VALORIZATION OF SAND WITH HIGH METHYLEN BLUE VALUE INTO BITUMINOUS MIXTURES FOR WEARING COURSES

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ABSTRACT

Most European nations use washed sand to design their bituminous mixtures for wearing course, primarily to prevent excessive water sensitivity of asphalt due to the presence of clays in sand natural fines. The lack of fines is then offset by an addition of limestone filler. These practices lead to high water and energy consumption. France, Belgium and Denmark are exceptions: they use quarry sands with their natural fines, without washing, but after checking the harmfulness of fines (clay content) according to French specifications described in the XP P 18-545 standard section 8, a maximum methylene blue value of 2 g/kg is permitted.

In order to save natural resources and improve the valorization of unwashed quarry sands, investigations have been carried out since 2007 on two quarry sands with methylene blue values in the range of 2 to 4 g/kg to study the behavior of bituminous mixtures for wearing course incorporating these unwashed sands.

These two sands coming from eruptive quarries of Burgundy (rhyodacite and rhyolite) were hence characterized by numerous tests, including cationic exchange capacity (CEC) measurements and mineralogical analysis by X-ray diffraction, showing the presence of illite-muscovite and montmorillonite. These sands were subsequently tested in the laboratory to design asphalt wearing course (0/10 Asphalt Concrete). Finally, these mixes were laid respectively in 2007 and 2009 on two experimental sites located on the French secondary road network. After 1 and 3 years of monitoring, no abnormal behavior induced by the presence of clay could be observed. This initial assessment allows us to consider valorizing these materials, particularly through the development of a regional technical manual regarding the sands of these two studied quarries, then by the writing of a user regional guide.

INTRODUCTION

A large majority of European countries realize their bituminous mixtures for wearing course using washed sand, in order to remove the finest fraction of sand containing clay. The removed fines are then compensated by an addition of limestone filler. France, Belgium and also Denmark are exceptions; they mainly use the sand without washing, but after checking the clay content.

In application to the note of CFTR n ° 10 of January 2005 [1], the sands used in the bituminous wearing courses must be in accordance with the code "a" of XP P 18 545 French standard [2], with among other characteristics, a value of methylene blue, representative of clay content (Methylene Blue MB by the NF EN 933-9 [3]), up to 2. In the particular case of quarries exploiting volcanic rocks, the specific alteration of minerals in these deposits produces a high proportion (12.5 to 35%) of tertiary sands with a MB value greater than 2.

Because of three cycles of crushing / screening allowing their manufacturing, these tertiary sands display in fact a substantial carbon assesment and a relatively high manufacturing cost (7 to 10 € / tons). It is thus important to make optimal use of these products for so called “noble” purposes, such as wearing courses, and optimize their use by aiming toward their full use and to natural resource conservation. For memory, the aggregate demand in Europe is 3 billion tons by year, slightly more than 6 tons by resident and by year, which almost 90% of natural origin (UNPG 2009/2010 [4]).

It’s also noted the reduction of alluvial quarries number get well underway in recent years. The substitution by resulting eruptive aggregates essentially in the production of concrete is currently accelerating. This sector of employment is also governed by a strict regulatory framework, which imposes sands with MB ≤ 1.5. This shift in resource use thus drains the category of sand with MB ≤ 2 to the production of concrete, and results in need of evaluating the possibility of using sand with upper MB value in bituminous mixtures.
The use of these so-called out of specification sands, in particular for wearing courses, would also supported the local economy by offering an alternative to import of products in accordance with a MB value ≤ 2 and so preventing the increase of transport distances.

The purpose of the experiments and research conducted since 2007 is to qualify in situ behavior of “out of specification” sands (MB > 2) into bituminous mixtures for wearing course, through the following of asphalt behavior versus time, and the comparing with control boards, to assess the influence of these fine clay on the mechanical properties of wearing courses. The goal of this study is also to use completely the acquired knowledge from these experiments to continue the studying of the influence of clays in asphalt mix materials as well as the understanding of the principles that govern it.

1. MATERIALS AND METHODS

1.1 Materials (aggregate and bitumen)

Tertiary sand and gravel used come from two quarries: Montauté quarry (Epiry - France Department 58 (Fig. 1), petrographic nature Rhyolite) and Moulin Neuf quarry (Fléty - France department58 (Fig. 2), petrographic nature Rhyodacite). Later in the section, quarries Montauté sands will be designated by S MT and those from the Moulin Neuf by S M9.

Photos 1 and 2 - Careers igneous of Montauté and Moulin Neuf
GBA Lafarge Department of Nièvre

Bitumen used in studies for the formulation of the bituminous mixtures is a pure bitumen (TOTAL type Azalt), with penetrability (European standard NF EN 1426) = 58/10 mm and ring and ball softening point test (European standard NF EN 1427) = 49 ° C.

1.2 Sands identifications methods

Sands 0/2 tertiary used in experiments have undergone several trials characterization: gap-grading analyses, methylene blue test, tests of sand equivalent. Additional tests were performed on the sand 0/0.125 mm fraction: Its blue methylene (MBf) value as well as sand equivalent (ES). In order to determine the different clay species of in the sands, investigations were realized by conducting test of carbonate and organic matter content, cation exchange capacity measurements (CEC), X-ray diffraction (XRD) as well as observations by scanning electron microscopy.

1.2.1 Gap-grading analysis

The gap-grading analyses of the sand have been conducted in accordance with European standard NF EN 933-1.

1.2.2 Methylene blue testing (MB and MBf)

Methylene blue testing at fraction 0/2 mm (MB) and at the fraction 0/0.125 mm (MBf) has been made according to European standard NF EN 933 - 9.

1.2.3 Sand equivalent test (ES)

Sand equivalent tests were conducted in accordance with the French standard NF P 18-597 and European standard NF EN 933-8.

1.2.4 Diffraction of X-rays (XRD)

Mineralogical analyses were performed with a Phillips PW1830 X-ray diffractometer. They allow identification of mineral phases in presence by performing analyses on crushed powder < 80 µm and oriented blades.

1.2.5 Cation exchange capacity (CEC)

The CEC capacity allows quantifying the exchangeable cations on the surface of clay minerals. These cations compensate the negative charge of substituted fact sheets (media electroneutrality principle). The measurements are inspired by the standard NF X 31 - 130. The protocol is as follows: it brings together the clay with of cobaltihéxamine Cl3Co (NH3) 6 which allows training in solution of $Cl_3Co(NH_3)_6^{3+}$ ion who explain the orange color of the solution. The concentration of this solution is measured by UV/visible spectroscopy after calibration. This supernatant proportioning allows the determination of the final concentration of ion and by comparing with the initial concentration of exchangeable cations.

1.2.6 Carbonate proportioning

The carbonate proportioning (calcite detected by XRD) is conducted by the French standard NF ISO 10693. This test consist in an addition of hydrochloric acid in a soil sample to decompose all present carbonates (here only calcite because the acid attack is carried out in cold condition). The volume of released CO_2 is measured using a “calcimeter” and is compared to the volume of CO_2 produced by pure calcium carbonate.

1.2.7 Analysis by scanning electron microscope (SEM)

The scanning electron microscopy is used to study the microstructure of sands. To facilitate comments, observation mode in low vacuum condition was used. It requires no preparation of sample of sand (no metallization steps). Images were obtained thanks to Quanta 400 microscope from FEI Company coupled to a EDX chemical probe from EDAX Company.

2. CHARACTERIZATION OF SANDS

2.1 Characterization of the MB and MBf sands 0/2

Quarrie of Moulin Neuf (S M9) fraction 0/2

The S M9 0/2 MB 3 showed a good homogeneity in their value of methylene blue as well as in their fines content (Fig. 4), with an MB average value equal to 2.7 and fines content in 0.063 mm fraction equal to 12%. On the other hand, figure 3 shows that MB 2 sands are more heterogeneous with a MB average of 1.7 and fines content in 0.063 mm fraction of 11.8%.

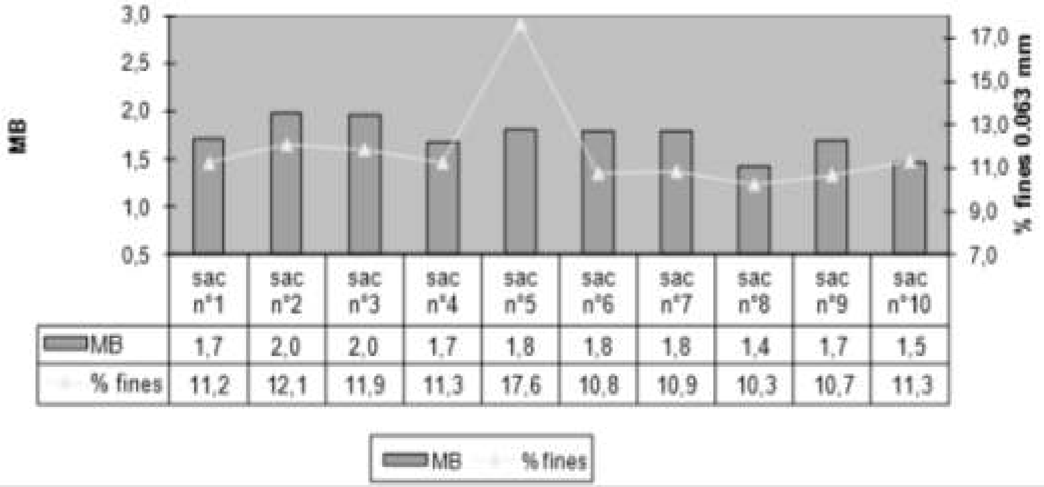

Figure 3 – MB tests and fines content of S M9 0/2 fraction (MB 2), mean value with % fines

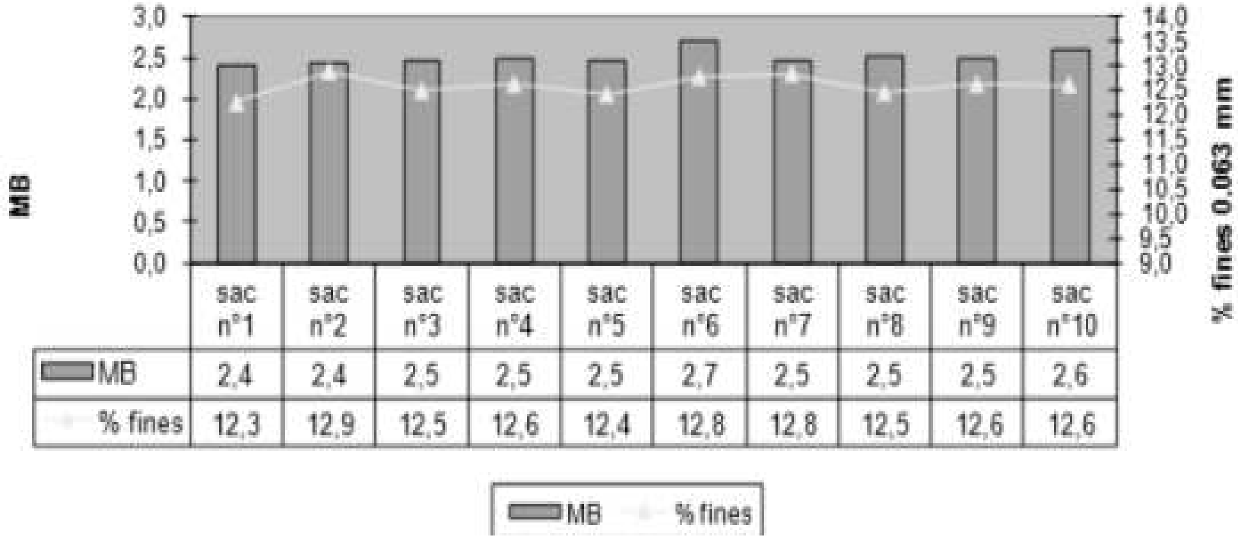

Figure 3 – MB tests and fines content of S M9 0/2 fraction (MB 3), mean value with % fines

Sand equivalent measures (ES) on 0/2 fraction of S M9 MB 2 and 3 are described in table 1:

Table 1 — Measures of ES on sand 0/2 M9

Sand from Moulin Neuf quarry (S M9) fraction 0/2 mm	Tests			
Identification	MB 2		MB 3	
	MB	ES	MB	ES
Sac n°7	1.8	55		
Sac n°10	1.9	52		
Sac n° 5			2.8	49
Sac n°10			2.8	48

For the record, the standard XP P 18-545 states in Section 7 (Section applicable only to the base layers and binder layers) indicates that ES minimum values are 60, 50 and 40% or MB values are respectively lower or equal to 2, 2.5 and 3. We see here that MB 2 of S M9 is relatively far from the sill of 60%, compared to the nearly 50% of ES measured for MB 3.

Montauté (S ME) fraction 0/2 quarry

The MB mean values measured on the S ME 0/2mm were below targeted values: average measured average of MB (Fig. 5) is 1.2, lower than the expected value of 2, with 8.8% of fines at 0.063. That measured on sand S ME 0/2mm with an expected MB value of 3 is 2.5 on

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averaged with 12.6% of 0.063 fines. They display however a good homogeneity in their blue value blues as in their fines element content (Fig. 6).

Figure 5 – MB tests and fines content in S ME 0/2mm (MB2)

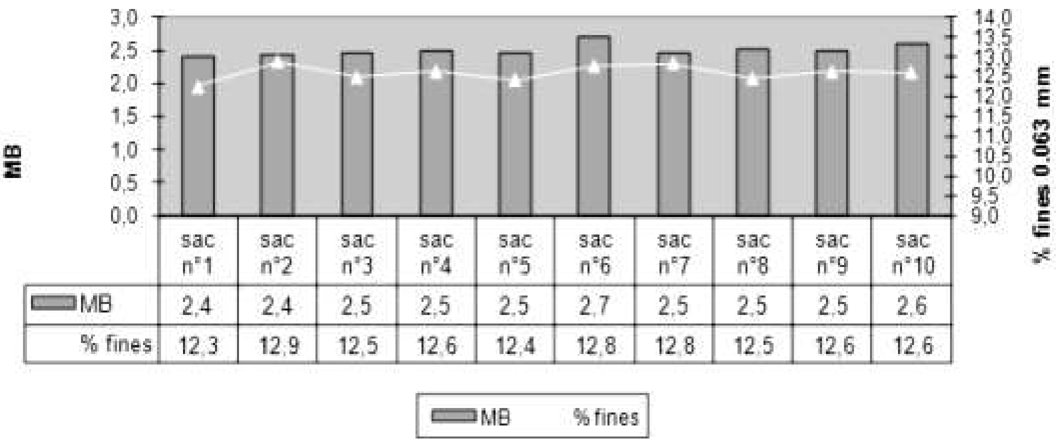


Figure 6 – MB tests and fines content in S ME 0/2mm (MB3)

Sand equivalent measures (ES) on the fraction 0/2mm of S ME of MB 2 and 3 are described in table 2:

Table 2 - Measures and on the sand 0/2 of S ME

Sands from Montauté quarry	**Tests**			
Identification	**MB 2**		**MB 3**	
	MB	ES	MB	ES
Sac n°5	1,4	59		
Sac n°10	1,2	60		
Sac n° 4			2,4	32
Sac n°7			2,7	32

We note here that ES measured for S ME MB 2 is close to the 60% sill, whereas that of S ME MB 3 is 32%, far from the 40% sill and the average of 49% obtained on the S M9 while their MB is slightly superior.
To investigate the effect of the storage conditions on the variability of sand in terms of MB, tests were conducted on several samples kept for one year and a half in a quasi-confined condition (closed bag / moisture content at the time of sampling). The results described in table 3 show that, under these storage conditions, MB measured values are stable (variability of 0.2, measurement uncertainty of MB test is ± 0.5).

Table 3 – Variability of MB measures on 0/2

	Measured MB		
Year		**2007**	**2009**

Table 4 - Variability of MBf measures on 0/0.125mm

S M9 MB 2	Sac n°7	1.8	1.8
	Sac n°10	1.5	1.9
S M9 MB 3	Sac n°5	2.7	2.8
	Sac n°10	2.6	2.8
S ME MB 2	Sac n°5	1.1	1.4
	Sac n°10	1.2	1.2
S ME MB 3	Sac n°4	2.5	2.4
	Sac n°7	2.5	2.7

		Measured MBf
S M9 MB 2	Sac n°7	6.9
	Sac n°10	6.5
S M9 MB 3	Sac n°5	8.6
	Sac n°10	8.7
S ME MB 2	Sac n°5	5.1
	Sac n°10	4.1
S ME MB 3	Sac n°4	7.7
	Sac n°7	8.6

2.2 Physicochemical and microstructural characterizations of sand 0/2mm

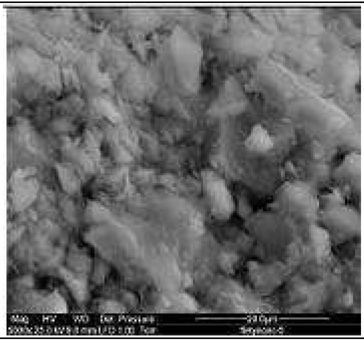
Physicochemical characterization of sands S M9 and S ME was conducted by measurements of the cation exchange capacity (CEC), the determination of carbonate and organic materials contents (CR tests LCPC Paris 2008-11-28 M. Duc and A. Maloula [5]). The results of these analyses are presented in table 5:

Table 5 - Physicochemical analysis of sand

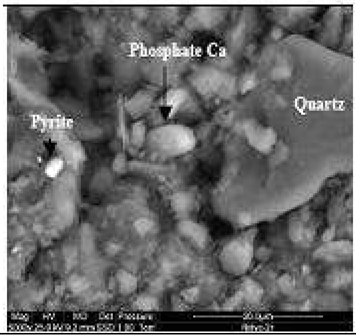
	S ME MB 2	S ME MB 3	S M9 MB 2	S M9 MB 3
Carbonate (%) (raw sample)	1	1	4	1
CEC	2.1	3.2	2.1	2.7
Organic content* (%)	0.02-0.03	0.01-0.02	0.60-0.65	0.5-0.6

* Sand sample with 80µm crushed fines.

Figures 7 and 8 present SEM images of MB 2 and 3 sands:

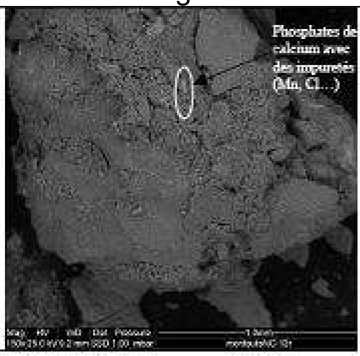


S M9 MB 3 (back scattered electron)

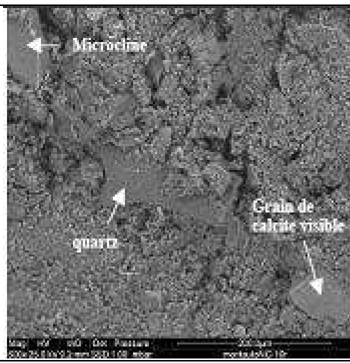


S M9 MB 2 (back scattered electron)

Figure 7 - Microstructural analysis of M9 sand 0/2mm



S ME MB 3 (back scattered electron)



S ME MB 2 (back scattered electron)

Figure 8 - Microstructural analysis of ME sand 0/2mm

Table 6 - Mineralogical and physicochemical sands analysis

Echantillons de fines (<63µm)	**Sable argileux MONTAUTE conforme C2**	**Sable argileux MONTAUTE non conforme A2**	**Sable argileux FLETY Conforme D2**	**Sable argileux FLETY non conforme B2**
Phase majeure	Quartz (I = 12721 coups)	Quartz (I= 12308 coups)	Quartz (I= 9245 coups)	Quartz (I= 10592 coups)
%carbonate	5	3	10	5
%MO	0.1-0.2	0.18-0.2	0.8-0.95	0.6-0.7
Autres phases	Plagioclase (albite) en faible teneur Feldspath K (microcline) en moyenne teneur	Plagioclase (albite) en faible teneur Feldspath K (microcline) en moyenne teneur	Calcite Plagioclase (albite) en moyenne teneur Feldspath K (microcline) en faible teneur Trace d'hématite (?)	Calcite Dolomite Plagioclase (albite) en moyenne teneur Feldspath K (microcline) en faible teneur
Argiles	Faible quantité	Faible quantité	Faible quantité	Faible quantité
CEC Fines extraites	8.3	9.9	7.9	10.2
Vb Fines extraites	18.3	21.6	19.3	21.5
Analyse semi-quantitative de la fraction <2µm Analyse exprimée (de 5% en 5%) pour 100% de minéraux argileux présents dans cette fraction	Pas ou peu de smectite (montmorillonite) Trace de muscovite (?) Kaolinite Trace de chlorite (clinochlore) (?)	Montmorillonite Na 30% Illite muscovite 40% Kaolinite 30% Palygorskite en très faible teneur	Montmorillonite Na 40% Illite muscovite 15% Kaolinite 35% Chlorite (clinochlore) 10% Trace d'interstratifiés smectite-kaolinite	Montmorillonite Na 50% Illite muscovite 10% Kaolinite 25% Chlorite (clinochlore) 10% Sépiolite 5% Interstratifiés smectite-kaolinite 5%
Autres phases de la fraction <2µm extraite à partir des fines <63µm.	Quartz microcline et albite en grande quantité Présence de calcite en très faible quantité	Quartz microcline et albite en moyenne quantité Présence de calcite en très faible quantité	Quartz Microcline et albite en moyenne quantité	Quartz Microcline et albite en moyenne quantité

Microstructural analysis of M9 and ME sands reveals that they are very similar. They display close mineralogical composition phases (Si, Al, K, Na, Mg, Fe), and similar forms and sizes, with an aspect of flake or lamina for clays and more massive particles with smooth surfaces and marked edges. Analyses of mineral phases are showed in table 6. The set is summarized in table 7. These analyses allow us to observe the non-negligible presence of Smectite (Montmorillite) in three used sands, clay known as expansive and harmful, that can create disorder in the coated and thus reduce their sustainability.

Table 7 - Summary of analyses for the determination of natures of clays

Mineral species	S ME MB 2	S M9 MB 2	S ME MB 3	S M9 MB 3
Muscovite/Illite	Trace?	15%	**40%**	10%
Kaolinite	Presence	**35%**	30%	25%
Montmorillonite	Few or none	**40%**	30%	**50%**
Sépiolite	-	-	-	5%
Chlorite	Trace?	10%	Traces	5%
Palygorskite	Presence ?	-	-	-
Interstratifé	-	Traces	-	5%

3. DESCRIPTION OF EXPERIMENTS

3.1 Effects expected with the use of clay sands in bituminous mixtures for wearing courses

Clays contained into sands may have two effects on the behavior of asphalt mix. On one hand they can cause a stiffening of asphalt mix, making it less easy to handle in the short term and more sensitive to ageing. On the other hand, clays, because of their fine fraction and their flake structure, swell in presence of water, and thus they increase the sensitivity of bituminous mix to water.

3.2 Contents and conduction of experiments 2007 and 2009

The experiments consisted into the realization of several test boards, on secondary road sections with T3 traffic (50 to 150 Truck Annual Average Daily Traffic per sense), with relatively severe climatic conditions (cold winters, rainfall, continental climate and high temperature ranges,...). These sections, in addition to their monitoring during their construction, have been subject to regular monitoring: point zero (immediately after construction), 6 months, 1 year, 2 years and 3 years for 2007 experimentation, to see the evolution of the boards experimental versus control. They will finally be completed by a 5 years monitoring.

Quarries of Montauté (Epiry-France Department 58 – Petrographic nature: rhyolite) and Moulin Neuf (France-Fléty Department 58 – Petrographic nature: rhyodacite), were selected to supply tertiary sands and gravels for the experimental sites. Two projects have been realized: the first on 2007, on RD 978 secondary road (France General Council 71) with 2 control asphalt mix boards (1 Formulation with MB 2 Montauté sand and 1 with MB 2 Moulin Neuf sand) and 2 boards with sands out of specification (1 Formulation with MB 3 Montauté sand and 1 with MB 3 Moulin Neuf sand).
The second was conducted in 2009, on RD 981 secondary road (General Council 58), with 2 control asphalt mix boards (1 Formulation with MB 2 Montauté sand and 1 with MB 2 Moulin Neuf sand) and 3 boards with sands out of specification (2 Formulations with MB 4 Montauté sand and 1 with MB 4 Moulin Neuf sand).

Photo 3 – Roadworks 2009 RD 981 CG 58

Photo 4 – Roadworks 2007 RD 978 CG 71

4. FORMULATIONS OF BITUMINOUS MIXTURES

3.1 Formulation of bituminous mixtures for wearing courses

The choice concerned a mix for wearing course, type semi-coarse bituminous concrete (BBSG) 0/10 (Class 2), to facilitate observation of the evolution boards, directly subjected to various climatic and mechanical constraints. This choice also allows to use a granular formulation of continuous curve (0/2 - 2/6 - 6/10), which do not displays a too large percentage of voids, the objective is to be able to observe these boards during a long term.

Table 8 – Summary of formulations for BBSG 0/10 2007 and 2009 and real density of aggregates and asphalts

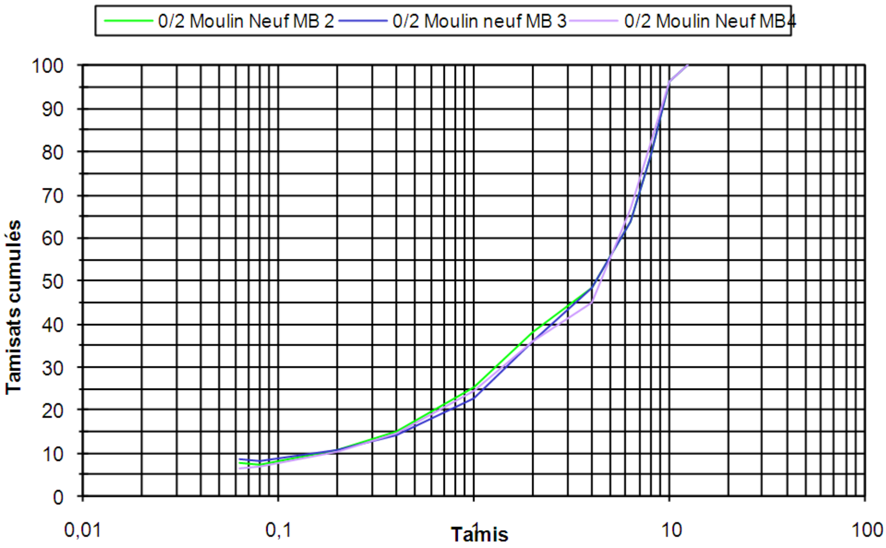
Components / Formulations BBSG 0/10	**Sand 0/2**	**Aggregates 2/6**	**Aggregates 6/10**	**Lime stone filler**	**Pure Bitumen 50/70 (TL externe**)**	**Asphalts real density g/cm3**
MOULIN NEUF MB2	34 %	25 %	38 %	3 %	5.9 ppc*	2.449
MOULIN NEUF MB3	34 %	25 %	38 %	3 %	5.9 ppc	2.449
MOULIN NEUF MB 4	37 %	27 %	35 %	1 %	5.8 ppc	2.477
Aggregates real density g/cm3	2.658	2.718	2.722	-	-	-
MONTAUTE MB 2	34 %	25 %	38%	4 %	5.9 ppc	2.380
MONTAUTE MB 3	34 %	25 %	38%	3 %	5.9 ppc	2.386
MONTAUTE MB 4	35 %	24 %	40 %	1 %	5.9 ppc	2.374
Aggregates real density g/cm3	2.573	2.573	2.567	-	-	-

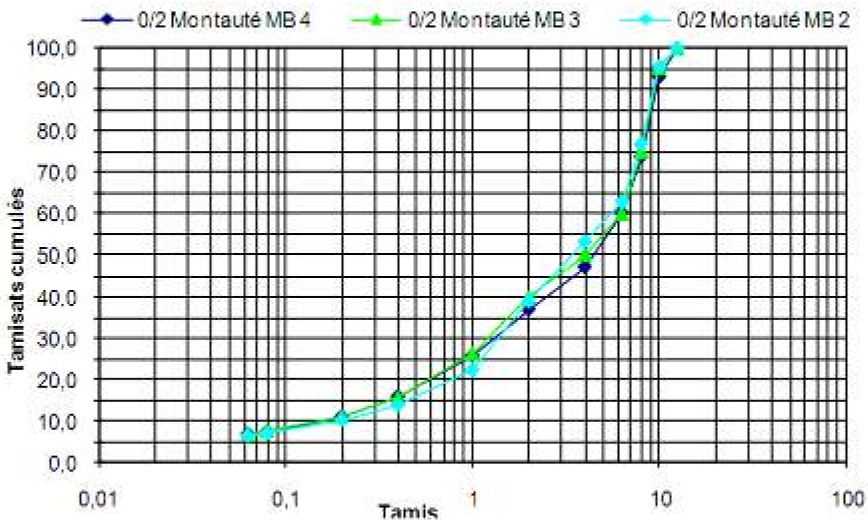
(* ppc: part per cent – the totality of aggregates = 100% and ** binder (bitumen) extern content)

Bituminous mixtures tested for "2007" and "2009" studies as well as actual densities of aggregates and bituminous mixes are presented in table 8 above. The formulations of level 2 (gyratory shear compactor PCG / Water sensitivity and Rutting), were conducted by the laboratory of Autun, with a pure bitumen at grade 50/70, without adding any additives, to better isolate the influence of the presence of fines clays into the mixes.

Table 9 - Summary of granulometric curves of granular mixtures theoretical studies 2007 and 2009

Formulations BBSG 0/10	% of aggregates passing sieves in mm			
	10	6.3	2	0.063
S M9 MB 2	96.2	63.7	36.4	7.8
S M9 MB 3	96.2	63.7	36.1	8.5
S M9 MB 4	96.4	66.9	35.8	6.4
S ME MB 2	95.6	62.8	39.0	6.3
S ME MB 3	95.3	60.0	39.9	6.9
S ME MB 4	93.0	60.0	36.9	6.7





Figures 11 and 12 – gap-grading analysis of theoretical granular mixtures for formulation studies

5 RESULTS LABORATORY FORMULATION STUDIES

Laboratory studies conducted to DL Autun formulations studies, were level 2 (gyratory shear compactor PCG / Water sensitivity “Duriez”/Rutting), according to the specifications of the former French standard NF P 98 - 130 governing concretes bituminous Semi Grenus (BBSG) [6], experiments having started in 2007, before the application of european standards.
The results are as follows:

5.1 Testing compactibility to gyratory shear compactor (MLPC PCG-2)

Table 10 – Summary of void percentages (%) measures for x gyrations

PCG (Voids in % for x gyrations)	V_1 (%)	V_{10} (%)	V_{40} (%)	**V_{60} (%)**	V_{100} (%)	V_{200} (%)
S M9 0/2 MB 2	21,10	16,5	10,9	**9,4**	7,6	5,3
S M9 0/2 MB 3	25	17.9	12.6	**11.1**	9.4	7.3
S M9 0/2 MB 4	23.9	19.2	11.5	**10.0**	8.2	5.7
S ME 0/2 MB 2	15.7	13.4	11	**9.6**	8.1	6.1
S ME 0/2 MB 3	16.1	13.7	11.2	**9.8**	8.2	6.2
S ME 0/2 MB 4	15.1	12.7	10.3	**9.0**	7.4	5.4

5.2 Rutting resistance test (large model rutting machine - MLPC)

Table 11 - Measurements of rutting percentage at 30,000 cycles

Rut (60°C)	Void rates %	Density %	% rut / number of cycles					
			100	300	1000	3000	10 000	**30 000**
S M9 0/2 MB 2	6.9	93.1	2.8	3.4	4.1	5	6.5	**7.4**
S M9 0/2 MB 3	7.7	92.3	3	3.6	4.3	4.9	5.9	**7**
S M9 0/2 MB 4	7.8	92.3	2.9	3.7	8.4	5	6.2	**7.6**
S ME 0/2 MB 2	7.5	92.5	1.9	2.4	3.1	4.4	5.5	**7**
S ME 0/2 MB 3	7.3	92.7	2.2	2.6	3.3	4.0	5.9	**7.1**
S ME 0/2 MB 4	7.7	92.3	2.4	2.9	3.5	4.2	5.5	**6.9**

5.3 water sensitivity tests for bituminous mixtures ("Duriez")

The results (table 12 and Fig. 14) during the determination of water sensitivity of BBSG 0/10 formulated with Moulin Neuf MB 2 and 3 sands are relatively close and do not actually distinguish a formulation from the other one. We notice for the MB 4 compared to MB 2 a slight decrease in resistance values to air and to water, as well as increase of the average percentage of absorbed water by the bituminous mixture after conservation by immersion lasting 7 days.

Characteristics	**S M9 0/2 MB 2**	**S M9 0/2 MB 3**	**S M9 0/2 MB 4**	Standard specifications	
				NF P 98-130	NF EN 13108-1
Hydrostatic Density	2.260	2.229	2.259		
Void content (%)	7.2	8.6	8.8		
Strength at 7 days in air, R (MPa) or C (MPa)	8.31	8.42	7.76		
Strength after immersion, r (MPa) ou i (MPa)	6.93	7.46	6.06		
Average percentage of absrobed water after 2h in vaccum condition	2.50	2.56	2.48		
Average percentage of absrobed water after 7 days	3.15	3.11	3.58		
r/R ou i/C ratio	**0.83**	**0.89**	**78 % (=0.78)**	>= 0,75	>= 70 %

Table 12 - Summary of water sensitivity tests for S M9 formula

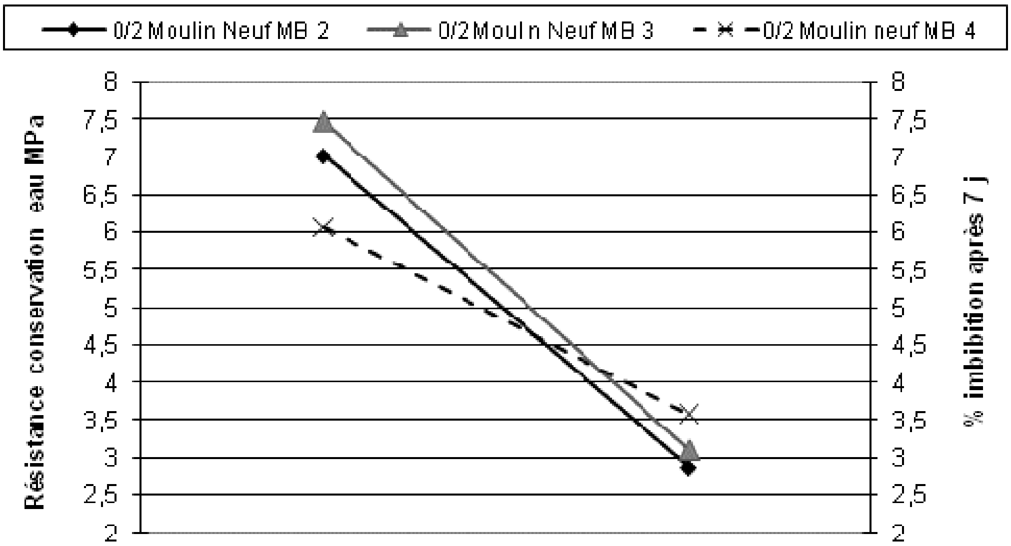


Figure 14 - Resistance test of samples kept 7 days in water /7 days imbibitions for S M9 bituminous mixture

The results (table 13 and figure 15) obtained for the determination of water sensitivity of BBSG 0/10 samples formulated with MB 2 and 3 Montauté sand are also close and do not actually distinguish one formulation from the other one. The results obtained for MB 4 sands compared with the reference MB 2 shows a decrease in resistance values to air and water as well as an increase of the average water absorbed by the samples after conservation (7 days immersion).

Table 12 - Summary trials of water sensitivity of bituminous mixture containing S M9 sand

Characteristics	S ME 0/2 MB 2	S ME 0/2 MB 3	S ME 0/2 MB 4	Standard specifications	
				NF P 98-130	NF EN 13108-1
Hydrostatic Density	2.193	2.177	2.157		
Void content (%)	7.9	8.8	9.1		
Strength at 7 days in air, R (MPa) or C (MPa)	7.90	8.43	6.35		
Strength after immersion, r (MPa) ou i (MPa)	6.87	7.09	4.83		
Average percentage of absrobed water after 2h in vaccum condition	2.69	2.51	2.75		
Average percentage of absrobed water after 7 days	3.67	3.38	4.03		
r/R ou i/C ratio	**0.87**	**0.84**	**76,1 % (=0.76)**	>= 0,75	>= 70 %

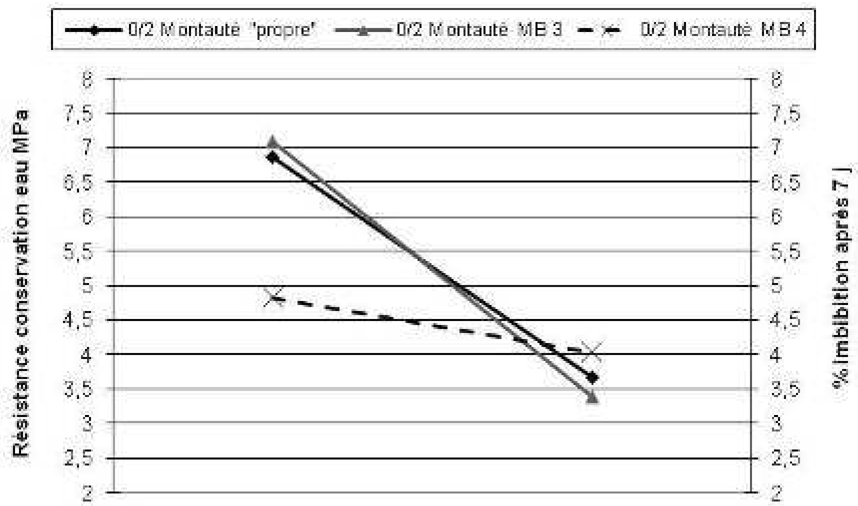


Figure 15 - Resistance test of samples kept in water for 7 days/7 days imbibitions for S M9 bituminous mixture

These findings join the opinion of European experts in the working group WG3 (JL. Delorme 2006 [7]), "denoucing" Duriez test as to be not sufficiently discriminating, due notably to the after uniaxial compressive test of samples after conditioning. Indeed, the obtained resistance would not explain that the aggregate-bitumen bonding is affected by water presence, nor the self mineral skeleton strength.

5.4 Findings on formulation studies

At the conclusion of these six level 2 formulation studies, it was possible for the laboratory bituminous mixtures type BBSG 0/10 for wearing course layer, to obtain mechanical characteristics corresponding to the normative specifications for class 2 materials.

Complementary observation made in the laboratory: we note that to increase their MB value, the 0/2 sands from, after drying in an oven, on the surface of storage trays a relatively hard gangue.

Fine clay, present in sand 0/2 MB 4, tend to remain agglomerated, even after 30 seconds of dry mixing in laboratory mixer, forming clusters of random diameters, that we find after in the bituminous material. These simple findings, inherent to fines clay characteristics, underline however the important of the storage conditions of sands “outside specifications” in term of MB high-value, in quarries as in central mixing plant for bituminous mixture, and is prerequisite for their use. All the precautions and the means operated before this use can only be a positive influence for the obtaining of bituminous mixture with satisfactory characteristics, the reduction of clay agglomeration risks and the increasing of bituminous material durability, and finally to better control the energy needed for drying and the temperature rising of the granular materials.

6 PRESENTATION OF EXPERIMENTAL SECTIONS 2007 AND 2009

6.1 Characteristics of selected secondary road sections

Table 14 - Summary of the work sections and their main - Summary table

		CG	Road	PR start	PR end	Formulation	Classe of Trafic	Configuration / preparatory works
Site 09/2007	S M9 MB 2	71	RD 978	14+591	14+956	BBSG 0/10 cl2 (50/70)	T3	out urban area – pavement width 7.00 m – preparatory works recovering of existing pavement with bitumen foam 07/2007
	S M9 MB 3	71	RD 978	14+956	15+285	BBSG 0/10 cl2 (50/70)	T3	
Site 10/2009	S M9 MB 2	58	RD 981	76+820	77+125	BBSG 0/10 cl2 (50/70)	T3	out urban area – pavement width 7.00 m – no preparatory works – flexible road structure + 5 bituminous concrete +ESU
	S M9 MB 4	58	RD 981	77+125	77+425	BBSG 0/10 cl2 (50/70)	T3	
Site 09/2007	S ME MB 2	71	RD 978	13+975	14+272	BBSG 0/10 cl2 (50/70)	T3	out urban area - pavement width 7.00 m – preparatory works recovering of existing pavement with bitumen foam 07/2007
	S ME MB 3	71	RD 978	14+272	14+591	BBSG 0/10 cl2 (50/70)	T3	

<table>
<tr><th></th><th></th><th>CG</th><th>Road</th><th>PR start</th><th>PR end</th><th>Formulation</th><th>Classe of Trafic</th><th>Configuration / preparatory works</th></tr>
<tr><td rowspan="4">Site 10/2009</td><td>S ME MB 2</td><td>58</td><td>RD 981</td><td>77+725</td><td>78+025</td><td>BBSG 0/10 cl2 (50/70) ½ voie droite sens +</td><td>T3</td><td rowspan="4">out urban area – pavement width 7.00 m – no preparatory works – flexible road structure + 5 bituminous concrete + ESU</td></tr>
<tr><td>S ME MB 2</td><td>58</td><td>RD 981</td><td>77+725</td><td>78+025</td><td>BBSG 0/10 cl2 (35/50) ½ voie gauche sens +</td><td>T3</td></tr>
<tr><td>S ME MB 4</td><td>58</td><td>RD 981</td><td>78+025</td><td>78+325</td><td>BBSG 0/10 cl2 (50/70) ½ voie droite sens +</td><td>T3</td></tr>
<tr><td>S ME MB 4</td><td>58</td><td>RD 981</td><td>77+425</td><td>77+725</td><td>BBSG 0/10 cl2 (35/50)</td><td>T3</td></tr>
</table>

The whole experimental boards was conducted on secondary roads subjected to type T3 traffic (pm: 50 to 150 heavy trucks in annual daily average traffic by sense), and outside urban area. The BBSG 0/10 wearing course were set up in 5 and 6 cm on sections with and without preparatory work. These sections are located in areas with a continental climate with relatively harsh winters (especially regarding the winter 2009/2010), precipitations and consistent thermal amplitudes.

Figure 16 – RD 978 2007 Visit March 2010

Figure 17 – RD 981 2009 Visit March 2010

6.2 Overview of 2007 and 2009 boards experimental following-ups

The different experimental boards have indeed be the subject of a follow-up during their setting-up, as well as the establishment of a "Point zero" (initial state) to ensure their follow-up. These follow-ups included:

- BBSG 0/10 formulas compositions control (gradations, contents binder),
- Control of compaction by measures of % voids in-situ (gamma-densitometer Troxler model),
- achievement of the core of pavement for the verification of their thicknesses and testing of the cores by gammadensimetry test,
- audits of the characteristics of surface by measuring the average texture depth (PMT in mm: glass bead test),
- Audits of the Visual State of the sections, coupled with measurements of rutting (with ultrasonic transverso profilograph TUS).

Monitoring carried out at 6 months, 1 year, 2 years and 3 years for the 2007 work, included the following measures:

- audits of surface characteristics by measuring the average texture depth (PMT in mm: glass bead tests),
- Audits of the Visual State of the sections, coupled with measurements of rutting (with ultrasonic transverso profilograph TUS).
- Realization of pavement cores to evaluate the state of the interfaces and wearing layers and additional testing on cores.

Table 15 - Summary of follow-up at 1-2 years in two experimental sections

		Road	PR start	PR end	Bitumen	% voids (Troxler) Point 0	PMT (mm) at 1 or 2 years	visual state at 1 or 2 years
Site 09/2007	S M9 MB 2	71 RD 978	14+591	14+956	50/70	7.3	0.61	No degradation
	S M9 MB 3	71 RD 978	14+956	15+285	50/70	7.7	0.70	
Site 10/2009	S M9 MB 2	58 RD 981	76+820	77+125	50/70	7.2	0.57	No degradation
	S M9 MB 4	58 RD 981	77+125	77+425	50/70	8.6	0.54	
Site 09/2007	S ME MB 2	71 RD 978	13+975	14+272	50/70	6.9	0.49	No degradation
	S ME MB 3	71 RD 978	14+272	14+591	50/70	7.0	0.55	
Site 10/2009	S ME MB 2	58 RD 981	77+725	78+025	50/70	7.0	0.51	No degradation
	S ME MB 2	58 RD 981	77+725	78+025	35/50	7.0	0.57	
	S ME MB 4	58 RD 981	78+025	78+325	50/70	7.4	0.59	
	S ME MB 4	58 RD 981	77+425	77+725	35/50	7.2	0.58	

6.3 Preliminary assessment of experiments

Tracked results from these two sites of experimentation are encouraging. On the one hand the mix bituminous BBSG 0/10 could be formulated in accordance with normative standards of the French standard NF P 98-130 (November 1999). On the other hand, monitoring of the evolution of the plates is overall positive: all the experimental planks of the 2007 site evolves in similarly way (MB 2 and 3), we do not detect during the following-up of measures any difference in their behavior for the present. The 2009 construction boards seem to evolve in the same way, that what we will see for the follow-up of the state of these experimental sections at 3 to 5 years, under the control of the SETRA.

CONCLUSIONS

The presentation of these two experiments, carried out after several previous studies of the influence of clays on bituminous materials essentially in the laboratory, shows that the formulation of road bituminous mixtures performing normative specifications tolerates the presence of a quantity of fine clay and that the limit of acceptability of this amount is linked to the typology of clays in presence. However, the threshold of acceptability of the presence of clays, normatively set and used today, ignore this typology, particularly because traditionally used in laboratory tests and methods are insufficient to do so (values of the sand equivalent and MB), and that the access to these characterizations by X-ray diffraction experiments is difficult.

To stop the systematic elimination of large quantities of materials that could be recovered, one of the upcoming issues will be the development of a scientifically based methodology, and more widely releasable, to characterize in qualitative and quantitative ways these clays in these barren of quarries.

This is one of the themes of the AGREGA research project [8] and also a topic of a proposed thesis this year by IFSTTAR institute (LCPC), entitled "Study physicochemical of opportunities to reuse waste rock quarries in bituminous materials" [9], following-up by a second thesis focusing on the development of a easy test and usable for the characterization of real harmless of clays in quarry sands.

Finally, this first report, built on the observation of experimental projects, allow us to establish an organization for the valorization of these clay sands, through the edition of a regional specification sheet for the use of these studied sands coming from these two quarries. These specifications sheets were widely distributed by our laboratory (DL Autun) to managers, consultants and professionals of public works [10]. This sheet will be completed by a regional guideline of the employment of these sands in bituminous mixes which will be submitted to the approval of the IDRRIM [11].

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REFERENCES

1. Comité Français pour les Techniques Routières (CFTR) info n° 10 de Janvier 2005, P. Dupont (SETRA)-G. Delalande (LRPC Angers) – F. Verhee (USIRF) – G . Aussedat (UNPG) - B. Roussel (LRPC Blois) – JF. Lafon (LRPC Toulouse)
2. Norme française d'application volontaire XP P 18-545 « Granulats – Eléments de définition, conformité et codification » de Mars 2008 – AFNOR
3. Norme européenne NF EN 933-9 « Essais pour déterminer les caractéristiques géométriques des granulats – Partie 9 : Qualification des fines – Essai au bleu de méthylène » de décembre 2009 – AFNOR
4. Union Européenne des producteurs de granulats (UNPG) – Rapport annuel 2009/2010 – Page 6 rubrique « En bref »
5. Compte-rendu d'essais – 28/11/2008 – LCPC Paris M. Duc et A. Maloula « Analyse de fines argileuses »
6. Norme française NF P 98-130 « Enrobés hydrocarbonés – Couches de roulement et couches de liaison : Bétons Bitumineux semi-grenus (BBSG) – Définition/Classification/Caractéristiques/fabrication/Mise en œuvre » de Novembre 1999
7. JL Delorme, 2006 « La transposition européenne vers les normes d'essai sur enrobés » Revue Générale des Routes et Aérodromes (RGRA) n° 850, pages 82 à 93.
8. Opération de Recherche LCPC 11L091 AGREGA « mAtériaux GRanulaires Et Granulats Alternatifs » 2009/2014 – Responsable d'opération Y. Descantes
9. Sujet de thèse LCPC Paris/Nantes « Etude physico-chimique des possibilités de réemploi de stériles de carrières dans des mélanges bitumineux routiers.
10. Note d'information CETE de Lyon-DL Autun sur des chantiers expérimentaux en Bourgogne – 26/01/2011
11. IDRRIM : Institut Des Routes, des Rues et des Infrastructures pour la Mobilité