997) which involves water-soluble compounds known as Concentrated solutions of sodium cobaltinitrite and rhodamine, Petrographic examinations of aggregates and mineral composition. They also recommend conducting x-ray diffraction and SEM study (Arora et al., 2019).

3 METHODOLOGY

3.1 Introduction

The present work is primarily concerned with the investigation of existing concrete structures suspected of being impacted by Alkali Aggregate Reaction. The objective was to check the feasibility of using an experimental procedure to detect potential AAR in such structures within the limited laboratory facilities available in a typical situation in Sri Lanka. After comparing the two staining tests discussed above it was decided to use the Los Alamos test because of its simplicity and applicability in field testing. Even though this test uses two different reagents separately, it was decided to confine the present work to one reagent: sodium cobaltinitrite which would be able to detect the vast majority of ASR cases.

The limitations that had to be considered were twofold: lack of extensive laboratory facilities and lack of experience in interpreting results of staining tests. The simplicity of the selected test addressed the former. The present study intended to address the latter by conducting a series of staining tests on concrete made with reactive aggregates as well as on control specimens made with non-reactive aggregates. If the tests, when interpreted by the authors (who do not have special expertise in such interpretations), can correctly identify potential AAR in the test specimens then it may be concluded that it would be feasible to use the selected test method for the detection of potential AAR in existing concrete structures.

The testing was done in two steps.

- Samples of reactive and non-reactive concrete specimens were cast and cured in different curing solutions for different time periods.
- Samples were tested for Alkali-Silica Reaction using staining solution.

There are several commonly available alkali reactive aggregates. In the present work waste white glass was used as the reactive aggregate. The white glass shows the highest expansion which is greater than that for Amber and Green glass (Christian, 2003). Natural coarse aggregate was used in control (non-reactive) specimens.

Three curing solutions were utilized to cure concrete specimens.

1. Water

In the Standard test for compressive strength of cylindrical concrete specimens-ASTM C 39, water is employed as a curing medium. In this research project also, distilled water was used as a curing solution.

2. 1M NaOH solution

This is widely used to accelerate ASR in aggregate identification tests such as ASTM C 1260 (Mortar-Bar Method), accelerated version of ASTM C 1293 (Concrete Prism Test) also known as the accelerated concrete prism test, and ASTM C 1567 (Accelerated Mortar-Bar Method). The NaOH solution provides a high alkaline media to accelerate ASR in concretes.

3. 1M KOH solution

To provide a more potassium-rich high alkaline medium.

These three different curing solutions, namely: water, 1 M NaOH, and 1 M KOH, were used to cure concrete specimens. KOH was primarily used as a curing solution in this study to determine whether the test results are altered by an external potassium medium in the absence of an alkali aggregate reaction. Another motivation for using potassium hydroxide is to test how this methodology reacts to different alkali kinds.

3.2 Experimental Procedure

When deciding on the design grade of concrete specimens, the capability of the specimen to provide a clean concrete surface was primarily evaluated. No strength testing, such as compression or tensile tests, were required in the methodology described. It was decided to cast reactive and control specimens with nominal Grade 25 concrete using the volume mix proportions shown in Table 1.

Table 1. Volume mix ratio for concretes

Specimen Type	**Cement**	**River Sand**	**Glass Coarse Aggregates**	**Natural Stone Coarse Aggregates**
Control	1	1	-	2
Reactive	1	1	2	-

Two batches of concrete were tested after curing for different time periods. Each batch consisted of non-reactive and reactive concrete specimens cured in respective curing agents. Timber moulds of 50 mm X 50 mm X 200 mm were used to cast the concrete specimens.

The concrete specimens were demoulded after 24 hours. They were kept for 24 hours in water and then moved to the three curing solution baths which were at room temperature.

3.3 Application of Sodium Cobaltinitrite.

3.3.1 Preparation of Specimens.

There are several methods for preparing the concrete specimen surface in the field, such as bush hammering, fracture of a core or hand sampling, and sawing/polishing to create a smooth surface for petrographic examination or preparation of a thin section. This stain testing is more efficient on fractured surfaces since fracturing causes the minimum amount of chemical change to ASR gel than surfaces created by bush hammering, sawing, or polishing. Chemical compounds that may interfere with the ASR gel resulting from methods like sawing, polishing lubricants etc. must be avoided (Guthrie & Carey, 1997). A crack was produced on the surface of the concrete specimen using a bursting wedge with a small force from a hammer. Then the concrete specimen was fractured. The fractured surface was then treated with the reagent.

3.3.2 Surface Treating.

The specimen surface was rinsed with distilled water to remove any loose dust particles and to wet the surface before treating with the Sodium Cobaltinitrite solution. Then Sodium Cobaltinitrite solution was applied to the fractured surface of the concrete. After 60 seconds the surface was rinsed again with distilled water. Following the final rinse, stained regions were slightly visible on some concrete surfaces. Specimens were dried to see the clearer yellow stains. The specimens were first examined with the naked eye to determine whether any discolouration or yellow stains were present. After observing the stains with the naked eye, the specimens were examined with a 10X hand lens to obtain a more magnified view of the stains.

4 RESULTS AND DISCUSSION

The results observed after treating with Sodium Cobaltinitrite are discussed under a few categories.

- Depending on the type of aggregate used to cast concrete specimens
- Depending on the curing solution used
- The difference in observations according to the curing duration.

4.1 Observations and Discussion According to the type of the Aggregate

Reactive and Non-Reactive Concrete Specimens cured in water were treated with Sodium Cobaltinitrite to see the reactivity of the aggregate types. It was observed that yellow stains appeared in concrete specimens produced with glass aggregates. As shown in Figure 1, Yellow stains were not evident in concrete specimens cast using non-reactive natural coarse aggregates. The same observations were made in the second batch according to Figure 2.

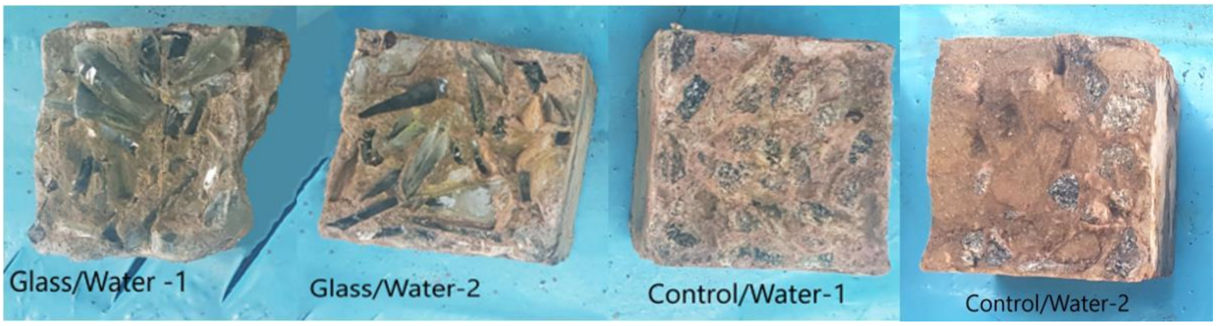

Figure 1. Concrete specimens cured in water (Batch 1)

Figure 2. Concrete specimens are cured in water (Batch 2).

In the case of curing in 1M solution of NaOH also it was observed that in both batch 1 and batch 2 yellow stains appeared in concrete specimens produced with glass aggregates, but according to Figure 3 yellow stains were not evident in concrete specimens cast using natural coarse aggregates. Figure 4 shows the second batch of concretes. In both batches, the staining was noticeable along the edges, inside the aggregate, and in the composite at the contact in broken sections of the reactive aggregate. The staining was most visible where the glass particles had been in contact before breaking the concrete specimen. The intensity of the reaction of aggregates to the curing solution is described in a different section of this paper. The amount of ASR in the concrete corresponds with the severity of yellow stains present. (Guthrie & Carey, 1998)

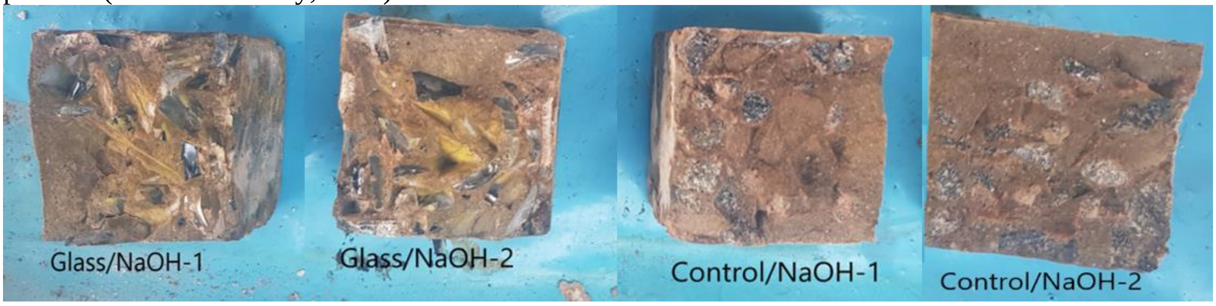

Figure 3. Concrete specimens cured in 1M NaOH (Batch 1)

Figure 4. Concrete specimens cured in 1M NaOH (Batch 2)

The results in the case of curing in 1M solution of KOH were different. Both reactive and non-reactive concrete specimens had yellow stains. This was observed in both batches as shown in Figure 5 and Figure 6. This anomaly has been addressed further under the type of curing solution.

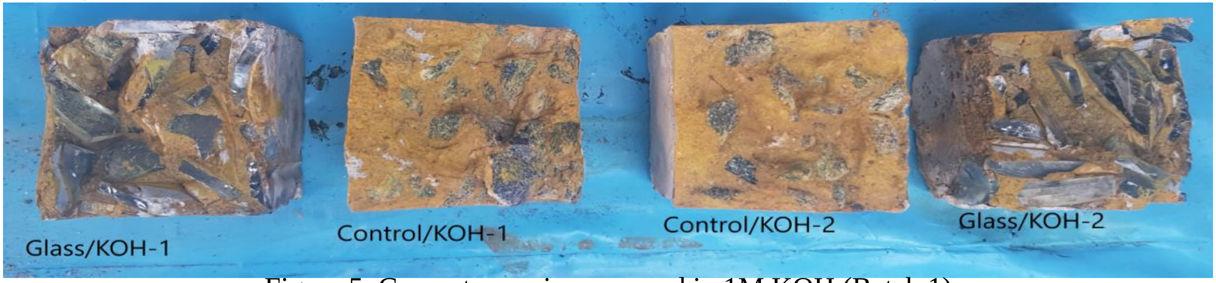

Figure 5. Concrete specimens cured in 1M KOH (Batch 1)

Figure 6. Concrete specimens cured in 1M KOH (Batch 2)

4.2 Observations and Discussion According to the type of the Curing Solution

4.2.1 Water

The concrete specimens of the first batch made using glass can be seen with slight yellow stains around the aggregates and the concrete surface. In both batches, yellow stains on concrete made with glass aggregates indicate that the AAR was likely to take place in concrete containing reactive aggregates even when cured in water, which does not contain any alkalis such as sodium and potassium. The absence of stains in control specimens of both batches suggests that the concrete cast using non-reactive aggregates was not subjected to the Alkali-Silica Reaction when cured in water.

4.2.2 1M solution of NaOH

NaOH is used here to accelerate ASR in the concrete specimens. There were no clear traces of yellow stains on the fractured surfaces of concretes containing non-reactive aggregates.

Glass aggregate concrete specimens of the first batch which were cured in 1M NaOH displayed more prominent yellow stains than those cured in water. ASR is accelerated by immersing concrete or mortar specimens in a strong alkaline solution such as 1M NaOH. These accelerated methods have been developed to minimize the length of tests and to enhance the practicality of testing.

The intensity of yellow stains in glass aggregate specimens cured in 1M NaOH is greater than that in glass aggregate concrete specimens cured in water. This confirms that the availability of alkalis in curing solution can accelerate AAR. A significant increase in yellow stain severity can be observed in batch 2 when compared with batch 1.

4.2.3 1M solution of KOH

It was observed that, when cured in KOH solution, specimens cast using control aggregates as well as reactive aggregates in both batch 1 and batch 2 displayed yellow stains all over the fractured surface of the concrete specimen. In the previous cases of curing in water and NaOH, the concrete specimens cast using control aggregates did not show any visible signs of yellow stains. The intensity of yellow colour in the two batches appear to be similar.

In this study, a simple spot test was carried out to check whether Sodium Cobaltinitrite might produce a false-positive result in the presence of K+ ions from the KOH solution even without any ASR. A concrete specimen made of non-reactive aggerates and cured in water was fractured. A few drops of KOH were added using an eyedropper onto the fractured surface of the concrete specimen. The other surface of this same specimen was not applied with KOH. When tested with sodium cobaltinitrite the surface treated with a few drops of KOH showed yellow stains as shown in Figure 7 while the surface not treated with KOH did not show any stains. This suggests that stains in the specimens immersed in KOH were caused by the alkali solution and not any ASR. The reason for yellow stains might be the reaction of Sodium Cobaltinitrite with the K+ ions available in the KOH solution.

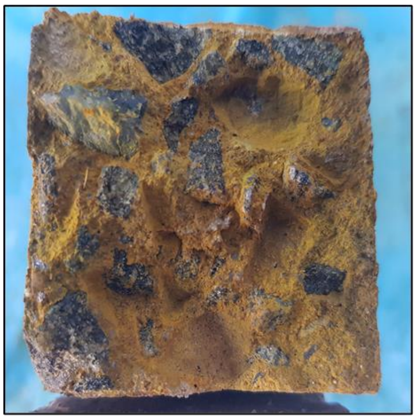

Figure 7. Control/Water specimen treated with Sodium Cobaltinitrite after applying KOH drops on the surface

The results seen from the concretes immersed in KOH is a limitation of this method. The original expectation was observations similar to those in concretes cured in NaOH. But it appears that K+ ions from other sources can produce false-positive results.

4.3 Observations and Discussion According to the curing duration.

Batch 1 specimens were cured for 44-days while those of batch 2 were cured for 60-days. It was observed that the intensity of yellow stains in the specimens containing reactive aggregates is higher in batch 2 compared to batch 1 as shown in Figure 8.

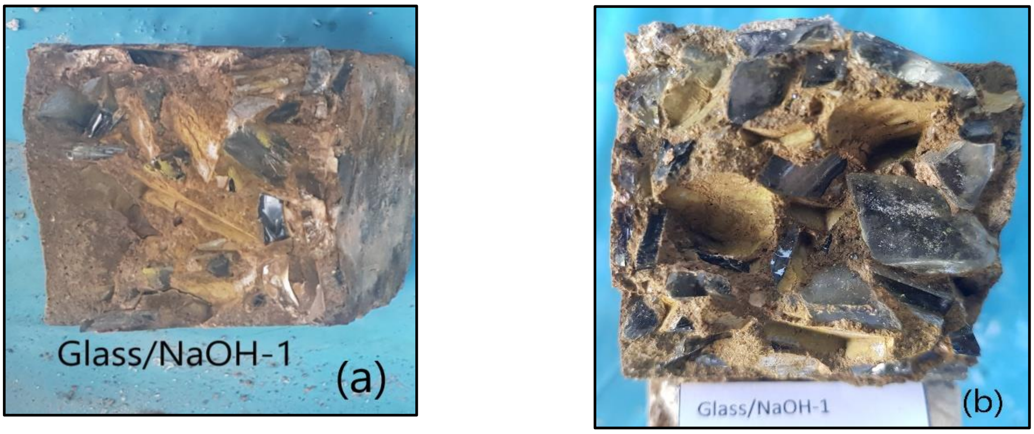

Figure 8. Glass/NaOH specimens of (a) batch 1 and (b) batch 2

AAR reaction typically continues over a long time (Swamy, 1991). Mortar Bars tests-ASTM C 1567, using recycled glass as the aggregate had been performed by Serpa et al. (2013). They observed that the expansion of mortars was greater in 28-days than in 14-days (Serpa, Silva, De Brito, Pontes, & Soares, 2013). Other researchers such as Christian (2003), Rajabipour et al (2010) have also done similar studies using mortar bar testing. They also confirm that the reaction becomes greater with exposure time (Rajabipour et al., 2010). As the intensity of yellow stains is higher in batch 2 than in batch 1, it can be concluded that the ASR has taken place in batch 2 more than in batch 1.

5 CONCLUSION

The testing outcomes were sufficient to draw some conclusions from this investigation. Sodium Cobaltinitrite staining can be used even by people without extensive experience and training to detect the presence of Alkali-Silica Reaction. The appearance of yellow stains on fractured concrete surfaces offers a rapid indication of the presence of ASR. Staining using sodium cobaltinitrite allows testing of concrete for alkali-silica reaction in a laboratory or even in the field. Using sodium cobaltinitrite to identify ASR is a viable test in the early phases of assessing a suspected structure for ASR. However, it is best to confirm any positive test results with a second test such as petrographic analysis under the supervision of an expert.

If this test does not provide a positive result on a concrete surface, it can be concluded that the Alkali-Silica Reaction is unlikely to occur in the concrete concerned.

The Los Alamos staining test examined in this study has only minimum environmental and health impacts in comparison with the uranyl acetate staining methods involving radioactive elements (Guthrie & Carey, 1997)

The Los Alamos test using sodium cobaltinitrite can be utilized as a rapid screening test in the preliminary stages of experimental identification of alkali-aggregate reaction in existing concrete structures.

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