

# ASSESSMENT OF THE ALKALI-REACTIVITY POTENTIAL OF SEDIMENTARY ROCKS

Isabel Fernandes<sup>1,2\*</sup>, Maarten Broekmans<sup>3</sup>, Maria dos Anjos Ribeiro<sup>2,4</sup>, Ian Sims<sup>5</sup>

<sup>1</sup>University of Lisbon, Faculty of Sciences, Department of Geology, Campo Grande, 1749-016 Lisbon, PORTUGAL

<sup>2</sup>ICT, Institute of Earth Sciences, FCUP, PORTUGAL

<sup>3</sup>Geological Survey of Norway - NGU, PO Box 6315 Sluppen, N-7491, Trondheim, NORWAY

<sup>4</sup>University of Porto, Faculty of Sciences, DGAOT, Rua do Campo Alegre, 4169-007 Porto, PORTUGAL

<sup>5</sup>RSK Environment Ltd, 18 Frogmore Road, Hemel Hempstead HP3 9RT, UNITED KINGDOM

## Abstract

The reactive forms of silica present in an aggregate depend on the origin and geological history of the rocks. The detection of specific reactive silica must be focused on characteristics such as the identification of polymorphs, the quantification of microcrystalline to cryptocrystalline quartz, and/or on the deformation manifestations for each aggregate.

In this paper the types of sedimentary rocks usually exploited as aggregates for concrete, such as sandstone, greywacke, chert, siliceous limestone and mudstone, are presented. In addition, the rocks exhibiting low metamorphic grade are included when the sedimentary structure is still preserved and the features of metamorphic conditions are slight. The main characteristics of the sedimentary rocks regarding alkali-aggregate reactions are discussed and the importance of complementary methods for the detection of reactive forms of silica explained.

**Keywords:** aggregates, reactive forms of silica, petrographic examination, sedimentary rocks

## 1 INTRODUCTION

Most supracrustal rocks have components identified as alkali-reactive somewhere, contributing to alkali-silica reaction damaged concrete structures, whereas the same rock types seem to perform as non-reactive elsewhere. Large volumes of sedimentary rocks from quarries as well as sand and gravel from natural sedimentary deposits are exploited all over the world for the manufacture of concrete.

Petrographic examination is the first step of the study when assessing new aggregates for concrete. RILEM AAR-0 gives guidance on the further testing that may be undertaken on the basis of the findings of the RILEM AAR-1.1 [1], petrographic examination procedure, namely laboratory expansion tests with boosted alkali contents and storage of concrete prisms in conditions of elevated temperature and high relative humidity. RILEM AAR-1.1 aims at providing the methodology to be followed in the petrographic analysis of new aggregates or periodically of quarries being exploited, as heterogeneities are commonly found in relatively short distances in the same rock mass.

The recognition of textural features and minerals in very fine grained rocks is usually quite challenging. Therefore, AAR-1.1 has been complemented with a petrographic atlas RILEM AAR-1.2 [2]. For the preparation of the atlas, a large number of samples were analysed and information collated about alkali-reactive rocks all around the world. The atlas aims to assist petrographers with the correct identification of reactivity potential of virgin aggregate materials from igneous, sedimentary, and metamorphic rock types.

The atlas shows micrographs of potentially reactive forms of silica and textural features in confirmed alkali-reactive lithologies, and describes how to identify these in thin-sections under the optical microscope. Reliable identification of very fine-grained rocks benefits from supplementary methods, most notably SEM-EDS in polished thin-sections, bulk whole-rock chemical analysis, or powder X-ray diffraction – XRD.

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\* Correspondence to: [mifernandes@fc.ul.pt](mailto:mifernandes@fc.ul.pt)

The present paper presents a summary of the main characteristics of sedimentary rocks. A list of the most common aggregates is included as well as of the forms of silica that can be found in different types of rocks. Overall, it appears that, on a global basis, cryptocrystalline quartz is the most frequent reactive component. However, specific groups of rocks also contain silica polymorphs thought to be involved in alkali-silica reactions.

## 2 MATERIALS AND METHODS

### 2.1 Materials

Different materials were studied, including rock fragments, gravel specimens, post-mortem concrete prisms from completed expansion testing, and pieces of concrete from damaged structures. The alkali-reactivity of all rock materials presented in the atlas had been prior confirmed by reliable laboratory expansion testing, or observed in field structures.

Thin sections of 23.0×48.0×0.030 mm were manually prepared using standard routine procedures, some using yellow fluorescent dye to facilitate observation of fractures and voids, and some were polished for later analysis by SEM-EDS.

### 2.2 Methods for assessment and analysis

The thin sections were studied in a Nikon Eclipse E400POL petrographic microscope in plane (PPL) and cross polarized light (XPL). Photomicrographs were acquired using a Zeiss MRc5 camera and Axiovision 4.8 software.

For the study of the samples, the methodologies used in petrography were followed aiming at the identification of the mineral composition and the texture of the rock. The reduced sizes of the samples of rock may raise questions regarding the representativeness of the samples, most notably in places where heterogeneous natural sedimentary deposits are common. The gravel contained polymictic aggregate materials with particles from a variety of rock types and textures. Due to the difficulty of identifying potentially alkali-reactive lithologies, individual particles of interest were selected in concrete samples post-mortem after completed expansion testing.

The polished thin sections were sputter-coated with carbon under vacuum in a VG MICROTTECH E6700/T800 instrument for analysis in a JEOL JSM-6301F scanning electron microscope instrument, equipped with a field-emission gun (FEG) and a NORAN VOYAGER energy dispersive spectrometer (EDS). The presence of quartz in very fine grained rocks was confirmed by element mapping using EDS.

## 3 RESULTS

For the atlas, a total of 56 samples of sedimentary rocks were characterised, listed in Table 1 in decreasing order of abundance, revealing the predominance of sandstone.

Regarding the forms of silica identified as potentially reactive, microcrystalline quartz (<100 µm) occurs in sandstone, greywacke and chert; cryptocrystalline quartz (<10 µm) is the main constituent of chert and of the silica fraction in mudstone, but may also be abundant in the matrix of sandstones and greywackes as well as finely dispersed in some siliceous limestones. Other forms of silica, such as opal and chalcedony (and possibly moganite), are most common in chert/flint, and may also occur in the cement of some sandstones consolidating the detrital/clastic grains into rock.

According to traditional nomenclature, sedimentary rocks can be divided into three main groups: detritic or clastic sediments (sandstone, greywacke and mudstone from above list), of chemical origin by precipitation from solution (chert and siliceous limestone), or of organic origin containing plant or animal remains and/or secretions (chert and siliceous limestones). Compaction and consolidation occur in the upper crust, and by definition exclude formation of metamorphic minerals. However, in this study, the rocks which were submitted to very low metamorphic conditions and where the sedimentary features were dominant, were kept in the sedimentary group.

Sandstone, as applied here, refers to arenite with less than 15vol% matrix [3][4]. The samples analysed are mainly composed of rounded clasts of individual quartz grains, and minor feldspar. Frequently, clasts also comprise quartzite, chert, and a variety of other lithic fragments, containing deformed quartz with sutured boundaries and cryptocrystalline quartz, respectively. Structural/textural features attributed to compaction/diagenesis are commonly observed, most notably pressure dissolution and/or stylolite formation perpendicular to the main (geological) stress direction, and formation of microcrystalline quartz along grain boundaries in stress-shadows. The texture showing stretched grains of quartz is common to a number of well sorted sandstones with scarce or absent

rock cement. Alkali-reactivity of sandstones appear most frequently associated with the rock cement (in rare cases containing opal and chalcedony), or scarce matrix filling interstitial space. Detailed study of thin-sections of ASR damaged field concrete reveal that ASR cracks with reaction product tend to concentrate along an inter-granular path, separating clasts rather than fracturing, though off-shoots may enter large grains  $>100\ \mu\text{m}$  (Figure 1). Matrix present in the interstitial spaces is normally composed of clay minerals, fine-grained sericite and possibly cryptocrystalline quartz, usually both discernible under optical microscope.

One peculiar example of sandstone illustrating the relevance of supplementary analytical methods is the alkali-reactive glauconitic sandstone from northern Germany. Detailed assessment of polished specimen using SEM reveals the presence of opaline cement and high rock porosity (in non-fluorescent thin-sections). In addition to glauconite, ‘pompoms’ of very thin acicular silica crystals are commonly found in interstices, which could be identified as radiolarian remains (Figure 2).

According to their definition, greywackes are immature sedimentary rocks with variable clast composition (quartz, feldspar, lithic) and matrix content 15-75vol% [3][4]. The samples analysed for the atlas exhibit a wide variability in granularity, from very fine grained to medium size grained. As already mentioned for the sandstones, greywacke clasts may also comprise (deformed) metamorphic rocks and chert. Some samples show signs of incipient prograde metamorphism, with noticeable development of cleavage. The main alkali-reactive components are found in the greywacke rock matrix, in most of the cases with abundant clay minerals and cryptocrystalline quartz (Figure 3). Detailed study of field concrete samples confirms that ASR cracks tend to follow the matrix in between coarser-grained clasts, sub-parallel to a developing foliation if present.

Chert/flint composed of cryptocrystalline quartz with chalcedony, opaline silica and fossil remains [5], is widespread and locally abundant in regions with outcropping Cretaceous rocks, notably NW Europe. Chert/flint particles commonly occur as minor constituent in limestone samples. Reliable identification of opaline silica and moganite in such fine-grained rocks is difficult and normally requires supplementary methods [6]. Texture, porosity and silica grain size is variable within and among chert/flint particles (Figure 4), but is effectively visualized in thin-sections impregnated with fluorescent dye.

Detrital quartz and other silicates, as well as neogenic clays in siliceous limestone, are micro- to cryptocrystalline and well beyond optical resolution of a petrographic microscope. Example of a siliceous limestone is shown in Figure 5. Element mapping by EDS reveals the presence of dolomite in characteristic rhombs, and of silica and clay minerals finely interspersed throughout the carbonate matrix. The combination of Si-Al-K maps suggest that some detrital feldspar may be present.

#### 4 DISCUSSION

Applying the correct lithological name to a sample material of unknown or undisclosed geological origin may pose a great challenge, even to an experienced petrographer. The atlas and this paper apply the most recent nomenclature schemes and glossaries such as Hallsworth and Knox [7] for sediments, in addition to local designations more common in the respective region of origin, as applicable. Thus, “sandstone” was preferred over ‘arenite’ or ‘psammite’, due to its greater popularity in alkali-silica reaction literature. The second source of ambiguity regarding application of correct nomenclature relates to the in fact arbitrary distinction from metamorphic rocks. After all, the transition of a well-compacted into a low-grade metamorphic quartzite is gradual, with a large overlap between the two. In the present paper as for the atlas, the distinction is based on the predominant characteristic. Thus, a sedimentary rock has a recognizable protolith and displays very subordinate blastesis (=formation of metamorphic minerals from sedimentary/diagenetic precursors).

The reactivity potential of sedimentary rocks has been subject of extensive study (eg. [8][9][10][11][12]). Some types of sedimentary rocks, which are used as coarse and fine aggregates for concrete all over the world, seem consistently identified as potentially reactive. Examples such as the crushed greywacke from the UK [13], the siliceous limestone from the Spratt quarry (Bobcaygeon Formation) [14][15][16] and from the Tournaisian Formation [11], and the chert and opaline lithologies in aggregates from northwestern Europe ([17][18][19][20][21]) and the Appalachians [22],[23] are well known. However, the reactivity of these rocks is variable depending on the mineral composition, texture, and internal porosity.

The most common clastic sedimentary rocks used as aggregate for concrete are sandstone and greywacke and, to a much lower extent, certain types of mudstone. These rocks are predominantly composed of siliceous clasts consolidated by diagenetic compaction and cementation. Compaction may induce pressure-solution, with dissolution of minerals and fluid transport generated by the differential stress. The shapes and sizes of the detrital and diagenetic components define the fabric of

the rock. According to Renard et al [24], this type of deformation at a grain scale is commonly attributed to the ‘water film diffusion’ mechanism, consisting of three successive steps: (i) dissolution at the grain interface, (ii) diffusion of solutes along an adsorbed water film along grain interstices, and (iii) precipitation on grain surfaces in open pore space. The slowest step controls the rate of the overall progress. This process is observed in well sorted sandstones with clast indentation in the main stress direction revealed by one quartz grain penetrating into another forming a curved grain contact, and deposition of silica in the direction of the lower stress, commonly seen as epitaxial overgrowths on detrital clasts with their original periphery decorated by impurities (e.g. [25][26]). Where pressure-solution is more intense, grain contacts become serrate (as in Figure 1a), and ultimately stylolitic (*‘stylol’*= Gr. pillar). Rock cement may be composed of neogenic silica, carbonate, limonite (ie. ‘iron [hydr-]oxides’) deposited in the interstices between detrital fragments, consolidating the clasts into solid rock. Embedded in concrete as aggregate, relic interstitial space may still be open and connected, facilitating access of concrete pore fluid to the rock particle’s interior. Matrix consists of detrital particles <30 µm and is mainly composed of clay minerals with micro- to cryptocrystalline quartz. Rocks subject to prograde metamorphism at low-grade conditions may contain chlorite, sometimes intercalated with white mica.

Examples of alkali-reactive sandstones have been published (e.g. [18][19][27][28][29]), identifying the neogenic siliceous cement as the cause of reactivity. A detailed study by Rivard et al [28] on reaction rims in NE Canadian concrete made with porous Potsdam sandstone confirmed alkali-reactivity and quartz dissolution along the periphery of the detrital fragments in the aggregate particle interior. Advanced dissolution of the intergranular rock cement eventually leads to progressive disintegration of the sandstone particle. Marfil et al [27] studied a sandstone mainly composed of well-sorted, equidimensional, subrounded to irregular quartz particles cemented by chalcedony and opal-CT. Post-mortem petrography on laboratory specimen after completed expansion testing showed the rock’s siliceous cement was corroded, confirming the rock cement as the alkali-reactive component. Sandstones, greywackes and argillites present in gravel in the Sudbury area of Canada are also considered to be potentially reactive to alkalis (e.g. [30]). In the UK and Canada (Nova Scotia, New Brunswick), greywacke was found to have given deleterious reactions in concrete structures [13][31][32][33]. Some potentially reactive varieties of greywackes are also found in Germany, Sweden, USA and Norway [34][35][36]. In South Africa, a number of structures were diagnosed with ASR in South West Cape region due to the use of Malmesbury Group metasediments which contain greywacke [8][9]. In their work on Dutch aggregates, Broekmans & Jansen 1998 [18], Broekmans [19] and Nijland et al [29] found that alkali-reactive particles from damaged field structures are most commonly porous chert with chalcedony and/or opaline silica, and ‘impure’ sandstones, in approximately equal proportions.

Chert (francophone: *‘silex’*) is a dense, very abrasion-resistant non-clastic siliceous sedimentary rock containing more than 50vol% micro- to cryptocrystalline quartz with porosity <10vol% [7][37]. The term flint applies to the nodular variety of (bedded) chert [38], but as the distinction is obliterated in particulate (aggregate) materials, chert applies as the common denominator [5]. Chert can be composed of authigenic silica due to precipitation from hydrous solution, or alternatively may be of secondary origin [6]. Primary chert forms from siliceous shells, sponge spiculae, radiolarians and diatoms. Chert of secondary origin results from the replacement of carbonate rocks in nodules (flint), or entire layers. Fibrous chalcedony typically fills voids, and clay minerals, pyrite, iron oxides and organic matter may also be present. A number of papers have been written addressing the performance of chert in concrete aggregate.

Arguably due to its very fine-grained nature and hard to distinguish ‘silica’ mineralogy, chert is often characterized by supplementary methods, in particular powder X-ray diffraction – PXRD. To facilitate comparison between different (sets of) samples, (partial) diffractograms are often recalculated as Quartz Crystallinity Index – QCI values, following the method originally described by Murata and Norman [39]. However, a recent critical assessment by Marinoni & Broekmans [40] revealed that this method suffers from a number of fundamental shortcomings, including lacking descriptions of sample and specimen preparation as well as of the reference material used for internal calibration (ie. prerequisites any analytical standard). Most important, their proposed index is not attributed to any specific crystal-structural feature, an explicit requirement elsewhere (see eg. [41]).

In general, a crystallinity index is a composite number representing contributions from crystallite size and shape, domain size and shape, and microstrain (p222, [40]). Individual contributions from these QCI-constituents will certainly vary per lithology, but variation within a single lithology from one specific locality or stratigraphic unit might be less and could possibly allow comparison within the sample set (eg. [42][43]). QCI values for chert of Dutch and Appalachian origin

(ie. the original samples of Kneller [22]) did not reveal any consistent correlation with expansion data [19]. Furthermore, Murata & Norman [28] implicitly use non-monochromated bulk  $\text{CuK}\alpha$  radiation (of wavelength  $\lambda=1.54184\text{\AA}$ ), whose two components  $\text{CuK}\alpha_1$  ( $\lambda=1.54056\text{\AA}$ ) and  $\text{CuK}\alpha_2$  ( $\lambda=1.54439\text{\AA}$ ) double (with fixed intensity ratio determined by quantum physics) the triplet centered at  $67.74^\circ 2\theta$  into a sextuplet, as applied in the original method. To eliminate this peak-doubling and achieve higher accuracy, McNally et al [44] use monochromatic  $\text{CuK}\alpha_1$  radiation. However, this also instantly invalidates their QCI values (as  $\text{CuK}\alpha_2$ -peaks are essential in their determination cf. Murata & Norman [28]), and consequently, their interpretations.

In summary, the highly incomplete description of the QCI-procedure, the omission of attributing values to a specific crystal-structural feature, and the inconsistent relation to alkali-reactivity potential makes the original method of Murata & Norman [39] unsuitable for routine ASR assessment, or indeed 'quartz crystallinity' in general [40]. On the other hand, the reactivity of chert is thought to be related with the porosity as explained by Barisone & Restivo [45] who concluded that Italian cherts are less reactive than from NW Europe, namely from Great Britain, Netherlands, Germany, and Denmark. The authors attribute the higher reactivity to a higher degree of crystal-structural disorder, and/or to a higher porosity due to different exposure conditions during weathering and transport. Their observation seems to coincide with the consistent presence of moganite in Cretaceous cherts from NW Europe [26], a somewhat obscure yet officially IMA-approved silica polymorph. Remarkably, sea-dredged porous chert that has resided on the Northsea floor for  $\sim 10\text{ka}$  does not react in expansion testing, which is attributed to leaching of soluble silica species [unpublished data, MATMB]. However, recent data on field concrete may contradict this again. In summary, chert should always be regarded as potentially deleterious in concrete [46] but should be studied in detail due to possible pessimum effect.

Regarding argillaceous dolomitic limestones, work has been developed to clarify the origin of reactivity of Kingston limestone (Gull River Formation) and cryptocrystalline quartz present in the carbonate mud has been identified as deleterious [47][48]. Previous work carried out in Canada on the Neuville Formation (Quebec), the western part of Newfoundland and on the Bobcaygeon Formation (Spratt quarry; Ontario) showed that micro- to cryptocrystalline quartz present in the carbonate mud was the cause of reactivity [49][50][51][52][49]. These formations are similar to the one reported in [11]. The identification of the reactive silica in such cases usually requires extraction through selective dissolution in excess acid and XRD analysis of the insoluble residue, complemented by SEM/EDS to study the texture and spatial distribution. In a detailed assessment of Spratt's aggregate, Rogers & McDonald [15] note that this Middle Ordovician rock is medium grey colored, fine to coarse crystalline limestone with about 9wt%  $\text{SiO}_2$ , also comprising chalcedony. However, little silica is observed in thin-section because it is very fine grained and inconspicuous in the highly birefringent calcite matrix. The micritic limestones containing phyllosilicates, minor chert, and cryptocrystalline quartz from the Tournaisian Formation produced deleterious reactions in a number of concrete structures. Guédon-Dubied et al [11] identified two sub-units in the limestone deposit. The lower unit consists of micritic limestone, clayey in the basis, impregnated with diagenetic silica and with abundant fossil fragments (bioclasts). The upper unit is composed of clayey micrites also impregnated with diagenetic silica and containing pseudomorphs of gypsum, anhydrite and black siliceous nodules, composed of chalcedony and opal. Both the nodular and diagenetic silica (microscopic) may be the cause of the rock reactivity, as confirmed by expansion laboratory tests.

## 5 CONCLUSIONS

This paper presents a summary of the main reactive components observed in detrital/clastic and chemical sedimentary rocks. A number of samples from different locations around the world were studied to obtain information on the most common sedimentary rock types, and their potentially reactive components.

- Optical thin-section petrography conforming to RILEM AAR 1.1 is considered the prime assessment method for aggregate materials, being most cost- and time-effective.
- Supplementary methods are essential for the reliable identification of cryptocrystalline silica, especially when dispersed throughout the lithology.
- Identification of reactive mineral constituents is complex, and operator skill and experience remain quintessential for reliable assessment by thin-section petrography.
- Micro- to cryptocrystalline quartz are the most common reactive component in sedimentary rocks.
- In coarser-grained rocks such as sandstones and greywackes, reactive mineral constituents usually occur in the rock cement, or throughout the finer-grained matrix.

- Aggregate materials are to be classified according to local criteria, based on local experience with ASR-damage in field structures and local geology.
- Rocks predominantly composed of cryptocrystalline quartz like eg. chert/flint are to be considered potentially reactive, but performance should be confirmed by expansion testing as eg. porosity may affect alkali-reactivity.
- Chert/flint may show pessimum effect which can be studied by performing expansion laboratory tests.

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TABLE 1: List of the sedimentary samples studied.

<b>lithology</b>	<b>number of samples</b>	<b>percent</b>
sandstone	26	45
chert/flint	12	21
greywacke	10	18
siliceous limestone	7	14
mudstone	1	2
<b>SUM</b>	<b>56</b>	<b>100</b>

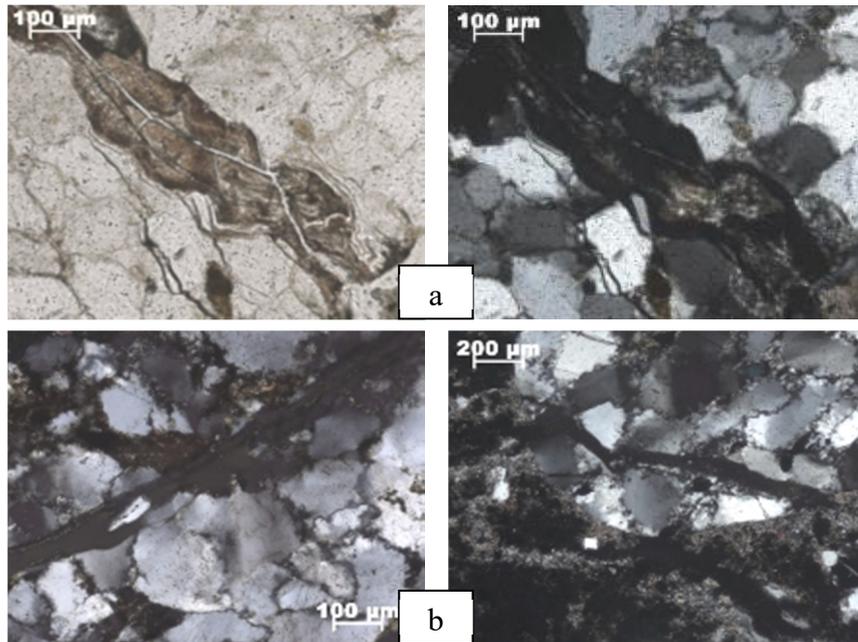


FIGURE 1: Example of sandstones: a) grain-supported sandstone with pressure-solution features such as elongation, sutured boundaries and deposition of quartz in the direction of lower stress (PPL and XPL); b) crack filled with alkali-silica gel; the crack develops mainly along the interstitial spaces between the clasts; some thinner cracks develop inside the clasts, parallel to the main crack (both in XPL).

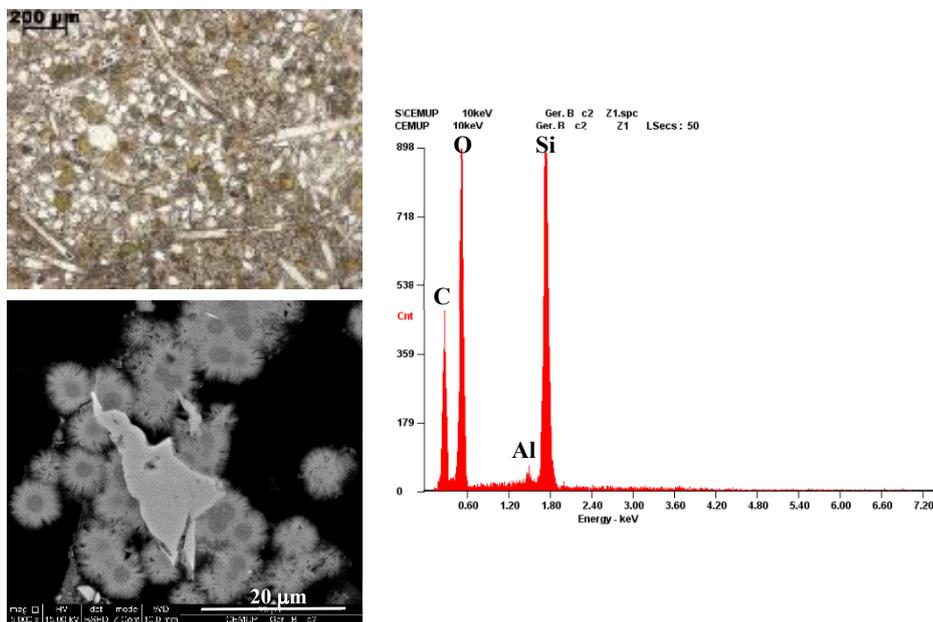


FIGURE 2: Opaline sandstone composed of glauconite (greenish) and sponge spicules. Photomicrographs in PPL (left). The rock is highly porous as can be observed in the SEM image (centre). The rosette-like crystals of pure silica may correspond to radiolarian or to an inorganic structure formed by silica (Si and oxygen are the main components). Image by SEM-BSE (BSE - back-scatter electrons) and spectrum obtained by EDS (right).

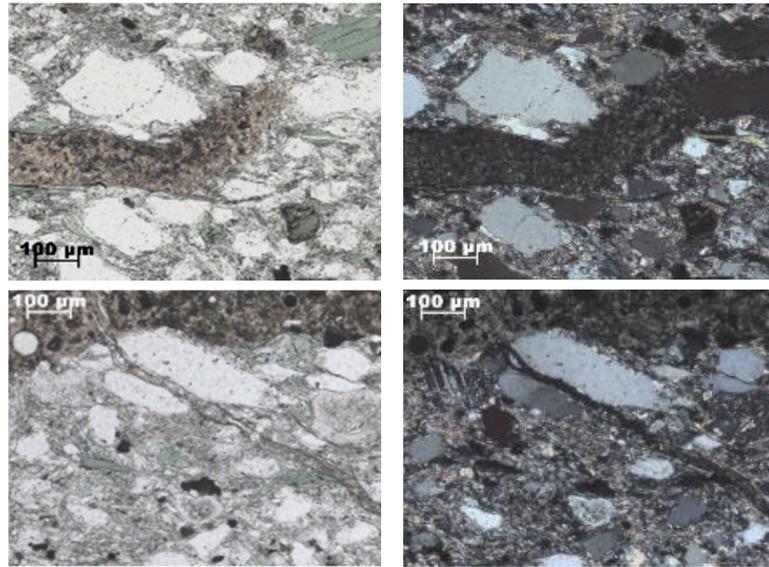


FIGURE 3: Examples of ASR in greywacke. The clasts are dominantly composed of monocrystalline quartz and feldspar. The abundant matrix is composed of cryptocrystalline quartz, sericite and chlorite (and possibly some clay minerals). The images show the preferential path of the cracks along the porous interstitial space containing matrix (left, in PPL; right, in XPL).

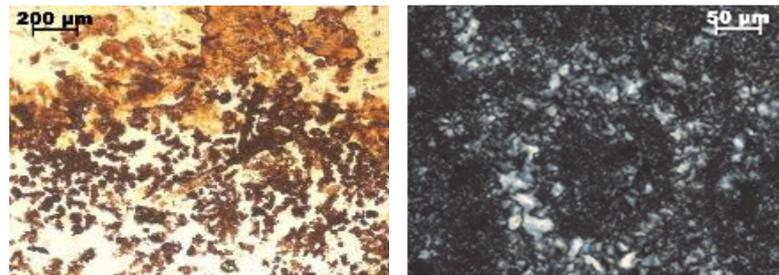


FIGURE 4: Chert showing variable porosity (light yellow colour of the fluorescence dye) and limonite staining in dendritic distribution (PPL) and fossil remains replaced by feather-like chalcedony (XPL).

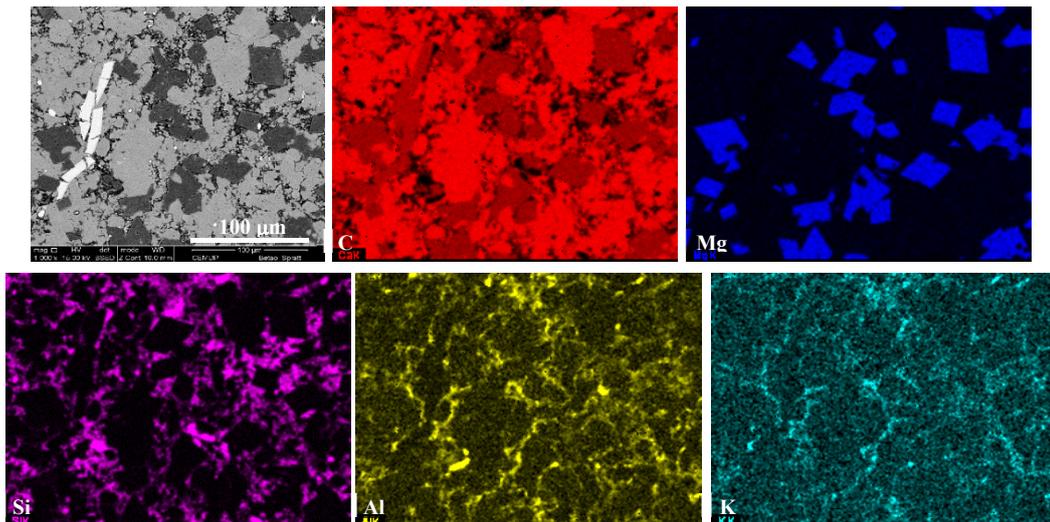


FIGURE 5: SEM-EDS element maps revealing porosity and presence of two different carbonate minerals, namely calcite and dolomite (Ca and Mg) (b), and presence of very fine-grained silica (Si) as well as clay minerals (Si-Al) and possibly some detrital feldspar (Si-Al-K). Image on the top/left, in BSE, with scale bar.