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Role of test method in detection of alkali–silica reactivity of concrete aggregates

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The risk of alkali–silica reaction of concrete aggregates in many parts of the world remains largely unexplored. In particular, a suitable approach for testing aggregates with marginal to moderate alkali–silica reactivity has not been clearly identified. In this study, the mineralogical compositions of aggregates from five different quarries were investigated. Mortar bar expansion for these aggregates was tested as per the guidelines of both C 227 and C 1260. Although the tested aggregate sources proved non-reactive under ASTM C 227 test conditions, ASTM C 260 identified one group of aggregates as potentially reactive. Scanning electron microscopy imaging confirmed that two sources of aggregates indicated that the strength reduction of those subjected to 1 month of ASTM C 260 exposure was similar to those of specimens under 6 months of ASTM C 227 exposure. Based on the experimental results, it appears that aggregates with potential alkali–silica damage may be characterised as non-reactive if the appropriate test method is not adopted. The findings indicate that the ASTM C 1260 procedure is more effective in determining the reactivity potential of marginally to moderately reactive aggregates.

1. Introduction

Alkali–silica reactivity (ASR) in concrete is one of the primary mechanisms of concrete damage. It essentially consists of a chemical reaction (Equation I) between the hydroxyl ions in the concrete pore solution and the reactive silica in the aggregate. The product of this reaction is an alkali–silica gel that forms within and around the aggregates. This gel can absorb moisture and expand, causing distress and damage to the cementitious matrix (ACI 221 (ACI, 1998); Yildirim and Sumer, 2014). Minerals such as opal, quartzite and chert are sources of reactive silica in aggregates (ASTM C 294 (ASTM, 1998)). Such minerals react with alkalis in the presence of moisture to form alkali silica gel (Dent and Kataoka, 1981) according to the following equation.

 $\label{eq:constraint} \begin{array}{ll} \text{I.} & SiO_2 & +2NaOH + H_2O \\ & (\text{reactive silica}) & (alkalis) \\ & (alkalis \pm silica \, \text{gel}) \end{array} \rightarrow \begin{array}{l} Na_2SiO_3.2H_2O \\ & (alkalis \pm silica \, \text{gel}) \end{array}$

ASR is typically a slow process. The associated damage can take several years to be observed. Expansion of 2-3% (by volume) can occur as a result of ASR in concrete (Yildirim and Sumer, 2014) and tends to be larger within the concrete mass than at its surface (Jones and Clark, 1998), eventually leading

to cracking (Stark *et al.*, 1993). ASR has been under study for nearly eight decades (Stanton, 1940), and reported in over 60 countries (Fournier and Berube, 2000). For instance, in Argentina, more than 100 concrete structures have been recognised as affected by ASR since the 1950s (Fava *et al.*, 1991). Moreover, ASR has caused damage in Norway's Elgeseter Bridge (Jensen, 2004) and affected more than 20 bridges in the Netherlands (Siemes *et al.*, 2002). The effects of ASR on the mechanical properties of concrete have been widely explored (Jones and Clark, 1996; Monette *et al.*, 2000; Siemes and Visser, 2000; Takemura *et al.*, 1996). It was found that ASR significantly affects the modulus of elasticity and tensile strength of concrete (Koyanagi *et al.*, 2000; Larive *et al.*, 1996).

Generally, ASR has been detected and monitored primarily using field inspection (ACI 221 (ACI, 1998)). Assessing structures more than 10 years old, made with the aggregate source under consideration and subjected to wetting and drying can usually give an indication of the reactivity of an aggregate source (ACI 221 (ACI, 1998)). The inspection typically involves visual observations with particular focus on indications of distress, such as movements and displacements, map cracking, presence of alkali–silica gel and scaled surfaces having aggregates surrounded by reaction rims (Stark, 1991a,

1991b) that usually consist of a calcium–alkali–silicate layer (Charlwood and Solymar, 1994). Petrographic examination of concrete cores from such structures should be carried out to confirm the presence of ASR.

Various methods to evaluate ASR potential have been proposed (Berube and Fournier, 1994; Diamond, 1978; Grattan-Bellew, 1981, 1989; Kosmatka and Panarese, 1988; Sims, 1981; Stark, 1991a, 1991b), and detailed methodologies for testing, evaluation and management of ASR have been provided, for instance, by the CSA (2005) and ASTM (Table 1) standards. Different testing methods for assessing the potential of ASR are presented in Table 1, among which ASTM C 227 (ASTM, 2010) and ASTM C 1260 (ASTM, 2014c) are the most commonly used.

ASTM C 227 (ASTM, 2010) has long been a commonly used method for detecting the potential of ASR. However, it requires 6 months to complete (Raja *et al.*, 2014; Stark, 1980). Moreover, the exposure conditions of ASTM C 227 (ASTM, 2010) are not sufficiently severe to generate ASR in a short time. Conversely, while ASTM C 1260 (ASTM, 2014c) is an improved method, it has been perceived as too severe and does not replicate field conditions (Lu *et al.*, 2006; Yildirim and Sumer, 2014). Moreover, there usually are discrepancies between the results of the two test methods. Thus, this study was carried out to evaluate the effectiveness of both the ASTM C 227 (ASTM, 2010) and ASTM C 1260 (ASTM, 2014c) guidelines in detecting the ASR potential of local aggregates.

2. Materials

Aggregates from five different sources were selected because they are widely used in local concrete construction, yet there is ongoing ambiguity regarding their potential ASR. The selected aggregate sources included the Sheikh Hills, Tuguwali Hills and Mach Hills from the Sargodha region, the Jhelum River at the Lehri Mangla and the Kamser Mountains from Muzaffarabad in Kashmir. From the Sargodha and Kashmir sources, large rock fragments were obtained through blasting, whereas, from the Jhelum River source, stones were collected from the riverbed. These rock fragments and stones were then crushed into aggregates at industrial crusher plants. From the Sargodha aggregate source, the dry technique was used for crushing, whereas aggregates from the Jhelum River and Kashmir sources were crushed using the wet technique. Samples of the various aggregates are shown in Figure 1. Locally produced ordinary Portland cement (Bestway Cement) was used throughout this study. Clean tap water was used for mixing the mortar.

3. Experimental procedures

3.1 Materials characterisation

To investigate the mineralogical composition of the aggregates, petrographic examination was performed according to ASTM C 295 (ASTM, 2012). Moreover, the physical properties of the various aggregates were characterised. The bulk density and voids content were evaluated as per ASTM C 29 (ASTM, 2016a). The specific gravity and water absorption of the aggregates were measured according to ASTM C 127 (ASTM, 2015b). The impact and crushing value tests were performed on the aggregates as per BS 812-112 (BSI, 2014b) and BS 812-110 (BSI, 2014a). The abrasion resistance of the aggregates was examined using ASTM C 535 (ASTM, 2016e).

The chemical properties of the cement used were quantified using X-ray fluorescence. To further characterise the cement, various other tests were carried out, including the standard consistency test described in ASTM C 187 (ASTM, 2011a); setting time according to ASTM C 191 (ASTM, 2013); fineness test as per ASTM C 184 (ASTM, 1994); fineness test outlined in ASTM C 204 (ASTM, 2016d). Moreover, soundness was explored using the EN 196-3 procedure (EN, 2005) and autoclave expansion as per the guidelines of ASTM C 151 (ASTM, 2015c).

3.2 Mortar bar expansion testing

3.2.1 ASTM C 227 test procedure

Mortar bar specimens incorporating aggregates from the various sources were prepared. Aggregates from the selected sources were graded according to ASTM C 227 (ASTM, 2010). Figure 2 shows the gradation curve. The mixture proportions for the mortars involved a cement-to-aggregates mass ratio of 1 to 2.25 and a water-to-cement ratio of 0.47 in accordance with ASTM C 227 (ASTM, 2010). Mixing was conducted using an electric mortar mixer (Figure 3(a)) according to ASTM C 305 (ASTM, 2014a). After 5 min of mixing, the flow diameter of the fresh mortar was determined in accordance with ASTM C 1437 (ASTM, 2015e). The flow diameter for all the tested mixtures was within 105-120 mm (Figure 3(b)). After flow table testing, the mixture was poured into mortar bar moulds in two layers. The mortar bar moulds were two gang prism moulds having dimensions of $25 \times 25 \times 285$ mm with an effective gauge length of 250 mm (Figure 3(c)). Each layer was well compacted using a plastic tamper.

The specimens were covered with a plastic sheet to avoid loss of moisture until demoulding at 24 h. The initial length of each bar specimen was measured using a digital length comparator (Figure 3(d)) as described in ASTM C 490 (ASTM, 2011b). Before measuring the length, a reference bar was used to calibrate the comparator. Specimens were then stored in airtight containers containing water, such that the specimens did not touch the base of the container (Figure 4(a)). The specimen containers were then placed in an oven at 40°C. After 14 d, the containers were stored at 25°C for 16 h, and then length readings were taken. Readings were taken using a

Table 1. Test methods for evaluating ASR potential of aggregates

Standards/references	Test	Testing material and status	Description
		A .	
ASTM C 295 (ASTM, 2012)	Petrographic examination	Aggregates (indirect method)	reactive materials
ASTM C 227 (ASTM, 2010)	Mortar bar test	combination (direct method)	6–12 months duration is required. Mortar bars are stored at 38°C under 100% relative humidity (RH)
ASTM C 1260 (ASTM, 2014c)	Accelerated mortar bar test	Cement aggregate combination (direct method)	Modified form of ASTM C 227 (ASTM, 2010), 14–28 d duration is required. Mortar bars are stored at 80°C under 100% RH in 1 N sodium hydroxide solution
ASTM C 289 (ASTM, 2003)	Quick chemical test	Aggregate (indirect method)	Crushed material is sealed at 80°C in 1 N sodium hydroxide solution for 24 h to measure the amount of dissolved reactive silica. Results can be classified as aggregate source is reactive or not
ASTM C 1293 (ASTM, 2015d)	Concrete prism test	Concrete prisms (direct method)	365 d are required. Specimens are placed at 38°C in sodium hydroxide solution having alkali level of 1.25% Na-Oe
ASTM C 342 (ASTM, 1997)	Potential volume change of cement aggregate	Cement aggregate combination (direct method)	365 d are required. Mortar bars are stored in various temperature and moisture conditions
USACE (1994)	US Army Corps of Engineers modified mortar bar test	Cement aggregate combination (direct method)	Modified form of ASTM C 227 (ASTM, 2010) to detect slow-reacting aggregates. With the help of petrographic examination, siliceous aggregates having strained quartz more than 20% are selected for evaluation through the mortar bar test. Mortar bars are stored at 60°C under 100% RH for 365 d
Bonzel and Dahms (1973), Dahms (1977)	Concrete cube test	Cement aggregate combination (indirect method)	Specimens are stored in a moist cabinet under 40°C. Periodic expansion is done for gel exudations and cracking. However, it is not stated whether aggregate source is deleterious or not
Chatterji (1978)	Nordtest accelerated ASR test	Cement aggregate combination (direct method)	Also a modified form of ASTM C 227 (ASTM, 2010). However, this method is used only for reactive aggregates in Denmark. Mortar prisms having smaller length and greater cross-section than ASTM C 227 (ASTM, 2010) are cured for 4–5 weeks in water and then saturated in sodium chloride solution at 50°C for 8 weeks
Sims (1981), NBS (1958)	Gel pat test	Cement aggregate combination (indirect method)	Mortar bars having smooth sawn surfaces are placed in alkali solution for 3 d. Gel will form in the presence of reactive particles and the percentage of reactive particles can be estimated
Fournier and Berube (1991)	Autoclave mortar bar test	Cement aggregate combination (direct method)	Also a modified form of ASTM C 227 (ASTM, 2010). However, sodium hydroxide is added to water to raise the alkali content. Mortar bars are stored under 100% RH at 23°C for 2 d before autoclave treatment. Autoclave test is performed with steam curing at 130°C
Natesaiyer and Hover (1988, 1989)	Uranyl acetate gel fluorescence test	In situ hardened concrete (indirect method)	This test is used in conjunction with petrographic examination to confirm the presence of ASR products in hardened concrete. Fluorescence will be produced by ASR products in concrete treated with uranyl acetate solution when observed under ultraviolet light. This is a rapid test. However, it should always be performed by trained specialists
Stark (1985, 1991a, 1991b)	ASR in hardened concrete	Hardened concrete (direct method)	To determine the alkalis and reactive silica amounts, cores are extracted and placed in different conditions – that is, in water, in 1 N sodium hydroxide solution and in sealed water at 38°C. The expansion of these cores is measured for 12 months. The results of length change under different conditions indicates possible future ASR expansion in concrete

(continued on next page)

Table 1. Continued

Standards/references	Test	Testing material and status	Description
Donnell <i>et al.</i> (2013)	Microwave method	Cement aggregate combination (direct method)	Mortar bar specimens are prepared according to ASTM C 1260 (ASTM, 2014c) and then sealed in water at 38°C, and microwave measurements are made. Specimens are then placed under ambient conditions for 36 d and final measurements are taken. This test is based on the property that due to the formation of gel and attraction of free water from the environment, readings of microwave methods vary

similar procedure after each month for up to 6 months, and the water in the containers was replaced. The length change was quantified using the following equation

$$1. \qquad L = \frac{(L_x - L_i)}{G} \times 100$$

where L is the change in length at age x (%), L_x is the comparator reading at age x (mm), L_i is the initial comparator reading of specimens (mm) and G is the nominal gauge length (250 mm).

3.2.2 ASTM C 1260 test procedure

Aggregates from the selected sources were graded according to ASTM C 1260 (ASTM, 2014c) and used to prepare standard mortar bars. Similar mixture proportions as described above were used. After measuring their initial length, the bar specimens were placed in water in sealed containers. The containers were then placed inside an oven at 80°C. After 24 h, the initial length of each specimen was measured. Specimens were then immersed for 14 d in a 1 N sodium hydroxide (NaOH) solution (Figure 4(b)) at 80°C. Length measurements were conducted after 14 and 28 d.

3.3 Mechanical properties

To explore the effects of ASR on the compressive strength and modulus of rupture of the various mortars, 90 cubes and prisms incorporating aggregates from the various sources investigated were cast. Cube specimens were cast in two layers using three gang $25 \times 25 \times 25$ mm cube moulds. Each layer was compacted by tamping it 32 times using a plastic tamper. Prisms of $40 \times 40 \times 160$ mm size were also cast in layers and compacted in a similar manner. The specimens were covered with a plastic sheet for the first 24 h to avoid loss of moisture. After demoulding, nine control cubes and prisms for each aggregate source were cured in fresh water. Moreover, nine cubes and prisms for each aggregate source were stored in an air-tight container containing water such that the specimens did not touch the base of the container. Six specimens (cubes and prisms) were then kept in an oven at 40°C according to ASTM C 227 (ASTM, 2010). The remaining three cubes and prisms for each aggregate source were kept in water for 1 d at 80°C (Figure 4(c)) and then immersed in

a 1 N sodium hydroxide solution inside air-tight containers placed in an oven at 80°C according to ASTM C 1260 (ASTM, 2014c). After 1 and 6 months, the control specimens and the specimens placed at 40°C were removed and tested. Specimens placed at 80°C were tested after 28 d of exposure.

Compressive testing was conducted at a loading rate of 1000 N/s as per ASTM C 109 (ASTM, 2016c). The modulus of rupture was obtained by applying a rate of 2640 N/s as per ASTM C 348 (ASTM, 2014b). Furthermore, scanning electron microscopy (SEM) imaging of selected mortar bar specimens was carried out to investigate the microstructural features of the tested mortars after various regimes of exposure.

4. Results and discussion

4.1 Binder characteristics

Table 2 shows the chemical analysis results of the cement, which indicates that all chemical compounds were in accordance with the limits specified in ASTM C 114 (ASTM, 2015a), except for the alkalis (i.e. 0.85%), which was higher than the ASTM C 114 (ASTM, 2015a) recommended maximum of 0.60%. The amount of alkalis mainly depends on the nature of the raw materials of the cement and on the production process. To reduce the alkali content, modifications in the manufacturing process of cement may be required, which is typically uneconomical (Johansen, 1989). Table 3 reports the results of physical tests on the cement. Generally, these physical properties were within ASTM and European (EN) standard limits. For example, the cement fineness (99% and 2996 cm²/g) satisfy the ASTM C 184 (ASTM, 1994) and C 204 (ASTM, 2016d) standard limits (fineness of cement should not be <90% and $2250 \text{ cm}^2/\text{g}$). The autoclave expansion was 0.075%, which satisfies the ASTM C 151 (ASTM, 2015c) guidelines. The soundness of the cement also met the EN 196-3 (EN, 2005) standard requirements.

4.2 Petrographic examination of aggregates

4.2.1 Sheikh Hills aggregates

Figure 5(a) shows the petrographic examination results for the Sheikh Hills aggregate, which were mainly composed of three





(e)

Figure 1. Illustration of samples from the various aggregate sources: (a) Sheikh Hills aggregate, (b) Tuguwali Hills aggregate, (c) Mach Hills aggregate, (d) Jhelum river stone and (e) Kamser Mountains stone

rock types: dolerite, quartzite and rhyolite. Dolerite is dominant (56%); with quartzite (27%) and rhyolite (17%) constituting the rest of the aggregates. The composition of the dolerite mineral is presented in Figure 5(b). The dolerite was fine- to



Figure 2. Gradation curve of aggregate sources

medium-grained and micro-porphyritic, having ophitic to subophitic texture. It was moderately chloritised, sericitised and calcitised. Dolerite was found to be a non-alkali reactive rock type (WAPDA, 2004). The mineralogical composition of the quartzite is outlined in Figure 5(c). The quartzite was mainly composed of fine-grained to vitreous quartz (about 87% of the rock) with interstitial muscovite and sericite. Tiny grains of quartz were interlocking and mono-crystalline. Muscovite and sericite occur as small anhedral flakes and represent about 8% of the rock. Some subhedral to anhedral crystals of secondary calcite were also present (about 3% of the rock). Sporadic fine grains of haematite were also observed (about 7% of the rock). According to previous studies (ACI 221 (ACI, 1998); WAPDA, 2004), quartzite is usually slowly alkali reactive. The composition of the rhyolite is shown in Figure 5(d). The rhyolite pieces were light to medium grey on fresh surfaces and rusty brown to yellowish grey on weathered surfaces. The rock was fine grained and microcrystalline to cryptocrystalline having about 36% of quartz. Rhyolite is generally alkali reactive (ACI 221 (ACI, 1998); WAPDA, 2004). The presence of reactive quartzite and rhyolite in the Sheikh Hills aggregates warrants a careful study of their potential ASR.



Figure 3. Illustration of (a) mortar mixing procedure, (b) flow table measuring, (c) typical mortar cube and bar casting and (d) digital length comparator



Figure 4. Illustration of (a) specimen positioning in container, (b) mortar bar specimens in sodium hydroxide solution and (c) cube and prism specimens placed at 80°C in water

Table 2. Chemical analysis of cement

Constituent: %	Ordinary Portland cement	ASTM C 114 limits (ASTM, 2015a)
Silicon dioxide (SiO ₂)	20.50	20 minimum
Aluminium oxide (Al ₂ O ₃)	5.08	6 maximum
Ferrous oxide (Fe ₂ O ₃)	3.18	6 maximum
Calcium oxide (CaO)	62.4	-
Magnesium oxide (MgO)	2.28	6 maximum
Free lime	1.24	2 maximum
Sulfur trioxide (SO ₃)	1.96	3 maximum
Insoluble residue (IR)	0.60	0.75 maximum
Loss on ignition (LOI)	2.04	3 maximum
Sodium oxide (Na ₂ O)	0.42	_
Potassium oxide (K ₂ O)	0.66	-
Equivalent alkali content (Na ₂ O _e)	0.85	0.60 maximum

4.2.2 Tuguwali Hills aggregates

Figure 6(a) displays the petrographic examination results of the aggregates from Tuguwali Hills. These aggregates were mainly composed of rhyolite rock, with the presence of several other minerals. For instance, K-feldspar was around 23% in the

rock. Rhyolite pieces were light to medium grey in fresh surfaces and rusty brown to yellowish grey on weathered surfaces. The rock was fine grained and microcrystalline to cryptocrystalline. Quartz occurs as anhedral to idioblastic, whereas K-feldspar was anhedral to sub-idioblastic to blastoporphyritic. Plagioclase was generally present as microblasto-porphyritic and as tiny crystals in the groundmass. Calcite was present as groundmass and ilmenite/leucoxene occurred as fine-grained crystals.

4.2.3 Mach Hills aggregates

Figure 6(b) portrays the results of the petrographic examination for the Mach Hills aggregate, which were mainly composed of a volcanogenic slate rock type, with the presence of a variety of other minerals. For example, muscovite represented 35% of the rock. The aggregates were generally uniform in appearance. Aggregate particles were found to be greyish green on fresh surfaces and rusty brown on weathered surfaces. Some particles were spotted with dark-coloured minerals. The rock pieces exhibited moderate fissility on a macroscopic scale. The rock was fine grained and showed a faint orientation of the

Table 3. Physical properties of cement

Property	Standard	Value	Limits
Standard consistency Initial setting time Final setting time Fineness (passing no. 200) Fineness (Blaine air permeability) Soundness	ASTM C 187 (ASTM, 2011) ASTM C 191 (ASTM, 2013) ASTM C 191 (ASTM, 2013) ASTM C 184 (ASTM, 1994) ASTM C 204 (ASTM, 2016d) EN 196-3 (EN, 2005) ASTM C 151 (ASTM, 2015c)	24% 91 min 165 min 98·6% 2996 cm ² /g 0·60 mm 0.075%	— > 45 min < 375 min Min. 90% Min. 2250 cm ² /g Max. 10 mm
Autoclave expansion	ASTM C 151 (ASTM, 2015c)	0.075%	Max. 0.8%



Figure 5. Mineralogical composition of Sheikh Hills aggregate: (a) composition of Sheikh Hills aggregate, (b) mineralogical composition of dolerite, (c) mineralogical composition of quartzite and (d) mineralogical composition of rhyolite. * shows reactive minerals

muscovite flakes. Quartz was present as small, rounded fragments and very fine-grained muscovite formed the matrix of the rock. A significant proportion of the rock was composed of slowly reactive quartz mineral.

4.2.4 Jhelum riverbed aggregates

Figure 6(c) illustrates the petrographic examination results of the Jhelum riverbed aggregates, which were mainly composed of quartzite (54%) and sandstone (36%). The mineralogical



Figure 6. Mineralogical nature of the various aggregates and their compositions: (a) composition of Tuguwali Hills aggregate, (b) composition of Mach Hills aggregate, (c) Jhelum River aggregate, (d) quartzite in Jhelum River aggregate, (e) sandstone in Jhelum River aggregate and (f) composition of Kamser Mountain aggregate. * shows reactive minerals

composition of the quartzite mineral is shown in Figure 6(d). The rock pieces were creamy to light grey in colour and showed no signs of weathering. The grain boundaries of the quartz were irregular and wavy. Most of the grains were polycrystalline and strained. Rare flakes of muscovite were also present. A small volume of pore spaces was lined with microquartz. Overall, quartz constituted about 99% of the rock. The composition of the sandstone is outlined in Figure 6(e). Rock pieces were medium grey, purple grey and greenish grey in colour. Some pieces were faintly weathered while most were fresh. Thin sectioning showed that the sandstone-containing quartz is known to be slowly alkali reactive (Munir *et al.*, 2016; Rivard *et al.*, 2002; WAPDA, 2004).

4.2.5 Kamser Mountain aggregates

Figure 6(f) portrays the petrographic examination results of aggregates from Kamser Mountain, which were primarily composed of dolomite (about 96%), with the presence of several other minerals. For instance, argillaceous and fine-grained quartz represented about 3% of the rock. Aggregates appeared medium to dark grey in colour and were found to be nonreactive using cold diluted hydrochloric acid (HCl). Results showed that the rock was mainly composed of fine-grained anhedral to subhedral dolomite crystals. The fabric of the rock may be described as fine-grained equi-granular to subequi-granular, having intergranular pore spaces between crystals. This may have resulted from the bulk volume shrinkage that accompanies calcite replacement by dolomite (ACI 221 (ACI, 1998)). Haematite (about 1% of the rock) was present as fine grains, distributed in the rock and as coating on the grain boundaries. This rock may cause alkali carbonate reaction, and is generally considered ASR non-reactive. Yet, a previous study (Abbas et al., 2017; NESPAK, 2011) concluded that such rock can be ASR reactive.

4.2.6 Overall reactivity of various aggregate sources

Figure 7 exhibits the amount of reactive constituents in each aggregate source based on petrographic examination. It can be observed that some of the aggregate sources present real alkali–aggregate reactivity potential. For example, the amount of reactive constituents in the Jhelum River source is 77%. With respect to ASR, the reactive constituents were more significant in the Jhelum River source and the least in the Kamser source. However, the Mach, Tuguwali and Sheikh Hills aggregate sources were also considered as slow reactive. Various researchers proposed different classifications for slow reactive aggregates. For instance, Magni *et al.* (1986) suggested 15% as an acceptable limit for the amount of reactive minerals, whereas, in Norway, if the quantity of reactive constituents is >20%, further testing is recommended (Borge, 1995). ASTM C 33 (ASTM, 2016b) recommends testing including the

chemical test and the mortar bar test to qualify ASR when reactive materials are present even at <1%. Therefore, it can be argued that all the tested sources of aggregates may possibly cause ASR, and thus should be further investigated, for instance under ASTM C 227 (ASTM, 2010) and ASTM C 1260 (ASTM, 2014c), before using such aggregates in full-scale construction works to ensure adequate service life performance of structures.

4.3 Physical properties of tested aggregates

Table 4 shows the physical properties of the aggregate sources. The highest bulk density was measured for the Kamser aggregates and the lowest was for the Mach Hills source. The bulk density generally ranged from 1200 to 1760 kg/m³. The Mach Hills aggregate had the highest voids content, whereas the Kamser source had the least. The volume of voids is typically governed by the grading and shape of the particles. It normally ranged from 33 to 42% (ACI 221 (ACI, 2001)), which is within typical values.

Specific gravity was higher for the Kamser aggregate source and the least for the Jhelum River source. However, there was an opposite trend for water absorption. The Kamser aggregates showed less water absorption compared with the Jhelum River aggregates (primarily sandstone). According to a previous study (Carlson, 1938), aggregates composed of sandstones usually have higher water absorption and relatively lower specific gravity.

Table 4 reports the impact, crushing and abrasion test results of aggregates from the various sources explored in this study. The Kamser source aggregate exhibited highest resistance against impact and abrasion, while the Jhelum River aggregate had the least. The aggregates from the Tuguwali Hills showed



Figure 7. Reactive constituents in the various aggregate sources

Table 4. Physical properties of aggregates

	Aggregate sources				
Property	Sheikh Hills	Tuguwali Hills	Mach Hills	Jhelum Riverbed	Kamser Mountain
Bulk density: kg/m ³ (ASTM C 29) (2016a)	1551	1548	1535	1596	1699
Voids content: % (ASTM C 29) (2016a)	39.99	41.24	41.66	38.54	37.53
Specific gravity (ASTM C 127) (2015b)	2.73	2.64	2.59	2.56	2.77
Water absorption: % (ASTM C 127) (2015b)	1.03	0.98	1.12	1.47	0.76
Impact value (BS 812-112) (2014b)	11.90	13.10	13.30	14.50	10.10
Crushing value (BS 812-110) (2014a)	18.90	14.80	25.20	21.30	17.10
Abrasion test (ASTM C 535) (2016e)	19.20	21.80	24.20	26.90	17.90



Figure 8. Mortar bar expansion results according to ASTM C 227 (ASTM, 2010)

the highest resistance against crushing, while the Mach Hills source showed the least resistance. The impact and abrasion resistance is usually higher for crushed aggregates than for round aggregates (Alexander and Mindess, 2005), which explains the results of the Jhelum River aggregates. Generally, the abrasion of aggregates ranges from 15 to 50% (Neville, 2000). Abrasion values of all the tested aggregate sources, which ranged between 17.9 and 26.9%, are within specifications. The impact value of aggregates is usually <25% and the crushing value generally ranges from 14 to 30% (Alexander and Mindess, 2005). Experimental results for the tested aggregates show that the impact and crushing values were <15 and 26%, respectively, indicating suitability of the aggregates for use in concrete construction works.

4.4 Results of ASTM C 227 and ASTM C 1260 tests

4.4.1 Mortar bar expansion

Figure 8 illustrates the average ASTM C 227 (ASTM, 2010) mortar bar expansion for specimens incorporating the various aggregates. After 3 months, mortar bars made with the

Tuguwali aggregate showed less expansion than those made with the Sheikh Hills aggregate. However, at the end of the 6-month exposure, the results were reversed. The Tuguwali aggregate had more reactive minerals than the Sheikh Hills source. Yet, due to its slower reactivity, more expansion was observed at later ages. Similar behaviour was reported for other aggregates in previous studies (Ahmed et al., 2013; Carlson, 1938; Islam and Ghafoori, 2013; Thibodeaux et al., 2003). The average expansion of mortar bars incorporating the Mach Hills aggregate was the highest. Specimens incorporating the Mach, Tuguwali and Sheikh Hills aggregates exhibited expansion in the range of 0.05-0.07% at the end of 6 months. Results were consistent with findings of the petrographic examination. According to ASTM C 33 (ASTM, 2016b), aggregates are considered alkalisilica reactive if the mortar bar expansion is more than 0.10%after 6 months under the ASTM C 227 (ASTM, 2010) test conditions. None of the aggregates tested herein has shown expansion >0.07%. No distress was observed on mortar bar surfaces after the 6-month exposure period. However, the ASTM C 227 (ASTM, 2010) is not used for acceptance and rejection of aggregate sources. The longer term ASTM C 1293 (ASTM, 2015d) is usually adopted for this purpose.

Figure 9 portrays the average ASTM C 1260 (ASTM, 2014c) accelerated mortar bar expansion values. It can be observed that the specimens incorporating the Mach Hills aggregate incurred the highest expansion, whereas those made with the Kamser aggregate had the least expansion. This is ascribed to the greater reactive minerals content in Mach Hills aggregates as observed in the petrographic examination. The specimens made with the Mach, Tuguwali and Sheikh Hills aggregate sources, which all come from the Sargodha region, exhibited expansion values >0.10 and 0.20% after 14 and 28 d, respectively. Hence, based on ASTM C 1260 (ASTM, 2014c), such aggregates can be considered alkali-silica reactive. These aggregates were not considered reactive under ASTM C 227 (ASTM, 2010) testing. Generally, ASTM C 1260 (ASTM, 2014c) has been considered as more effective for testing slow reactive minerals in previous studies (Davies and Oberholster, 1987; Hooton, 1990; Stark et al., 1993).



Figure 9. Accelerated mortar bar expansion results according to ASTM C 1260 (ASTM, 2014c)

Mortar bar expansion results showed that the aggregates from the Jhelum River and Kamser are not reactive under both ASTM C 227 (ASTM, 2010) and C 1260 (ASTM, 2014c) standard test methods. The results for the Kamser source were consistent with both the petrographic examination and past findings (Islam and Akhtar, 2013). However, despite petrographic examination indicating the highest content of reactive minerals (i.e. 77%) in the Jhelum River aggregate, mortar expansion results for this aggregate source were <0.04 and 0.085%, in accordance with ASTM C 227 (ASTM, 2010) and ASTM C 1260 (ASTM, 2014c), respectively. This can be explained by the so-called pessimum effect (Ichikawa, 2009; Johansen, 1989). The pessimum effect is defined as the largest expansion of concrete due to a definite proportion of reactive siliceous aggregates. However, if the reactive aggregates content decreases or increases from the pessimum level, a decrease in expansion can be observed (Ichikawa, 2009; Stanton, 1940). It is believed that the 77% reactive mineral content in the Jhelum River aggregate largely exceeded the pessimum value, thus leading to low expansion. However, this needs to be investigated further using the ASTM C 1293 (ASTM, 2015d) concrete prism test.

4.4.2 Effect of ASR on compressive strength

Figure 10 displays the compressive strength results of mortar specimens tested under ASTM C 227 (ASTM, 2010). As expected, the control specimens reached a higher compressive strength at 6 months than that at 1 month due to the progress of hydration reactions. Conversely, the specimens tested under 1 month of ASTM C 227 (ASTM, 2010) ASR exposure conditions had compressive strength values at 6 months comparable to, and in some cases lower than, that reached at 1 month. For instance, after 6 months of ASTM C 227 (ASTM, 2010) exposure, a 25% decrease in compressive strength was recorded



Figure 10. Compressive strength of specimens under ASTM C 227 (ASTM, 2010) exposure conditions



Figure 11. Relationship between compressive strength and expansion of specimens under ASTM C 227 (ASTM, 2010) exposure conditions

for specimens incorporating the Mach Hills aggregate compared with that of the control specimens. This decrease in compressive strength was more prominent for the Sargodha aggregate sources due to its alkali–silica reaction as discussed earlier and reported by others (Ahmed *et al.*, 2013; Bignozzi and Saccani, 2012). However, the specimens incorporating the Kamser and Jhelum aggregates also incurred a slight decrease in compressive strength (about 3%), likely due to the limited ASR expansion associated with these aggregates as discussed above. Figure 11 shows the relation between compressive strength and 6-month mortar bar expansion under ASTM C 227 (ASTM, 2010) test conditions. A decreasing trend can be observed between

compressive strength and expansion values, as reported by others (Giaccio *et al.*, 2008).

Figure 12 portrays the compressive strength of specimens incorporating the various sources of aggregates under ASTM C 1260 (ASTM, 2014c) exposure conditions. Results show that specimens tested under ASTM C 1260 (ASTM, 2014c) demonstrated a decrease in compressive strength. For instance, specimens incorporating the Mach Hills aggregate incurred a 22% decrease in compressive strength. Similar to the case of ASTM C 227 (ASTM, 2010) exposure, specimens incorporating aggregates from the Sargodha region exhibited a significant decrease



Figure 12. Compressive strength of specimens under ASTM C 1260 (ASTM, 2014c) exposure conditions



Figure 13. Relationship between compressive strength and expansion of specimens under ASTM C 1260 (ASTM, 2014c) exposure conditions

in compressive strength under ASTM C 1260 (ASTM, 2014c) exposure conditions compared with that of the control specimens. Specimens made with aggregates from the Jhelum and Kamser sources incurred a comparatively lower decrease in compressive strength. This is ascribed to lower ASR expansion as discussed above. The relationship between compressive strength and ASTM C 1260 (ASTM, 2014c) expansion also showed a decreasing trend (Figure 13). Increased expansion due to ASR resulted in reduced compressive strength (Giaccio *et al.*, 2008).

It can be concluded that the compressive strength of specimens incorporating aggregates from the Sargodha region was more affected by ASR, especially at later ages. The more reactive aggregates yielded a more significant decrease in compressive strength. The overall reduction in compressive strength for specimens tested under ASTM C 227 (ASTM, 2010) ranged from 22 to 25%, while it ranged from 10 to 22% for identical specimens tested under ASTM C 1260 (ASTM, 2014c) conditions. This is ascribed to the difference in exposure conditions between the two test methods and to the fact that ASTM C 227 (ASTM, 2010) results represent later age (6 months) behaviour, whereas those for ASTM C 1260 (ASTM, 2014c) are obtained after 1 month of exposure, in accordance with previous research (Ahmed *et al.*, 2013; Islam, 2010).

4.4.3 Effect of ASR on modulus of rupture

Figure 14 shows the flexural strength results of control specimens and specimens tested under ASTM C 227 (ASTM, 2010) conditions. For specimens tested under 1-month ASR conditions of ASTM C 227 (ASTM, 2010), an increase in flexural strength was observed as compared with 1-month control samples. This may be due to the presence of slow reactive



Figure 14. Modulus of rupture tested under ASTM C 227 (ASTM, 2010) exposure conditions

aggregates. As expected, the control specimens gained higher modulus of rupture after 6 months compared with that at 1 month. However, the 6-month flexural strength of specimens exposed to ASTM C 227 (ASTM, 2010) conditions was comparatively lower than that of the control specimens at the same age. For instance, there was a 22% decrease in flexural strength of specimens incorporating the Tuguwali Hills aggregate compared with that of the control. This decrease in flexural strength can be attributed to ASR, which can create micro-cracks that decrease flexural strength (Thibodeaux *et al.*, 2003). Specimens incorporating aggregates from the Kamser and Jhelum sources showed no significant decrease in flexural strength (< 2%) compared with the control specimens, which is



Figure 15. Relationship between modulus of rupture and expansion of specimens under ASTM C 227 (ASTM, 2010) exposure conditions



Figure 16. Modulus of rupture of specimens under ASTM C 1260 (ASTM, 2014c) exposure conditions

consistent with the mortar bar expansion results. Figure 15 shows the relation between flexural strength and ASTM C 227 (ASTM, 2010) expansion, indicating decreased flexural strength with increased expansion.

Figure 16 shows the flexural strength results for specimens tested under ASTM C 1260 (ASTM, 2014c) exposure. A decrease in flexural strength for specimens tested under ASTM C 1260 (ASTM, 2014c) compared with the control specimens can be observed. For example, specimens incorporating aggregates from the Sheikh Hills source suffered a 22% decrease in flexural strength compared with the control specimens. All tested aggregates from the Sargodha sources have shown a distinct decrease in flexural strength under ASTM C 1260 (ASTM, 2014c). This decrease was more prominent for specimens incorporating the Mach Hills aggregates, likely due to its higher ASR expansion and the associated micro-cracking as discussed above. Specimens made with aggregates from the Jhelum and Kamser sources had comparatively smaller decrease in flexural strength (up to 8%) under ASTM C 1260 (ASTM, 2014c) exposure compared with the control specimens. Furthermore, a decreasing trend was also observed for flexural strength plotted against ASTM C 1260 (ASTM, 2014c) expansion values (Figure 17).

Similarly to compressive strength, it was observed that the flexural strength of specimens from the Sargodha aggregate sources was more significantly affected by ASR. The more reactive the aggregate, the greater the decrease in flexural strength. The overall reduction in the flexural strength of the specimens tested under ASTM C 227 (ASTM, 2010) ranged from 23 to 29%, while it ranged between 22 to 34% for those tested under ASTM C 1260 (ASTM, 2014c). Marzouk and Langdon (2003) also reported a reduction in flexural strength of up to 24%



Figure 17. Relationship between modulus of rupture and expansion of specimens under ASTM C 1260 (ASTM, 2014c) exposure conditions



Figure 18. SEM images of mortar bar incorporating Mach Hills aggregates under ASTM C 1260 (ASTM, 2014c) exposure conditions: (a) presence of ASR gel around reactive aggregate particle, (b) cracking observed in mortar specimens around reactive aggregates and (c), (d) cracks due to ASR

associated with moderately reactive aggregates. Highly reactive aggregates can drastically decrease flexural strength (up to 84%), as reported elsewhere (Ahmed *et al.*, 2013).

4.5 Microscopic examination

SEM was performed to examine the microstructure of mortar bar specimens under different ASR exposure conditions. Figure 18 shows SEM images of specimens incorporating the Mach Hills aggregate tested under ASTM C 1260 (ASTM, 2014c) conditions. SEM images (Figures 18(a) and 18(b)) indicate the presence of ASR gel around the reactive aggregate particle. It was also observed that more abundant cracking developed in those mortar bar specimens. Such cracks had a size of 4 μ m or less (Figure 18(c)), and were not observed in the control specimens. This indicates that cracks are not related to specimen preparation. However, no cracks or ASR gel formation were observed in specimens incorporating the Sheikh Hills aggregate source tested under both ASTM C 227 (ASTM, 2010) and C 1260 (ASTM, 2014c) conditions (Figure 19). Likewise, SEM images of specimens incorporating the Tuguwali and Mach Hills aggregate sources and tested under ASTM C 227 (ASTM, 2010) showed no presence of cracks or ASR gel (Figure 20). Generally, SEM findings identified ASR gel in those specimens incurring higher expansion and exhibiting an associated decay in mechanical properties.

5. Conclusions

This study explored the characterisation of ASR risk in aggregates with slow reactivity with an attempt to define a suitable test method to quantify such a risk. The mineralogy and physical properties of various aggregate sources were characterised. The ASR-related expansion of mortar bars incorporating





Figure 19. SEM images of mortar bar incorporating Sheikh Hills aggregate source: (a) mortar bar under ASTM C 1260 (ASTM, 2014c) condition and (b) mortar bar under ASTM C 227 (ASTM, 2010) condition

such aggregates was explored, along with the associated decay in compressive and flexural strength. SEM imaging was used to capture the microstructural features related to the measured expansion and decay in mechanical properties. The following conclusions can be drawn from the experimental results.

- (a) A petrographic examination of the aggregates explored in the study confirms the presence of reactive minerals (3-77%). However, the reactive minerals were considered to be only slowly reactive.
- (b) The ASTM C 227 (ASTM, 2010) mortar bar expansion for specimens incorporating aggregates from the Sargodha region ranged from 0.05 to 0.07%. However,



(a)



Figure 20. SEM images of mortar bars incorporating (a) Tuguwali and (b) Mach Hills aggregate sources tested under ASTM C 227 (ASTM, 2010) exposure conditions

specimens made with the Jhelum and Kamser aggregate sources exhibited lower expansion of <0.04%, either due to the pessimum effect or due to their non-reactive nature.

- (c) Using the ASTM C 1260 (ASTM, 2014c) test method provided different results, showing all tested sources of aggregates from the Sargodha region to be reactive with an expansion >0.20%.
- (d) The compressive strength of specimens incorporating aggregates from the Sargodha region was more affected

by ASR, especially at later ages. The more reactive aggregates yielded a more significant decrease in compressive strength, as expected.

- (e) The overall reduction in compressive strength for specimens tested under ASTM C 227 (ASTM, 2010) ranged from 22 to 25%, while it ranged from 10 to 22% for identical specimens tested under ASTM C 1260 (ASTM, 2014c) conditions.
- (f) A decrease in flexural strength was observed for Sargodha aggregate sources tested under ASTM C 227 (ASTM, 2010) and ranging from 23 to 29%. Similar specimens incurred flexural strength reduction under ASTM C 1260 (ASTM, 2014c) ranging from 22 to 34%. Specimens incorporating aggregates from the Kamser and Jhelum sources exhibited a smaller decrease in flexural strength (i.e. 8%).
- (g) Specimens included from the Kamser and Jhelum aggregate sources did not incur significant reduction either in compressive strength, or in flexural strength under conditions of exposure conducive to ASR.
- (h) SEM imaging confirmed that the Mach Hills aggregate source has potential ASR. SEM results obtained on the Mach Hills aggregates also confirmed the effectiveness of the ASTM C 1260 (ASTM, 2014c) in better identifying potential ASR in slow reactive aggregates as opposed to ASTM C 227 (ASTM, 2010). In addition, ASTM C 1260 (ASTM, 2014c), which requires less time to conduct, better agreed with the petrographic analysis results.
- (i) The findings of this study should be substantiated with additional testing using the ASTM C 1293 (ASTM, 2015d) concrete prism test. In addition, the effectiveness of using locally available supplementary cementitious materials in mitigating the reactivity of some of the tested aggregates needs dedicated future research.

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How can you contribute?

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