APPLICATION of REFERENCE TESTS METHODS, of the KINETIC TEST and of the PERFORMANCE TEST to ASSESS AAR

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The authors present the methodology developed to examine aggregates used in concrete. This methodology comprises of several steps :

1. Geological survey of the site
2. Sampling, taken in accordance to the complexity of the deposit
3. Petrographic and chemical study
4. Characterization tests
5. Preventive measures assessment

In the four examples presented, steps 1, 2 and 3 are compulsory. Step 4 is invoked when the innocuous character of the aggregate is not certain. The step 5 is employed if the aggregate is shown to be deleteriously reactive.

INTRODUCTION

LAFARGE BETONS GRANULATS operates more than 100 quarries and produced approximately 30 millions tons of aggregates in 1991.These aggregates are primarily used for concrete. In face of the wide petrographic variety of the sites exploited, a need appeared in 1988 for the characterization of the aggregates by means of reliable methods, applied to representative samples. The development of these methods has been reported in several articles. Ranc et al. (1), Ranc et Debray (2) Sorrentino et al. (3).

At the same time, systematic sampling of the quarries belonging to LAFARGE BETONS GRANULATS had been undertaken. As this large programme is approaching completion, this article relates the methodology in use and its application to the characterization of quarries with regard to Alkali Aggregate Reaction (AAR). This includes the following steps :

- geological survey and sampling
- petrographic study
- characterization of the aggregates (end products) by suitable methods.

These successive steps lead to :

- assurance of the representativity of the studied samples, taking into account the exploitation constraints
- knowledge of the mineralogical composition of the deposit hence determining the choice of the method of evaluation
- characterizing the end products in an economical way

DESCRIPTION of the METHODOLOGY

The experience arising from several years of investigation in the AAR field, has allowed us to take an active part in the preparation of a document issued by the Ministère Français de l'Equipement of which describes measures for the prevention of deterioration due to AAR. This document will be adressed by Godart et Leroux (4) during this conference. Therefore, we will only recall the main features.

Sampling records

The procedure used for sampling in accordance to appendix D of the Ministère Français de l'Equipement (4), comprises of the following steps :
- geological survey and sampling of the various facies
- petrographic and chemical study of the facies
- establishment of an appropriate exploitation scheme

Characterization of the aggregates

This has been achieved, using three types of testing :
- reference test methods with mortar bars (NF P.18.585) or concrete prisms (NF P 15.587)
- kinetic tests of chemical reactivity (3)
- performance tests at 38°C and 60°C (2)

EXAMPLES of the METHOD APPLIED to the CHARACTERIZATION of DEPOSITS

The methodology has now been applied to more than 60 quarries of LAFARGE BETONS GRANULATS. Four examples have been chosen to illustrate this among those quarries.They are located in various geological contexts and offer different levels of potential reactivity. In every case, a detailed geological survey preceded the sampling ; only the conclusions will be given here in each case. Similarly, only the conclusions of the petrographic determinations will be reported.

Massive limestone (Provence)

Geology and sampling. The geological setting of upper jurassic limestones is well known, since it was used as corrective limestone for the preparation of the raw materials for cement production.
The deposit (60 m thick) lies in the northern limb of an anticline. The layers have a regular northen dip of 20° and are exploited by four benchs. The limestone is characterized by an homogeneous bedding of a more or less grey colored layers. Several karstic washouts are associated with normal small faults. They are filled with reddish clays and calcite. Clays are illiminated in the course of extraction and processing.
26 samples have been taken from the 60 m working face under exploitation. Given the apparent homogeneity of the rock, this number of samples seemed to achieve accurate representativity.

Microscopic determinations using thin sections confirmed the uniformity of the petrographic facies : it consists of a micritic limestone containing a few bioclasts. The rock is sometimes crossed by veins of recrystallized calcite. Detritic quartz was observed very occasionally.

The deposit homogeneity is confirmed by 2 drillings (75 and 105 m). The average chemical

composition, from 168 analyses, shows that the silica content is much lower than the 4 % limit imposed by the recommandation of the Ministère Français de l'Equipement. The chemical variations are very small :
SiO_2 : 0 to 1.85 %, Al_2O_3 : 0 to 0.55 %, CaO : 55.4 to 55.6 %, and MgO : 0.30 to 0.60 %.

Conclusion. Geological, petrographic and chemical studies demonstrate the deposit homogeneity. The available low content silica permit the deposit characterization, excluding any AAR risk. Moreover, the petrographic study shows that the small amount of free silica is present in the form of detritic quartz, which is almost insoluble in an alkaline medium.

It is therefore not necessary to conduct further investigation. This decision is justified because no occurence of AAR is known in this region even though the local cement is alkali rich.

Silico-calcareous river deposit (case 1 : Yonne valley)

Geology, petrography and sampling. The gravel-pit is situated along a river which flows across the Northern part of the French Massif Central and the southern borders of the Parisien Basin, thence the wide variety of petrographic types : magmatic rocks (coarse or fine grained granite, aplite) and sedimentary rocks (flint, siliceous limestone, limestone, ferruginous sandstone).

The deposit lies in a low terrace and is exploited below the water table with a dragline. It is therefore impossible to be sure of the representativity of the sampling. This is why counting of the petrographic facies had been undertaken. In this manner, we were able to verify the characterisation and the representativity of the bulk material sampled, as the exploitation progress.

50 kg of bulk material had been sampled. The granulometric distribution curve had been established in order to reconstitute the overall proportions of the various petrographic types. Several thin sections were prepared from fragments bigger than 20 mm. These were chosen in order to be representative of the petrographic types encountered in the deposit. Their study aims to validate the macroscopic determination of the elements.

A classification of the petrographical types were made on the 5/10, 10/15 and 15/20 mm granulometric fractions. The 0/5 mm sand was split into two parts : 0/1 mm and 1/5 mm.These were then impregnated with resin and studied with a polarizing microscope.

TABLE 1 - Petrographic counting.

(mm)	Quartz Quartzite	Flint (%)	Magm. rocks (%)	Sandstone (%)	Limestone (%)
20/40		1.1	42.6	4.9	51.4
10/20			16.7		83.3
05/10			13.6	1.0	85.4
01/05	27.1		37.3	5.2	30.4
00/01	73.4	3.0	6.0	2.2	15.4

TABLE 2 - Chemical analysis.

(mm)	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	K_2O (%)	Na_2O (%)	L.O.I. (%)	Total (%)
05/20	17.1	2.55	1.5	42.75	0.51	0.85	0.2	34.35	99.92
00/05	72.75	4.55	0.95	10.05	0.1	2.6	0.75	8.3	99.95

The silica content and the complexity of the mineralogical composition do not lead to a conclusive answer at this stage of the study, concerning the non reactivity of the aggregates.

Characterization of the aggregates. The mortar bar expansion test (NF P.18.585) had been applied to the 0/5 mm end-product sand, and the concrete prism test (NF P 18.587) was used for the 5/20 aggregates. The kinetic test (2) had been applied to both of these samples. The results are presented in figure 1.

. The sand is qualified as non reactive : the expansion almost ceases from 4 months on, and does not reach the 0.1 % limit value at 6 months. The kinetic test results are in agreement with these.

. The 5/20 aggregate is also qualified as non reactive, the expansion value being 0.013 % at 8 months (the limit is 0.04 %) and only shows slight changes at the end of the test at 15 months. The kinetic test again confirms the innocuous character of the 5/20 fraction.

Conclusion : for this kind of deposit, the petrographical and chemical analyses alone are not sufficient to qualify the aggregates. Sophisticated investigations (petrographic counting, study of several tens of thin sections, chemical analyses) were not really necessary to reach this conclusion. However, knowledge of the mineral composition of this site will allow in future, a quick check of the AAR characterisation of this deposit. If the variations in composition are not significant, it will not be necessary to repeat the expansion tests.

These tests give a clear answer. A fast, reliable and cheap diagnostic may be given for this quarry using the kinetic test.

Silico - calcareous river deposit (case 2 : Rhône affluent)

Geology and sampling. The gravel pit is located on a low terrace of a Rhone tributary which flows from the Alpes, thence the petrographic diversity of the aggregates : magmatic and metamorphic rocks (various granites, gneiss, more or less weathered amphibolites), and sedimentary rocks (limestones, siliceous limestones, sandstones).

The deposit (6 to 10 meters thick, 3 m being above water) is exploited by hydraulic excavator. Due to the nature of the deposit, the sampling cannot be considered fully representative of the site composition. This is why, here again, a study of the petrographic composition of the samples will allow an estimate of its representativity compared to future samplings.

The studied sample (50 kilos) had been taken on a pre-stock by mixing and quartering of several sub-samples of 25 kilos each. At this stage, the same procedure as previously described has been followed.

TABLE 3 - Petrographic counting.

Coarse aggregate (mm)	Quartz, Sandstone (%)	+/- calc. Limestone (%)	Magmatic rocks (%)
20/40	11	33	56
10/20	17	45	38
05/10	30	48	22

Sand (mm)	Limestone (%)	Quartz (%)	Sandstone (%)	Flint (%)	Granite Amphibolite
01/05	24.1		52.8	12.2	52.8
00/01	13.6	27.4	41.4	5.9	11.7

TABLE 4 - Chemical analysis.

(mm)	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	K_2O (%)	Na_2O (%)	L.O.I (%)	Total (%)
05/20	49.95	6.5	1.9	20.9	0.92	1.1	1.58	16.7	99.55
00/05	69.7	5.4	1.25	10.7	0.55	1.68	1.03	8.2	98.51

The petrographic composition displays the mineralogical complexity of the deposit. It is not possible to exclude any AAR risk. Especially as the thin section observations shows an abondance of limestones containing chalcedony and opal. Some of these limestones are undergoing a process of complete silicification and these elements are present in both the fine and the coarse aggregates.

Evaluation of the aggregates. The NF P 18.585 mortar bar test was used for the 0/5 mm sand, and the NF P 18.587 concrete prism test for the 5/20 mm aggregates. Additionally, the kinetic test was applied to both samples. The results are shown in figure 3.

. The potential reactivity of the sand appeared from 3 months on (0.0506 %). The diagnostic is confirmed at 6 months, the expansion value being 0.146 %. The kinetic test also qualifies this sand as deleteriously reactive.
. The coarse aggregate is characterized as potentially reactive from 6 months on (0.042 %), again confirmed by the kinetic test.

Facing the risk presented by the use of such aggregates, the decision has been taken to assess them according to the following trials :

- on one hand, the potential reactivity of the mix (sand and coarse aggregate) in a concrete with a 410 kg/m3 cement content of 0.94 % Na_2O eqv.
- on the other hand, the real behaviour of the same composition but using the local low alkali cement (Na_2O eqv. < 0.3 %).

The results of these tests (figure 2) confirm the expansive reactivity of the mix with high alkali cement (0.053 % expansion at 4 months). The concrete prims do not expand when using the low alkali cement, even after 15 months. These findings are confirmed by the performance test at 60 °C (cf. ref 1, table II, n° 21-22).

Conclusion : the petrographic determinations have revealed the presence of potentially reactive minerals which was confirmed by the expansion tests. In this case, the kinetic test also provided a reliable and quick answer.

The application of a performance test (1) taking into account in a realistic manner the local material supply possibilites, shows that the use of potentially reactive aggregates would be permitted, as the deleterious expansion would be totally suppressed using a low alkali cement.

Silico-calcareous river deposit (case 3 : Seine Valley)

Geology, sampling and petrography. The quarry exploits a low and a middle terraces, located in a loop of the Seine river. The lower terrace is submerged and the alluvia thickness is approximately 10 m. The middle terrace is above water (4 m thick). Both terraces have been separately investigated.

The sample taken from the middle terrace is representative of this part of the bed. 10 sub samples were taken from the working face, then mixed and quartered in order to produce a 25 kilo sample.This sample was studied according to the methodology already described above. The following results deal with this middle terrace sample.

TABLE 5 - Petrographic counting of coarse aggregates.

Coarse aggreg. (mm)	Flint (%)	Calc. Limestone (%)	Limestone (%)	Sandst. ,Granite (%)
40/80	91.9	0.8	7.3	
20/40	67.2	11.6	21.2	
05/20	57.2	9.6	26.2	7

TABLE 6 - Petrographic counting of natural sands.

natural sand (mm)	flint (%)	quartz quartzite (%)	limestone (%)	granite (%)	ferrugenous sandstone (%)
01/05	11.5	65.5	16	4	3
00/01	3	81.5	12.5	2	

It is observed that the coarsest fractions consist mainly of flints, with an increasing limestone content with the decreasing fraction size. In the sand, the flint content strongly diminishes whereas the quartz increases.

The treatment plant produces a concrete sand containing approximatly 90 % of natural sand and 10 % resulting from the crushing of the 10 mm plus aggregates. Therefore the mineralogical composition of this sand differs from that of the natural sand. It is enriched in flint and limestone particles coming from the crushed coarse fractions.

TABLE 7 - Petrographic counting of sand end product.

sand end product (mm)	flint (%)	quartz quartzite (%)	limestone (%)	granite (%)	ferrugenous sandstone (%)
01/05	18.8	40.4	34.2	1.1	5.5
00/01	2.7	85.6	6.2	5.2	0.3

It is worth noting that the chemical analyses of the sand and of the coarse aggregate are very close, (table 8) while the mineralogical compositions are markedly different (high proportions of flint in the coarse aggregate and quartz in the sand)

TABLE 8 - Chemical analysis of aggregates.

(mm)	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	K_2O (%)	Na_2O (%)	L.O.I (%)	Total (%)
05/20	87.74	0.25	0.39	5.84	0.04	0.04	0.4	5.65	99.99
00/05	89.3	1.61	0.40	3.87	0.01	0.97	0.22	3.5	99.88

The high silica content, partly in the form of flint (chalcedony and opal) and very soluble in a basic medium, led us to apply the expansion tests (mortar bars for the sand and concrete prisms for the coarse aggregate). As was done for the previous cases, the expansion test was conducted on the mortars bars and concrete prims.

Sand : the mortar bar method qualifies this sand as potentially reactive for 3 months on. At 6 months, the expansion value reaches 0.23 %. The kinetic test confirms this result.

Coarse aggregate : the expansion value is extremely low (0.009 %) well below the 0.04 % limit. The kinetic test characterizes these aggregates as chemically highly reactive, but not expansively deleterious, if used alone. This is in agreement with the concrete prism test.

Our study would not be complete if it was stopped at this stage, because we are dealing with a deleterious sand and a not deleterious coarse aggregate. The question is what would happen if both the fine and the coarse aggregate were mixed ? The true behaviour of the mix was evaluated using two concrete performance tests at 38°C. The concrete mixes tested included :

-A concrete having a cement content of 410 kg/m3 (0.94 % equivalent Na_2O)

- the same composition was tested with a local low alkali cement (0.24 %)

The granular composition qualified itself as non expansively reactive with both the HAC and the LAC cement. These conclusions were confirmed by the performance tests at 60 °C ((2), table II, n° 26-27).

Conclusion : the reference test methods on mortar bars and concrete prisms allowed the independant characterization of the fine and coarse fractions. Additionally, the kinetic test on both sand and gravel allow us to believe that the mix of the sand with the coarse aggregate could be expansively reactive (pessimum effects.). At this stage, only the performance tests at 38 °C and at 60 °C show the real behaviour of the aggregates composition (faster response).

CONCLUSION

The evaluation of an aggregate exploitation must rely upon an accurate and rigorous methodology, requiring multi-disciplinary team. A petrographic study and the evaluation of the representativity of the sampling are essential to set up the quarry's log and will facilitate up-dating. With these 4 examples, we intended to show that the methodology employed allows for an inexpensive evaluation of the quarry, taking into account their individual characteristics.

- the first example shows that the chemical and petrographic determinations can be sufficient to show non reactivity.

- In the second example, the first procedure was not conclusive and only the reference test method and the kinetic test show that the aggregates are not reactive.

- In the third example, the reference expansion tests and the kinetic test demonstrate the potential reactivity of the aggregates. The performance test permits the setting of an adequate prevention measure (use of a local low alkali cement).

-The last example is more complex : the reference expansion test evidences that the sand is expansive and the coarse aggregate is not. It was therefore necessary to characterize the combination of both fine and coarse aggregates, especially as the kinetic test suggested the possibility of a pessimum effect. The concrete performance test (at 38 °C and 60 °C) shows that the mix composition is not expansive, even with a high alkali cement.

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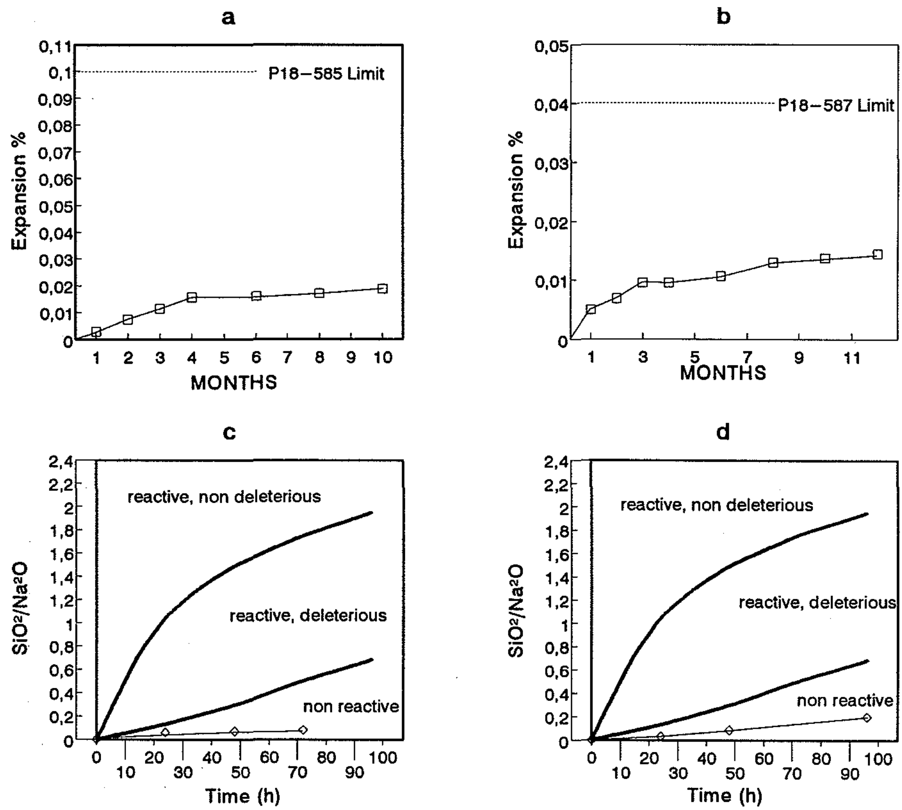


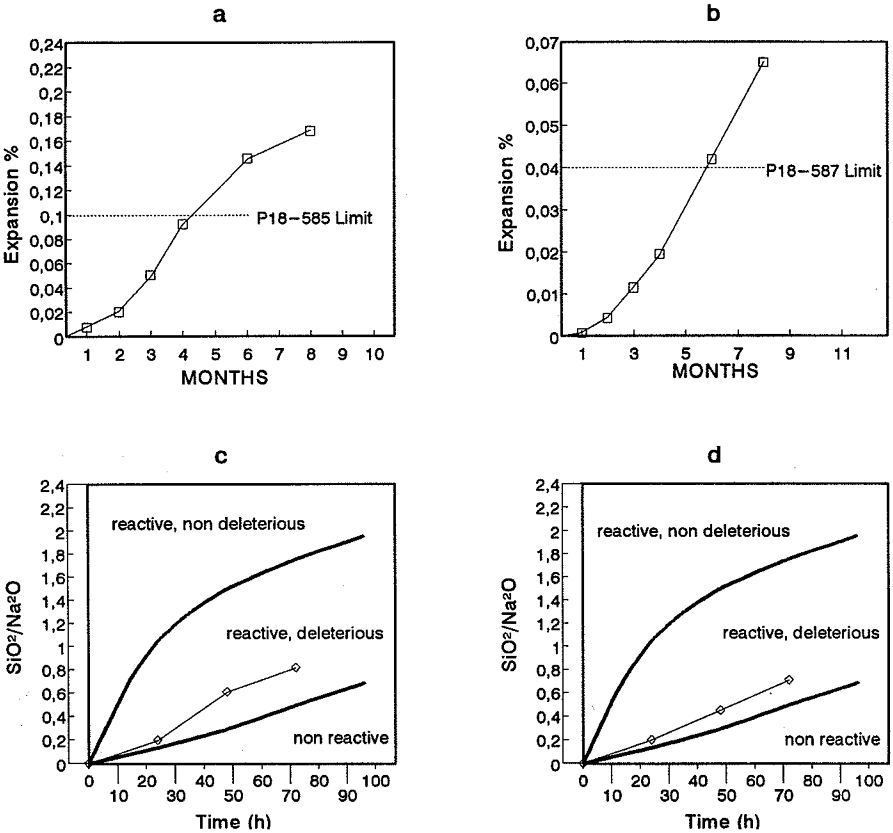
Figure 1 : Yonne valley

a sand: NF P18–585 test (mortar)

b 5/20: NF P18–587 test (concrete)

c sand: kinetic test

d 5/20: kinetic test



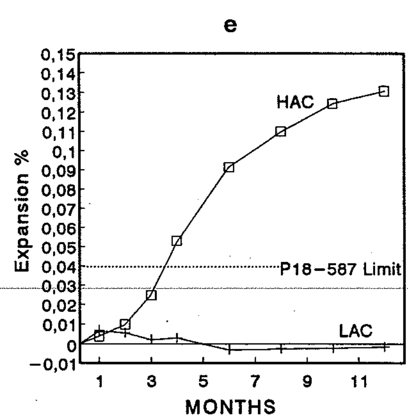


Figure 2 : Rhône affluent

a	sand: NF P18–585 test (mortar)
b	5/20: NF P18–587 test (concrete)
c	sand: kinetic test
d	5/20: kinetic test
e	sand + 5/20 : 38°C performance test

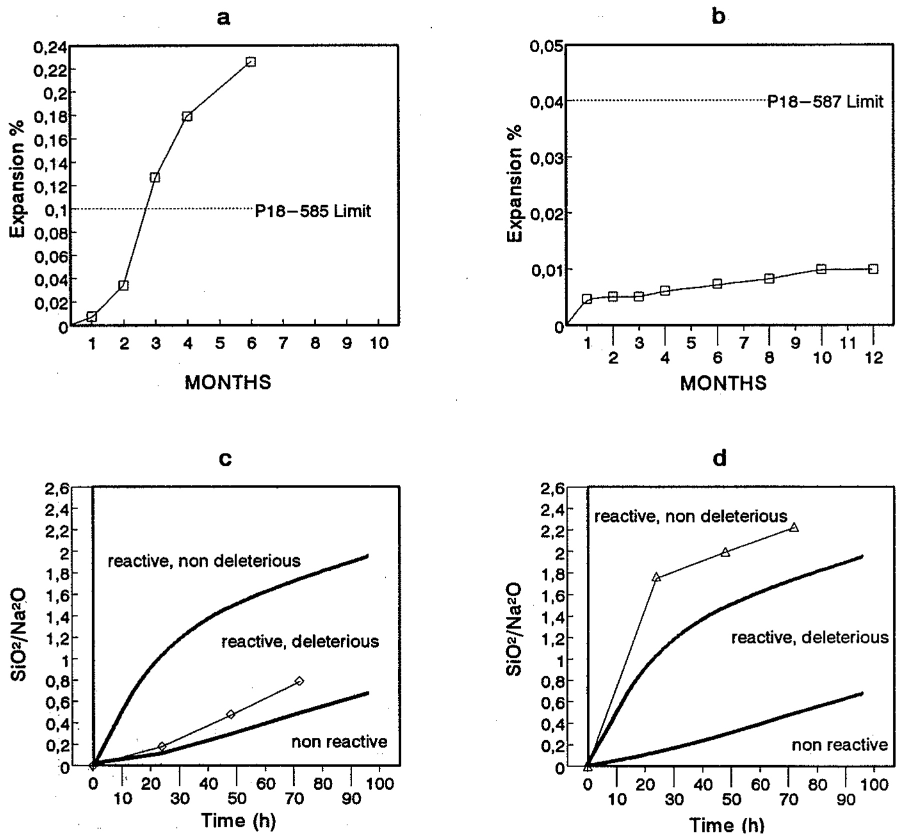


Figure 3: Seine valley

- a **sand: NF P18–585 test (mortar)**
- b **5/20: NF P18–587 test (concrete)**
- c **sand: kinetic test**
- d **5/20: kinetic test**
- e **sand + 5/20 : 38°C performance test**