RILEM PETROGRAPHIC METHOD FOR ANALYSIS OF ALKALI REACTIVE CONCRETE AGGREGATES

Viggo Jensen
SINTEF Civil and Environmental Engineering, N-7465, Trondheim, Norway

Gabriel Lorenzi
ISSEP, Rue du Chera 200, B-4000 Liege 1, Belgium

Abstract

The paper present the RILEM petrographic method for visual recognition and quantification of rock and mineral constituents with special emphasis on their alkali reactivity. It is assumed that alkali reactive constituents are known e.g. by field experience and recognizable by visual examination and/or thin section examination. The petrographer shall be competent to recognize unambiguously the entire relevant mineral constituents and reactive constituents in the sample. The RILEM method suggests two procedures that can be followed, namely method 1 which is used when reactive constituents are easily identified by the visual observation of particles, and method 2 which is used when identification and quantification has to be carried out by point counting in thin sections. Method 2 is able to give a full petrographic composition of rock and mineral constituents of the material concerned. For aggregates where the reactive mineral constituents can not be identified by method 1 or 2, complementary microanalysis methods are recommended e.g. microprobe analysis, chemical analysis, XRD analysis etc. Alkali reactive constituents and their limit values should be restricted to follow national or regional practices and specifications. The paper gives four examples on precision test by use of the RILEM method. It can be concluded that the RILEM method is acceptable reproducible when the analysis is carried out by qualified personnel with good knowledge of alkali reactive aggregates.

1. Background

A working group under the direction of RILEM Committee TC 106 has prepared the RILEM method for petrographic analysis of alkali reactive aggregates. The work is now in a final phase and a final draft will be presented for the committee in June 1999, **RILEM petrographic method draft no. 9 (1)**. The paper presents the petrographic method according to draft no 9. It has to be remembered that the RILEM committee not has accepted the draft and therefore changes might occur in a future petrographic method.

2. Scope

The RILEM method specifies a general procedure for the petrographic examination of concrete aggregates, to identify rock types and minerals, which might react with alkalis derived from the concrete. The method, including sampling, is intended to be used for the examination of natural aggregates including sand, coarse gravel, all-in and crushed rock aggregates. The method can be also used to quantify the amounts of various mineral and rock types where required.

The petrographic analysis is the first step for assessment of potential reactivity of concrete aggregates. In some countries, petrographic analysis is mandatory for assessment of reactivity, while in other countries, it is only recommended as a screening test. In such countries the results should be assessed by other methods e.g. RILEM method "Ultra accelerated mortar bar test" and "Concrete prism test" to give the complete answer.

3. Assumptions and limitations

The RILEM petrographic method is based on the following assumptions:

- Alkali reactivity of rock and minerals as well as petrographic descriptions of the same are known and available for the investigator
- Alkali reactive constituents and minerals are visible by the naked eye, stereoscopic microscope and/or thin section examination
- The list and acceptance of alkali reactive rocks and minerals, limit values and pessimum relations vary between countries. Therefore, national practice and recommendations always shall be followed where available.

Information on alkali reactivity should be accessible for the analyst e.g. any recommendations, specifications, petrographic descriptions of reactive aggregates, field experiences, laboratory results and other information of importance's use for the assessment of reactivity. Alkali reactive aggregates consist of many different rock and mineral types. There is strong evidence that apparently similar rock types can vary greatly in reactivity depending on their genesis and geographical location. As mentioned, acceptance and experience of reactive constituents is different between countries, and thus, determination of reactive constituents should where possible follow national or regional experiences, recommendations and specifications.

It is very important that the petrographic analysis is carried out by a qualified geologist or petrographer with experience of materials used for concrete and good knowledge of alkali reactive aggregates and minerals.

Generally for volcanic rocks, chert (flint) and certain limestone are the reactive constituents are amorphous and/or cryptocrystalline. In such cases petrographic analyses e.g. thin section examination often is not sufficient and should be supplemented or replaced by other test methods. The RILEM method gives short descriptions of the most common supplementary analysis methods for the assessment of alkali reactivity (ANNEX)

In cases where the knowledge of reactivity of aggregates is not known, is not established or not available for the petrographer the RILEM method cannot be used for assessment of alkali reactivity. It is then recommenced to collect relevant information or possible start up investigations to obtain knowledge of the potential reactivity of concrete aggregates. The RILEM method gives guidance how to obtain knowledge of alkali reactivity (ANNEX).

4. Nomenclature

Internationally acknowledged nomenclature for geological classification of rock - and minerals shall be used. When local names of aggregates are in common use, this should also be given in brackets. The RILEM method gives suggestions for naming and classifying aggregates for igneous rocks, metamorphic rocks, sedimentary rocks and artificial rocks. It has to be remembered that the petrographic analysis normally is carried out on small fragments and aggregate particles compared to field conditions where additional geological information on large scale is available. Generally, the smaller particles/fragments give lesser information about origin and genesis for naming and classifying aggregates. For particles in the fine sand fraction naming and classifying mostly are based on mineralogical composition rather than texture and mineralogical composition.

The following nomenclature suggests the use of common rock and mineral names, ***preferred name***, accepted by most geologists around the world. However, because some fragments/particles can not be satisfactory classified the rock should be given a ***mineralogical name*** based on the mineralogical composition. In other cases it is more convenient to name the fragment/particles with a ***genetic name*** e.g. volcanic rock etc. Special and local names, mineralogy as well as additional important information should be given in a bracket after the name of the aggregate. Tables in ANNEX suggest preferred names, mineralogical names and genetic names for the main groups, namely igneous rocks, metamorphic rocks, sedimentary rocks and artificial rocks. Minerals should be named by its mineralogical names eventually with supplementary information in a bracket.

For natural aggregates with many different rock and mineral types it is often convenient to have more than one rock type in a “counting group”. This to reduce the number of aggregate groups during the counting procedure. However, reactive rock and minerals should not be grouped but recorded individual. Mineral particles should counted and named by mineralogical name e.g. quartz. Based on local knowledge and specifications the reactivity of aggregates should be marked as: *reactive, potentially reactive, innocuous* or *unknown reactive.* Table 1 gives an example of point counting according to RILEM method 2.

Table 1: Glacio-fluvial 0-4 mm sand from Northern Norway

Rock and mineral composition of sand	Vol%	AAR
Sandstone	66	reactive
Siltstone and claystone	18	reactive
Cataclasite	5	reactive
Mafic rocks (mostly amphibolite)	4	innocuous
Granite and gneiss	4	innocuous
Feldspathic rock/feldspar/diorite	2	innocuous
Quartzite	1	innocuous

Evaluation of reactivity: *The content of reactive rocks is 89 volume percent. According to Norwegian recommendations (DGB) the sand should be classified as reactive with respect to AAR.*

Results are given as volume percent (vol. %) of rock and mineral composition. Note the use of both preferred geological names and mineralogical names and that more than one rock type is included in a counting group. Alkali reactivity (AAR) is assessed according to Norwegian experience and requirement (DGB – "Declaration- and Approval of Concrete Aggregate, 1993). Limit value of reactive aggregates is 20 % volume in Norway.

5 Sampling and preparation in the laboratory

Sampling should be carried out in accordance with the procedures described in European CEN standard prEN 932-1, American standard ASTM D75 or similar standards. The aggregate sample shall be taken from processed material and shall be representative of the aggregates as used in the concrete. It is recommended that a qualitative examination of laboratory samples is carried out as first step in the examination. The method also gives guidance for splitting and sieving of samples.

For coarse aggregates, which it is intended to examine by the point counting method, reduction of particle size is to be carried by crushing. Crushing shall be applied in steps to avoid the production of flaky particles and unnecessary disintegration of aggregates. Sieve the crushed sample and use the 2-4 mm fraction for preparation of thin sections.

6 Procedures

6.1 Generally

Where the assumptions and limitations are obtained a petrographic analysis can be carried out. The method can be used for both qualitative and quantitative determination of rock and mineral composition of natural sand, coarse gravel, all-in and crushed rock used for concrete aggregates. The petrographic analysis may be carried out by two methods, method 1: "Simplified petrographic analysis" or method 2: "Point counting on thin sections". Method 1 is used where alkali reactive rock/mineral constituents are easily identified by the naked eye or stereoscopic microscope or where it is relatively easy to group aggregate constituents on the basis of colour, shape or texture. It may be necessary to study thin sections of selected aggregates to enable full identification of the material. On the other hand, when alkali reactive rock/mineral constituents cannot be identified, method 2 shall be employed.

6.2 Method 1: Simplified petrographic analysis

The petrographic examination has to be carried out on at least one size fraction of the fine aggregate and the coarse aggregate respectively. For fine aggregate it is recommended that the 2-4 mm size fraction is examined. This method is also used for sand, coarse gravel, all-in and crushed rock.

Examination of aggregate particles should be carried out by use of hand lens or stereoscopic microscope and in some samples by scratching for hardness or the use of weak acid for testing carbonate minerals. Some particular aggregate staining methods are useful for identification of certain constituents e.g. blue staining for identification of porous flint.

Aggregate particles from each size fraction are divided into individual rock/mineral groups by hand sorting. As a minimum requirement, this grouping must include one group designated as innocuous aggregates and one group designated as reactive aggregates. It is recommended to divide the aggregate particles into more specified groups of constituents based on an assessment of grain form, colour, texture rock/mineral type, and/or classification of the aggregates. In samples where the reactivity of certain aggregates is difficult to assess, a group designated, as potentially reactive aggregates should be included. Where there is concern over the identification of a particular rock type it is recommended that thin sections of representative particles are prepared to assist in the identification (e.g. dense and fine grained rock types).

Aggregate particles, in each group, are counted and the percentages are calculated. When more than one size fraction is counted, the average of all the counted fractions shall be calculated. More precise results are obtained by correlating each size fraction with the weight % of the fraction. Alternatively, results can be given as weight percentage by weighing each aggregate group and relating these to the total size fraction.

The minimum number of particles to be examined and counted depends on the percentage of rock/minerals of interest and the confidence limits required. The RILEM method (in ANNEX) gives a chart for estimating errors of counted particles of interest versus total counted particles valid for the 95 % confidence interval.

6.3 Method 2: Point counting on thin sections.

When reactive aggregates cannot be assessed by method 1, the point counting of thin sections shall be used. The method is mostly used for polymictic aggregates. In cases, where doubt about the homogeneity of a monomictic aggregates exist, method 2 is recommended to be used.

Quartering or dividing shall obtain a representative test portion of each size fraction. The sample shall be consolidated with resin prior to the preparation of the thin sections. Where reactive aggregates are porous, thin sections should be prepared by vacuum impregnation with epoxy containing the fluorescent dye (e.g. Hudson Yellow). The porosity of constituents may then be identified under the petrographic microscope by fluorescence.

The precision of the point counting depends on representatively of the test portion, the number of particles in the thin sections and number of points counted. To obtain a sufficient number of particles in standard size thin sections it is recommended that more than one thin section are used. For fine aggregate (sand) it is recommended to use a minimum of two thin sections containing representative material from the fraction 2-4 mm, and one thin section with material from the fraction 0-2 mm. For slow/late-expanding type aggregates where reaction is caused by the more coarse aggregates then thin sections from the 1-2mm and the2-4mm fractions should be prepared. For crushed coarse gravel and rock, it is recommended that a minimum of two thin sections containing material from 2-4 mm fraction should to be examined.

The point counting is carried out by traverses in regular increments in two directions to simulate the imaginary grid and give the point to point coverage of the section. It is important that point counting covers the whole thin section. Point counting can be carried out by use of microscope stage attachments or electro-mechanical stage activated by a tabulator. Alternatively a counting graticule placed within the eyepiece of the microscope can be used.

During the point counting, the operator must identify all rocks and minerals located under the cross hairs.

The point counting aims to quantify reactive aggregate and mineral constituents present within an aggregate. All cross points placed within one particle e.g. a sandstone should therefore be recorded as the same. When particles consist of more than one type of rock e.g. sandstone with quartz veining the cross point falling onto the sandstone should be recorded as sandstone, and cross points falling into quartz vein should be recorded as quartz-vein material.

It is recommenced that a minimum of 1000 points exclusive points falling onto the resin are counted for the 2-4 mm fraction (two thin sections) and 1000 points on the 0-2 mm or 1-2 mm fraction (one thin section).

The number of points recorded from each rock/mineral group is related to the total number of points counted excluding points within the resin. The average percentage is calculated and results from each rock/mineral group are given as a whole volume percentage. As a minimum requirement, results are given as percentages of reactive rocks and innocuous rocks.

The precision of the point counting method depends on several parameters. The representative nature of the test portion, the petrographers ability to obtain the correct classification and grouping of rocks and minerals, the number of particles in thin sections, the number of points counted, and the proportion of constituents of interest within the sample.

7 Cases

Petrographic analyses have been in use in many countries but is often carried out on different ways. Therefore the results often are difficult to compare. The following examples of precision tests are all carried out by similar procedures as RILEM method 2.

7.1 Parallel testing on natural aggregates from Northern Norway

The parallel testing between SINTEF and NGU (The Norwegian Geological Survey) was carried out on 47 samples from the most important natural aggregate deposits in Northern Norway, **Jensen (2)**. Procedures followed RILEM method 2 that is similar to the Norwegian DGB method. SINTEF had at that time carried out petrographic analyses on a commercial basis for the last 5 years. Moreover, knowledge of reactive aggregates was bases on research carried out by SINTEF, **Jensen (3).** Before testing, personnel from NGU were educated to identify and classify Norwegian reactive aggregates in thin sections. After about a week intensive education in front of the microscope 6 “pilot point counting tests” were carried out with aims to inter-calibrate results and discuss deviations. Hereafter, the final parallel testing of the 47 samples were carried out.

Figure 1 shows the results and correlation between SINTEF and NGU. Note the excellent correlation between the laboratories. This clearly demonstrates that education of personnel (which must have a fundamental knowledge of geology and petrography) is very important and that the method is reproducible when carried out by qualified personnel.

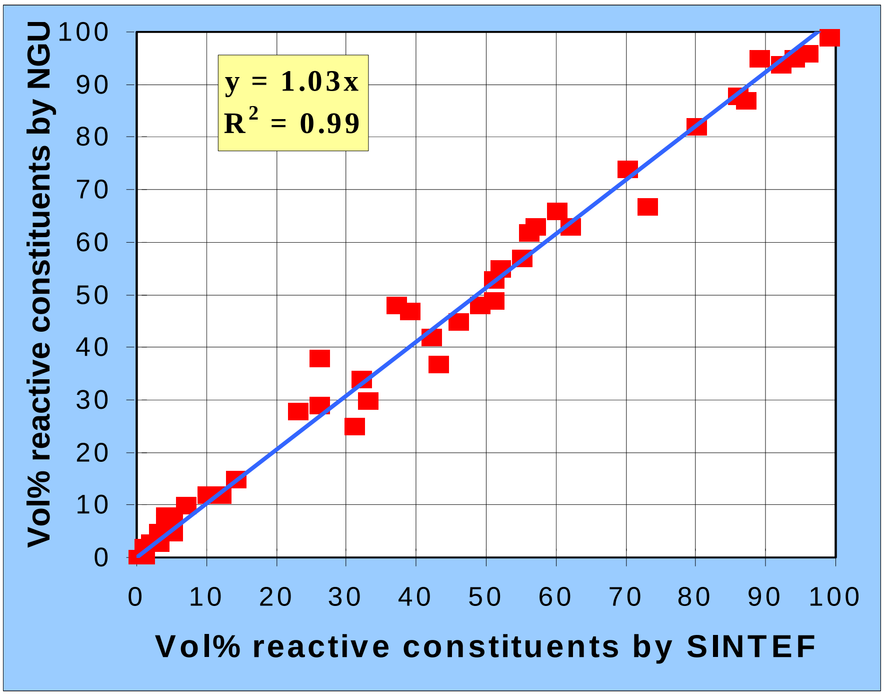

Figure 1. Correlation of reactive constituents obtained by SINTEF and NGU

7.2 Round robin test in Norway

Recently 6 Norwegian laboratories participated in a round robin test of 18 Norwegian aggregate samples analyzed by the DGB method (similar to RILEM method 2), **Wigum (4)**. One of the participating laboratories was SINTEF, labeled as laboratory 1. Before testing, a one-day course about Norwegian alkali reactive aggregates was performed. One of the laboratories did nor participates in this course. Pilot point counting of thin sections before the final testing was not performed.

Figure 2 shows the results and correlation between laboratory 1 and the 5 other laboratories. Note the generally poor correlation of results shown in figure 2. Table 2 gives some statistical parameters. It can be seen that only one of the laboratories obtained acceptable results, namely laboratory 5.

Tables 2 give the statistical parameters between laboratory 1 and laboratory 2-6

Laboratory 1 and	Statistic		**Laboratory 1 and**	Statistic	
laboratory 2	y=0.71x	R^2=0.63	**laboratory 5**	y=1.12x	R^2=0.84
laboratory 3	y=0.89x	R^2=0.57	**laboratory 6**	y=0.88x	R^2=0.59
laboratory 4	y=0.66x	R^2=0.34			

The results of this round robin testing clearly points out the importance of education and inter correlation (pilot test) before the final testing are carried out.

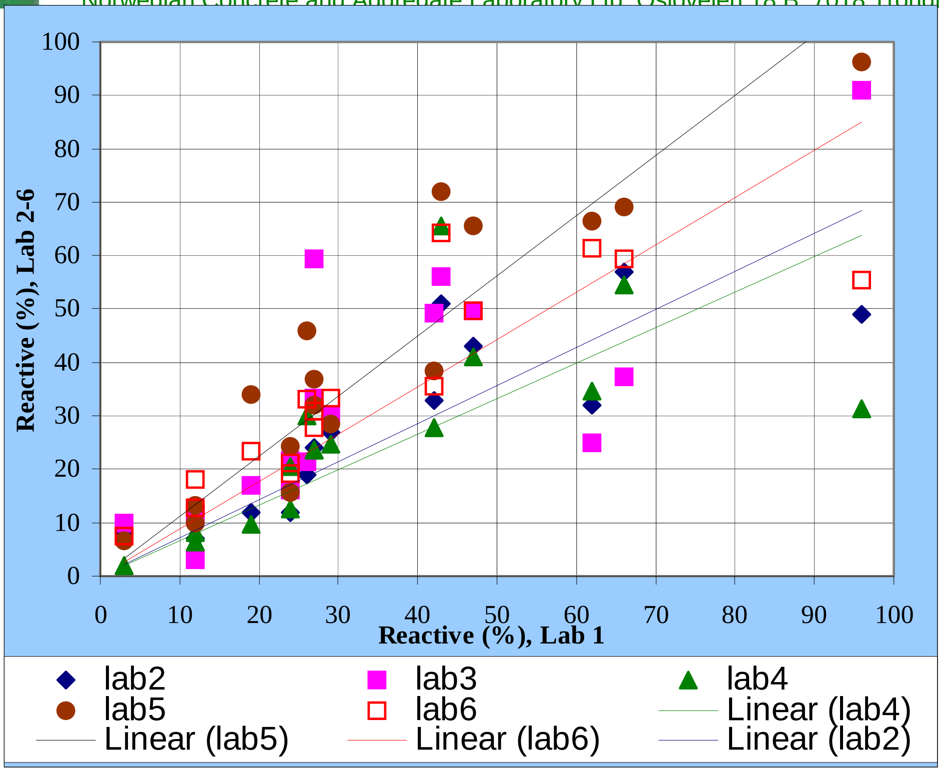

Figure 2. Results and correlation between laboratory 1 and the 5 other laboratories

7.3 Round robin test in Belgium

Seven Belgian laboratories were involved in a round robin test according to RILEM petrographic test method 2, **Lorenzi (5)**. The test was carried out in two steps on a Meuse river sand where the reactive constituent is chert. First, each laboratory prepared their own thin sections for point counting. The results are presented in table 3. Second, all the laboratories performed point counting on the same thin sections of the 0-2 mm fraction made by one of the laboratory (lab no. 1). The results is presented in table 4. Numbers given in tables 3 and 4 are volume percent reactive chert counted in thin sections.

Table 3: Results of point counting on own prepared thin sections.

Laboratory no.	Total chert %	Comments
1	**4.1**	
2	**3.6**	
3	*10.3*	*Inexperienced personnel*
4	**6.4**	
5	*1.5*	*Measured only porous chert*
6	*7.8*	*Inexperienced personnel*
7	**2.0**	
average	4.0	Laboratories 1,2,4 and 7
standard deviation	1.8	Laboratories 1,2,4 and 7

In Table 3 it can be seen that the total amount of chert varies from 1.5 to 10.3 volume %. However, an inquiry reviled that both laboratories 3 and 6 had used inexperienced personnel without knowledge of Belgian alkali reactive aggregates. Moreover, laboratory 5 only recorded porous chert and not the total chert content, as done by all the others laboratories. By excluding the results by these 3 laboratories and only use results from more qualified laboratories (highlighted) the total amount of chert varies from 2 to 6.4 volume percent with an average on 4 volume percent and standard deviation on 1.8%.

Results of the point-counting performed by each laboratory on the same thin sections containing the 0-2 mm fraction of Meuse river sand is shown in Table 4

Table 4: Results of the point counting on the same thin section

Laboratory no.	Volume % chert	Comments
1	**2.0**	
2	**2.3**	
3	*7.9*	*Inexperienced personnel*
4	**3.3**	
5	*3.5*	*Measured only porous chert*
6	*18.3*	*Inexperienced personnel*
7	**3.0**	
average	2.7	Laboratories 1,2,4 and 7
standard deviation	0.6	Laboratories 1,2,4 and 7

By excluding the results from the same 3 laboratories it can be shown that the chert content varies from 2.0 to 3.3 volume percent with an average on 2.8 volume percent and a standard deviation on 0.6 %.

The Belgian inter laboratory test demonstrate that variances within the laboratory as e.g. sampling, quartering and thin section preparation might influence the results. More important is it that qualified and experienced personnel with knowledge of Belgian alkali reactive aggregates perform the petrographic analysis.

7.4 The EU project "STAR" involving 6 European countries

The "Star project" was a newly concluded research project dealing with test methods on Alkali Silica Reaction (ASR) and financed by the European Community, **Jensen (6).** Six European partners worked together on this project. Among other tests, a precision test was carried out on same thin sections prepared from Danish natural sand. Reactive constituents in the sand are porous chert, which in thin sections has to be identified by use of fluorescence light. Procedures followed RILEM method 2, which also gives option for vacuum and fluorescence impregnating of samples. Results of the point counting of porous chert are given in Table 5.

Table 5: Results of point counting of porous chert on the same thin sections

Laboratory	Porous chert %	Comments
Belgium	6.9	No experience in porous chert
Denmark	5.4	
Norway	3.5	No experience in porous chert
Portugal	8.6	No experience in porous chert and petro-analysis
Sweden	6.4	
UK	2.8	No experience in porous chert
average	5.6	All laboratories
standard deviation	2.2	All laboratories

The point counting reviled a content that varies from 2.8 to 8.6 volume percent porous chert. It has to be mentioned that several of the laboratories (as marked in the comment) did not have experience with porous chert, which moreover has to be identified by use of fluorescence light. One of the laboratories did not have experience with petrographic analysis. An educational programmer dealing with identification of porous chert by use of fluorescence technique as well as a pilot test before the final testing would certainly have improved the reproducibility of the test.

8 Conclusion

The RILEM method gives procedures for performances of petrographic analyses. The method is based on the assumptions that knowledge of reactivity of rock and minerals are known and these are identifiable by use of normal petrographic techniques. National and local practice and recommendations shall always be followed where available. It is very important that the petrographic analysis is carried out by qualified personnel with experience of materials used for concrete and good knowledge of alkali reactive aggregates and minerals. The four cases given in the paper points out that the RILEM petrographic method is acceptable reproducible in case the analysis is carried out by qualified personnel. The RILEM petrographic analysis is an international method which can be used for most types of concrete aggregates.

Referances

1 RILEM petrographic method draft no 9 prepared by a working group under committee TC 106
2 Viggo Jensen & Marit Haugen 1996: “Alkalireaksjoner i Nord-Norge: Rapport nr. 2: Løsmasseforekomster og petrografiske analyser”, SINTEF rapport STF22 A96806 (in Norwegian)
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5 Gabriel Lorenzi 1999: Unpublished results from a round robin test in Belgium
6 Jørn Jensen 1998: STAR Project: Final report, European Commission, DG XII, contract no. SMT4-CT96-2128, PC laboratoriet, Fjerritslev, Denmark