

Chemical and petrographic characterization of aggregates used in the 1st AAR-8 Round Robin Test

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Several rocks commonly used as aggregates contain minerals with Na and/or K in its composition. Plagioclase, K-feldspar, K/Na-feldspar exist in a large number of aggregates but also feldspathoid, some mica, clay minerals, zeolites, amphibole, volcanic glass and unwashed marine sands contain alkalis. Fundamental research has been developed along the last decades related with the solubility of mainly feldspars and mica under aggressive conditions. Aggregates containing these minerals have been investigated due to the suspicion that they might increase the alkalis content in concrete, but also provide Si and Al to the solution, which are highly soluble at high pH conditions.

Alkali release is affected by diverse properties of the aggregate such as particle size, lithological composition, mineral grain size, mineral modal content, total alkali content and crystallographic structure of mineral constituents. Also the degree of alteration, the spatial microstructure, fabric or texture, as well as internal porosity and permeability of the aggregate particle must be considered.

The test method AAR-8 was developed in the scope of RILEM TC 258-AAA activities. The test method, which aims to assess the potential amount of alkalis released by aggregates, is based on the determination of the potential amounts of sodium and potassium ions discharged by aggregates by immersion of portions of rock reduced by grinding to a sand size fraction, in KOH and NaOH solutions, respectively, when in contact with excess of calcium hydroxide.

Five samples of aggregate known to contain alkalis were tested in the AAR-8 test. They correspond to four samples of crystalline rocks of igneous and metamorphic origin, namely phonolite (Canada), cataclasite (Norway), granodiorite (Spain), granite (Portugal) and one sample of sea dredged, basaltic polymictic sand (Iceland).

Particles of the fraction 4.00-2.00 were selected for the petrographic characterization. The thin sections were analysed under optical microscope and the fine-grained rocks were also studied using Scanning Electron Microscope (SEM) with qualitative and semiquantitative microanalysis by Energy Dispersive Spectroscopy (EDS). Also Electron Probe Micro-Analyzer (EPMA) was used to obtain the chemical composition of the volcanic glass in the sample of sand. Major oxides of the samples were obtained by X-ray fluorescence to determine the content in Na₂O, K₂O and Na₂O_{eq}.

The phonolite sample is a fine-grained volcanic rock of intermediate chemical composition showing radiate texture with abundant green clinopyroxene crystals (aegirine-augite), alkali-feldspar, feldspathoids (nepheline) and rare sodalite. Clinopyroxene forms the abundant green to brownish euhedral to prismatic crystals which in some sectors show some preferred orientation. Nepheline is conspicuous and occurs as large anhedral to subhedral crystals forming the groundmass and also as bladed phenocrysts with the characteristic cloudy appearance due to alteration.

The cataclasite sample is composed of K-feldspar, plagioclase, quartz, epidote, chlorite and some apatite. Quartz occurs in strained long crystals, showing ribbons and subgrains. The larger crystals of quartz exhibit undulatory extinction and strain lamellae and there is abundant micro- to cryptocrystalline quartz. Porphyroclasts are composed of quartz and plagioclase with deformation features. Due to deformation, there is an alignment of the main minerals, in particular of epidote grains interbedded with both chlorite and very small crystals of quartz.

The particles of the granodiorite show some variability in the granularity of the rock. It is mainly fine to medium-grained granodiorite composed of K-feldspar (mainly microcline), plagioclase, quartz, muscovite, rare biotite and titanite. The rock is strongly deformed with the crystals of quartz showing

strain lamellae. The borders of the crystals are sutured, with bulging and subgraining. Abundant micro-to cryptocrystalline quartz was identified in the boundaries of the larger crystals.

The granite sample corresponds to fine- to medium-grained granite composed of quartz, plagioclase, K-feldspar, muscovite, biotite, chlorite, apatite, zircon, rutile, tourmaline and opaque minerals. The borders of the quartz crystals are straight to concave-convex. Microcrystalline quartz occurs in the boundaries of the larger crystals. Feldspar forms the larger crystals in the rock. K-feldspar corresponds mainly to orthoclase and microcline, in minor amount, usually showing alteration to clay minerals. Plagioclase is altered to sericite. Biotite occurs in tabular crystals of smaller dimension than those of muscovite and is partially chloritized.

The basalt sea dredged sand sample presents a large variety of lithologic compositions, in which dark particles of basalt *lato sensu* are dominant, containing plagioclase, augite, olivine and opaque minerals. Most of the particles correspond to porphyric glassy basalt *lato sensu*. There are also particles of acidic rock, possibly rhyolite, and some shell fragments. Some of the minerals in the rocks that form the sand are altered to clay minerals. The volcanic glass shows alteration to clay minerals occurring in radial texture of orange-acicular crystals. In some of the sand particles, the vesicles are filled with colourless minerals of zeolite.

The results of the chemical analysis show that the content in alkalis in the sample of phonolite is much higher than in any of the other samples. In addition, the content of Na₂O is much higher than K₂O in the phonolite, the cataclasite and the sea dredged sand and slightly lower for granodiorite and granite.

The results show that most of the aggregates tested release higher quantity of alkalis at 60 °C than at 38 °C, except for cataclasite and granodiorite which present similar results at both temperatures. It was observed that the release of Na₂O is higher than K₂O in all samples, with the exception of granodiorite and granite, which can be attributed to the higher content in K₂O in comparison with Na₂O in the bulk rock composition.

The results obtained in the test follow the tendency of the original chemical composition of each aggregate with regard to the proportions of Na₂O and K₂O released. However, although the alkali content of phonolite is more than five times greater than in dredged sand the Na₂O release is just 1.3 higher for both temperatures. The results suggest that the alkalis contained in the volcanic glass are more susceptible to be release to the solution than those within the crystalline minerals.

The results show that the elementary composition of the aggregates obtained by bulk rock chemical analysis does not necessarily reflect the release potential of the alkalis. This demonstrates the importance of properly identifying the phases in aggregates that have significant release potential such as feldspathoids and volcanic glass.

It was concluded that:

- All the samples are composed by minerals that contain sodium and/or potassium;
- The minerals that compose these aggregates have different susceptibility to release alkalis;
- The mineral composition of samples as well as the size of the crystals, the existence of cracks and the texture of the rocks increase the exposition to the solution used in the tests;
- The phonolite is the one that releases the highest amount of alkalis and also presents much higher alkalis content in bulk rock analysis than the other samples;
- However, the much higher content in alkalis is not reflected in a substantially higher release in alkalis, suggesting that the release of alkalis depends on other factors such as the stability of the mineral or glass in which the alkalis are bound, texture, etc.;
- For most of the samples, the release of alkalis increases with temperature;
- The dredged sand, although presenting a low content of alkalis in the bulk rock analysis, has a relative high release of Na₂O, which suggests that the alkalis contained in the volcanic glass are much easier released than those contained in crystalline minerals;
- The investigation showed that the potential for alkali release was related more closely to mineralogy of the sample than to its alkali content determined by bulk rock chemical analysis.

Keywords: *alkalis release; aggregates; chemical composition; minerals; petrography.*

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