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Determination of the compaction of cements and mineral admixtures using the Vicat needle

Thierry SEDRAN*
Franois de LARRARD
Laurdan LE GUEN
LCPC, Bouguenais, France

■ ABSTRACT

The scientific method applied to concrete mix design, as developed by LCPC and introduced into the BetonlabPro software over the past several years, is based on granular concepts; for this reason, BetonlabPro users need to know about packing density for all elementary granular fractions, especially for mineral powders (cements, mineral additions, etc.). Concerning this last category, a visual assessment of the water demand, by means of searching for a state change between a mix of wet pellets and smooth paste, was recommended. The evaluation of state change depends on operator interpretation, which could lower the level of reproducibility, even though repeatability for any single operator is quite high. Researchers have recently proposed an alternative method based on determining the water demand for a normal consistency measured using the Vicat needle. This paper shows both water demand measurement methods are consistent with one another within the framework of the granular model employed. The benefit of this new and less subjective method has thus been confirmed, and the operating procedure is detailed for application using *BetonlabPro*.

Détermination de la compacité des ciments et additions minérales à la sonde de vicat

■ RÉSUMÉ

La méthode scientifique de formulation des bétons, développée par le LCPC et mise en œuvre dans le logiciel BétonlabPro depuis plusieurs années, est fondée sur des concepts granulaires. C'est pourquoi l'utilisation de ce logiciel nécessite la connaissance de la compacité de l'ensemble des fractions granulaires élémentaires, et notamment celle des poudres minérales (ciments, additions etc.). Pour ces dernières, une mesure visuelle de la demande en eau, par recherche d'un changement d'état entre mélange de boulettes humides et pâte lisse, est préconisée. L'appréciation du changement d'état dépend de l'opérateur, ce qui peut diminuer la reproductibilité, bien que pour un opérateur donné la répétabilité soit bonne. Des chercheurs ont récemment proposé une méthode alternative fondée sur la détermination de la demande en eau pour une consistance normale mesurée à la sonde de Vicat. Il est montré que les deux méthodes de mesure de la demande en eau sont cohérentes entre elles dans le cadre du modèle granulaire utilisé. On confirme ainsi l'intérêt de la nouvelle méthode, moins subjective, et on en précise le mode opératoire pour une utilisation dans BétonlabPro.

*CORRESPONDING AUTHOR :

Thierry SEDRAN
thierry.sedran@lcp.fr

INTRODUCTION

The so-called scientific method for concrete mix design developed by LCPC is based on granular concepts [1] introduced into the BetonlabPro software package [2]. As a result, use of this software requires a certain knowledge of the packing density of all elementary granular fractions, especially regarding mineral powders (cements, admixtures, etc.). For such powders, a visual assessment of water demand, by means of identifying a state change between smooth paste and a mix of wet pellets, had until now been the prescribed method. This paper will present results from tests that

serve to validate a new and more objective method based on use of the Vicat needle, as proposed by Lecomte *et al.* [3].

PREVIOUS WORK ON THE TOPIC

Two methods are currently applied to evaluate the packing density of mix components. It is determined in a dry condition for materials whose grain diameter exceeds 80 μm [4] ; on the other hand, it is evaluated by a water demand test for fine granular elements such as binders and mineral admixtures. This approach enables incorporating the influence of not only water on the powder behavior, but also an admixture of the superplasticizer type if and when necessary. The water demand test chosen up until now within BetonlabPro applications [2] proceeds as follows. By using a mortar mixer like that described in Standard NF EN 196-1, a mass M_p of powder is mixed with water and, as needed, with a superplasticizer. Next, through successive tests, the quantity M_e of water showing the mix to be wet-like soil is determined, to which adding a small increment ΔM_e transforms the mix into a state of homogeneous paste. The packing density C of the powder can then be derived using the following formula :

$$C = \frac{1000}{1000 + M_v \frac{M_e}{M_p}}$$

where M_v is the mass density of the powder (in kg/m^3) and M_e and M_p are the respective water and powder masses (in kg).

The packing density of a material depends on the energy input to ensure compacting the material. This value is taken into account in the Compressible Packing Model (CPM) [1] used as part of BetonlabPro β , by means of a scalar K (called the packing index), which increases with the amount of packing energy. A given set of compaction operating procedures would thus correspond with a fixed packing index.

In adopting the hypothesis that monosized fractions making up a single component display the same basic packing density and with the knowledge gained from both the component experimental packing density and the test packing index, the CPM enables deriving the theoretical packing density of monosized fractions corresponding to an infinite packing index. This packing density, also called virtual elementary packing density, has been denoted C_v . It is an intrinsic¹ property of the component and knowledge about it serves to authorize packing density calculation of any mix that includes the particular component and for any mode of compaction described by a packing index.

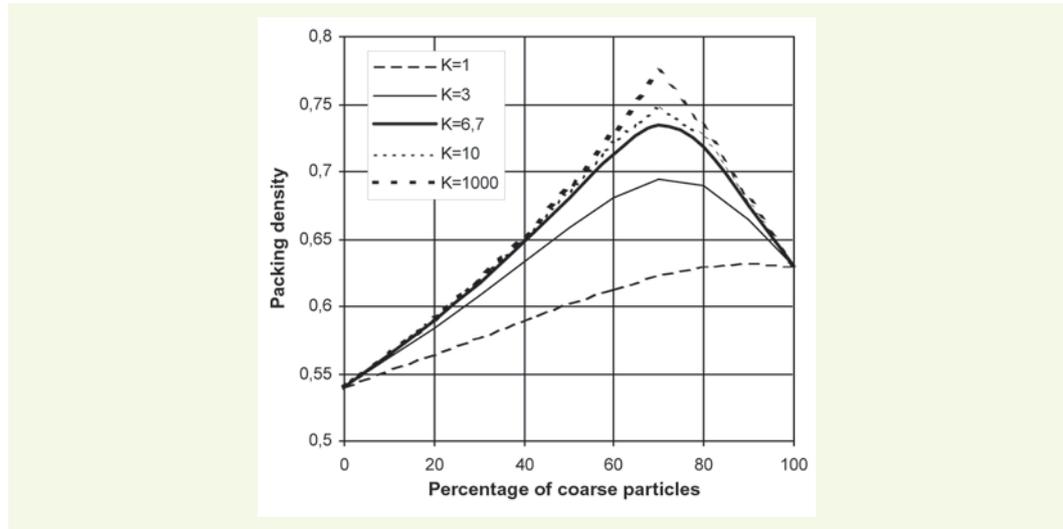
This packing index cannot be directly measured, but instead is calibrated by means of self-consistency of the granular model. As observed in **Figure 1**, the theoretical packing density according to CPM of a binary mix evolves along an increasingly "pointed" curve as the packing index value increases, for actual constant packing density values of the two basic components. For a given compaction test, it then becomes possible to calibrate the packing index value on the basis of experimental data relative to binary mixes. The packing indices corresponding with dry compaction and with the water demand test could thus be evaluated at 9 and 6.7, respectively [1, 6].

The water demand measurement on homogeneous paste, as described above, displays one major difficulty : the state change assessment is strictly visual, which could hinder reproducibility, even though for any single operator the level of repeatability proves satisfactory. This concern is particularly evident when the state change is quite gradual, e.g. in the absence of an admixture or in the presence of ultrafines. Moreover, such an assessment has not been standardized and therefore

¹ In principle. As a matter of fact, it appears that the operating procedures requiring a low-energy setting do not always lead to the same values of b , and for sands in particular. It is thus recommended to perform high-energy compaction for dry measurements with respect to aggregates [5].

only gets performed on rare occasion, and even less frequently transmitted to the client by the component supplier.

Figure 1 :
Influence of the packing index value K on the compaction of a binary mix, with constant elementary packing density values



As a means of circumventing this difficulty, Lecomte *et al.* [3] recently proposed introducing the notion of normal paste consistency, as set forth in Standard NF EN 196-3. In this case, water demand corresponds to the quantity of water necessary for the Vicat needle to penetrate to within 6 mm from the bottom of the mold containing the paste. Besides the fact that this test is indeed standardized, it proves advantageous from the standpoint of making reference to a quantitative measurement, which *a priori* is not affected by operator subjectivity. The authors have demonstrated, through calibration on binary mixes composed of cement and mineral admixtures, that the packing index equaled 4.8 for this test. The objective of the experimental campaign presented below is to verify, for the purpose of complementary validation and in order to ensure consistency between both water demand measurement methods, the possibility of deducing from one method the compaction given by the other.

TESTING PROGRAM

Tables 1 and 2 list the components selected to conduct the tests. The particle size distribution of the cement from Saint-Pierre-la-Cour was determined using a MasterSizer 2000 laser granulometer. The same particle distribution was then adopted for the other two cements. This approximation exerts only limited influence on the calculations carried out with the CPM since both cements were employed in pure form. Precise knowledge of the mix's grain size distribution is in fact essential just for mixes containing several components with very similar distributions, for which granular interactions (loosening and wall effect) are considerable. The grading of silica fume has been deduced from the technical data sheet for the coarse part and then extrapolated for the fines [2, 6] based on the specific surface value provided by the supplier ($14 \text{ m}^2/\text{g}$), in adopting the hypothesis of spherical grains and a linear size distribution in $\log(d)$, with d being the grain diameter.

table 1 :
Components used in the tests

Name	Mass density (kg/m ³)	Dry extract (%)
CEM I 52.5 N CE CP2 NF, from Lumbres (Holcim)	3,150	
CEM I 52.5 N CE CP2 NF, from Saint-Pierre-la-Cour (Lafarge)	3,100	
CEM I 52.5 N CE CP2 NF, from Le Havre (Lafarge)	3,160	
White thermal silica (SEPR)	2,200	
Sika Viscocrete Tempo 10		30

Table 2 :
Materials grading

Cements		Silica fume	
Diameter (μm)	Cumulative passing (%)	Diameter (μm)	Cumulative passing (%)
0.5	0	0.04	0
0.63	2	0.05	4.4
0.8	3.5	0.063	9
1	4.9	0.08	13.7
1.25	6.3	0.1	18.1
1.6	8	0.125	22.6
2	9.8	0.16	27.4
2.5	11.9	0.2	31.9
3.15	14.5	0.25	36.3
4	17.9	0.315	40.9
5	21.7	0.4	45.6
6.3	26.4	0.5	50
8	32	0.63	58.3
10	38.2	0.8	67
12.5	45.1	1	75
16	53.8	1.25	83
20	62.8	1.6	92
25	72.7	2	100
31.5	83.2	2.5	100
40	92.8		
50	98.8		
63	99.8		
80	99.9		
100	100		

The saturation amount of superplasticizer (Sp*), as measured according to the grouting method [7], yields a value of 0.3% dry extract with respect to cement mass for the three cements.

The experiments have consisted of measuring water demand both to obtain a homogeneous paste and to derive normal consistency on the following set of samples :

- the three cements, for superplasticizer dosage Sp, such that Sp/Sp* equals 0, 0.25, 0.5, 0.75, 1, 1.25 and 1.5 ;
- binary mixes dosed at 95/5, 90/10 and 80/20 (in weighted percentages) of Saint-Pierre-la-Cour cement and silica fume, respectively. Superplasticizer was added to the mixes at a dosage of 0.6% dry extract with respect to total weight (cement + silica fume), to ensure reaching saturation.

For the sake of homogeneity, it was decided to establish a single protocol for determining the water demand at normal consistency, whether or not measured in the presence of a superplasticizer. This protocol was generated on the basis of tests conducted by Lecomte *et al.* [3] and following a preliminary series of tests. It will be observed further below however that for operational reasons, the protocol finally adopted is not exactly the same : this test is performed using a standardized mixer (as described in Standard NF EN 196-1) and the Vicat device equipped with a 10-mm diameter consistency probe, as presented in Standard NF EN 196-3.

Regarding paste production, the following procedure is employed :

- tap water is stored at 20 ± 2 °C for all test runs ;
- the surfaces of both the tank and blade of the mixer are wetted, while being sure that no excess water remains ;
- a specific quantity of water is weighed inside the mixer tank (e.g. 125 g from the outset), eventually containing the desired concentration of superplasticizer ;
- a mass $M_p = 500$ g of powder is weighed and added into the mixer ;
- the mixer is activated for 90 seconds at slow speed ;
- mixing is stopped, then the tank is scraped for 15 seconds using the blade ;
- slow-speed mixing for another 180 seconds.

Next, penetration measurements are carried out for various water content levels, in accordance with the prescriptions laid out in Standard EN 196-3, which yields a graph similar to that shown in **Figure 2**, where h is the distance between the bottom of the mold and the end of the probe.

The quantity of water M_{e6} that results in a height $h = 6$ mm can be determined by means of successive tests. In the aim of limiting the number of tests, it is possible to deduce through interpolation the quantity of water M

M_{e6} from two points framing the normal consistency ; yet in order to minimize error, points A and B must verify the following conditions :

$$h_A \leq 30 \text{ mm}$$

$$h_B \geq 2 \text{ mm}$$

$$M_{eB} - M_{eA} \leq 5 \text{ g}$$

It then becomes possible to calculate powder compaction via the following equation :

$$C = \frac{1000}{1000 + M_v \frac{M_e}{M_p}}$$

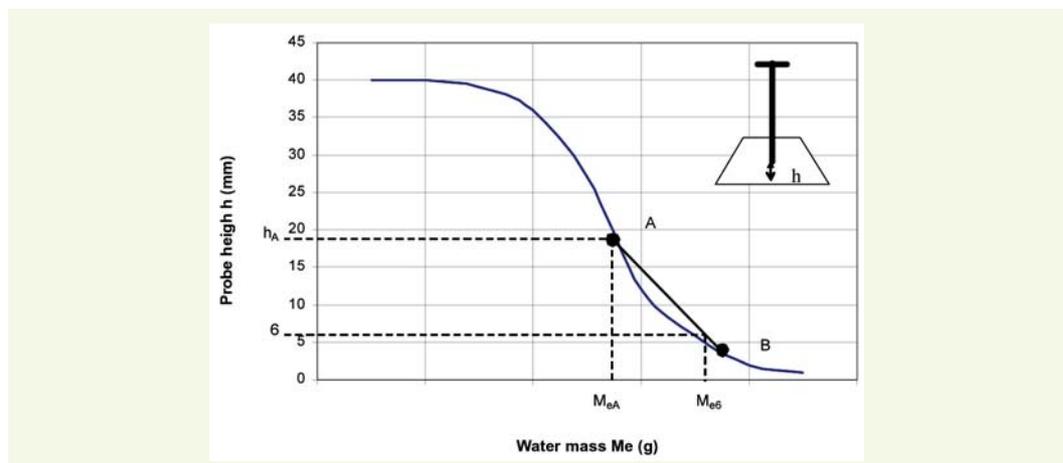
where :

$M_e = M_{e6}$ is the mass of all added water (additional water plus water included in admixture),

M_v is the mass density of the powder (in kg/m^3), and

M_p is the powder mass.

Figure 2 :
Evolution of probe penetration vs. water content level



RESULTS AND DISCUSSION

■ Adjustments of the packing index

Table 3 and **Figure 3** display the full set of results obtained for cements. The level of packing density measured with the Vicat needle tends to rise with superplasticizer concentration, as could be expected. Similarly, according to the Vicat test, adding superplasticizer at saturation (i.e. $Sp/Sp^* = 1$) leads to an increase in compaction of between 0.04 and 0.05. The same trend is revealed with the water demand test on homogeneous paste, except for the Saint-Pierre-la-Cour cement, whose packing density determined from this test remains just about stationary as superplasticizer concentration rises from 0 to 1.25 times the saturation dosage.

For each cement and superplasticizer concentration level, the values of β have been calibrated on the basis of water demand tests applied to a homogeneous paste, with $K = 6.7$. The packing index corresponding to the compaction value obtained for the water demand test on a normal paste (see **Fig. 4**) can then be derived. The average value obtained of 4.7 lies very close to the 4.8 value found by Lecomte *et al.* By applying a value of 4.8, the theoretical compaction values for normal pastes may be recalculated. As indicated in **Figure 5**, the correspondence with experimental results is satisfactory, and the average error is limited to 0.011 in absolute value terms. Another way of presenting the results (**Table 4**) consists of comparing the values of β obtained from tests conducted on both homogeneous pastes ($K = 6.7$) and normal pastes ($K = 4.8$). The average deviation between the two values also amounts to 0.011.

Figure 3 :
Cement packing density
measurements using the
two water demand methods

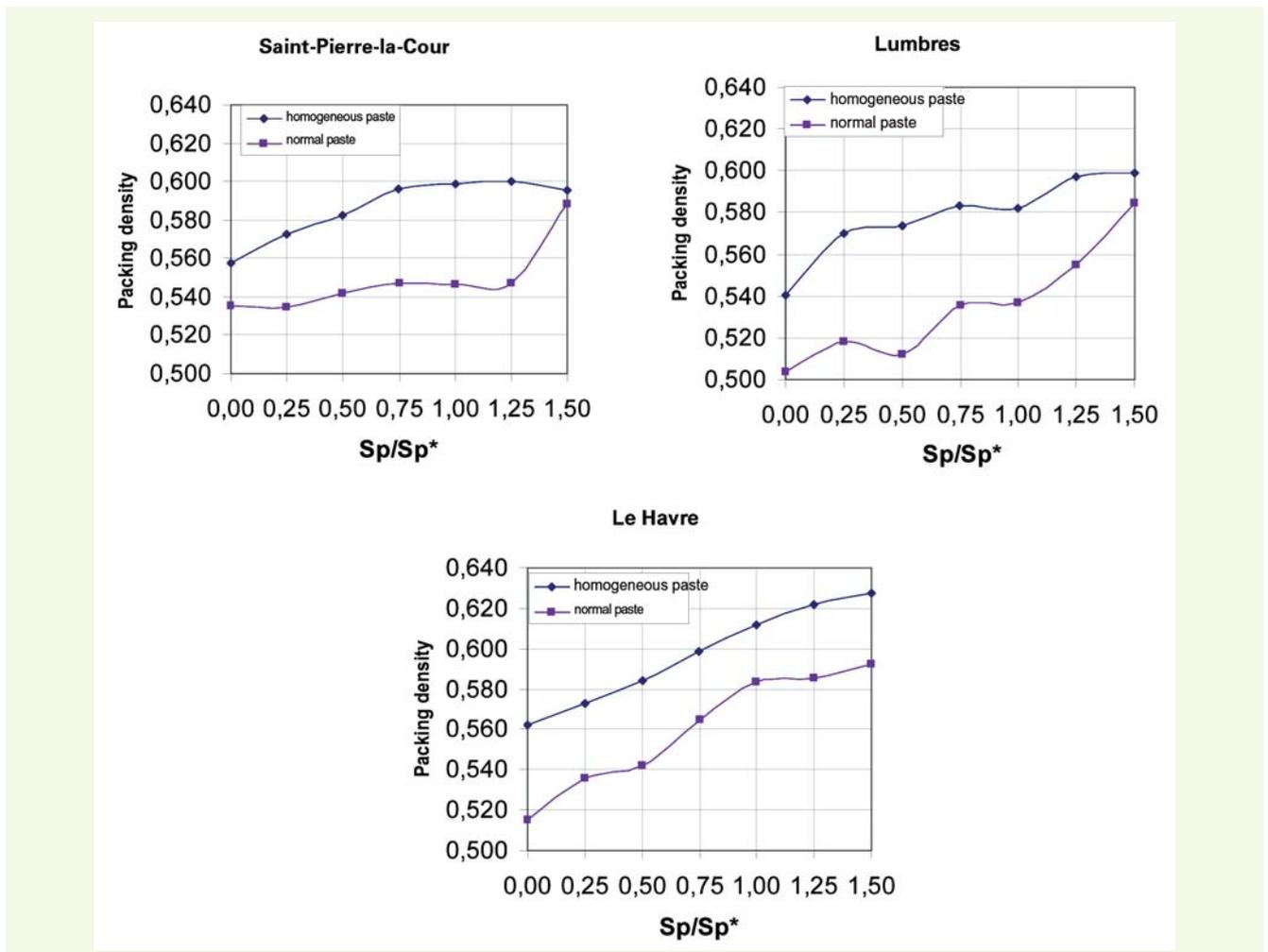


Table 3 :
Results from packing measurements

Sp/Sp*	Saint-Pierre-la-Cour		Lumbres		Le Havre	
	Homogeneous paste	Normal paste	Homogeneous paste	Normal paste	Homogeneous paste	Normal paste
0	0.558	0.536	0.5402	0.5039	0.5619	0.5153
0.25	0.572	0.535	0.5700	0.5181	0.5731	0.5357
0.5	0.582	0.542	0.5738	0.5120	0.5844	0.5419
0.75	0.596	0.547	0.5834	0.5359	0.5986	0.5649
1	0.598	0.547	0.5823	0.5370	0.6117	0.5836
1.25	0.6	0.547	0.5973	0.5549	0.6217	0.5854
1.5	0.595	0.589	0.5991	0.5845	0.6272	0.5925

Figure 4 :
K values calculated for the water demand on normal pastes using b values calibrated with the water demand on homogeneous pastes

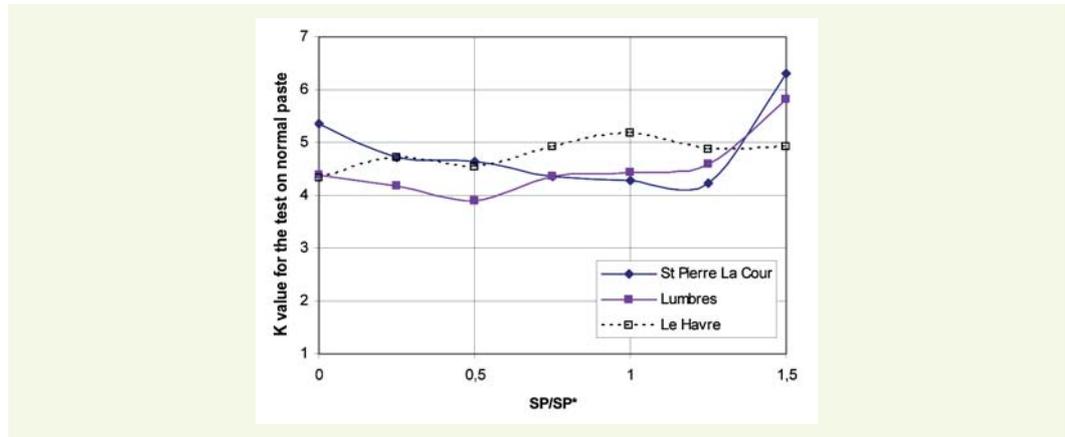


Figure 5 :
Comparison between the experimental packing density values measured on normal pastes and the theoretical values deduced from tests conducted on homogeneous pastes with $K = 4.8$.

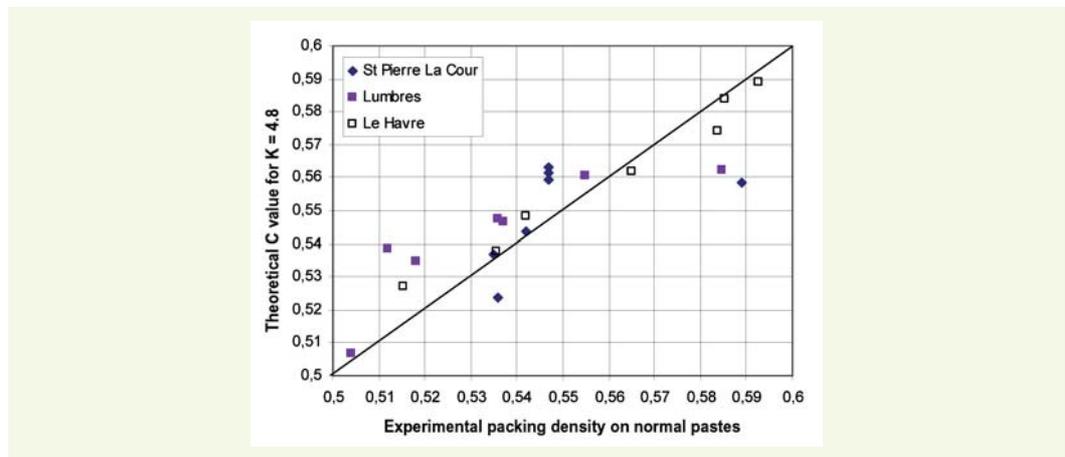


Table 4 :
Values of β calibrated using the CPM. β (6.7) : calibrated values using packing densities measured on homogeneous pastes with a packing index of 6.7 ; β (4.8) : calibrated values using packing densities measured on normal pastes with a packing index of 4.8.

Sp/Sp*	Saint-Pierre-la-Cour		Lumbres		Le Havre	
	β (6.7)	β (4.8)	β (6.7)	β (4.8)	β (6.7)	β (4.8)
0	0.4687	0.4813	0.4518	0.449	0.4724	0.4604
0.25	0.4821	0.4803	0.4802	0.4632	0.4832	0.481
0.5	0.4918	0.4875	0.4839	0.457	0.4941	0.4874
0.75	0.5054	0.4926	0.4931	0.4812	0.508	0.5111
1	0.5074	0.4926	0.4921	0.4824	0.5209	0.5307
1.25	0.5094	0.4926	0.5067	0.5007	0.5309	0.5326
1.5	0.5044	0.5364	0.5085	0.5317	0.5364	0.5401

■ Verification for composite binders

The results obtained on cement and silica fume mixes are shown in **Table 5**. Cement packing densities, which had not been measured for a 0.6 % (i.e. 2SP*) concentration with either of the two methods, was thus extrapolated linearly based on results obtained for the mixes with 5 % and 10 % silica fume. The silica fume packing density was also not measured directly since the presence of cement is necessary for the admixture to play its designated role; consequently, this packing density was initially calibrated for tests on homogeneous pastes in such a way that the CPM was fitted optimally the experimental results. Having determined the packing density of both cement and silica fume in this manner for $K = 6.7$, their corresponding β values can be deduced, i.e. 0.5426 and 0.6321, respectively. The simulations using the CPM were performed in accordance with this approach, at $K = 4.8$, in order to reconstruct the compaction evolution stemming from tests on normal pastes. **Figure 6** reveals that the model quite reasonably describes these experimental results. Moreover, the model yields the same average deviation (around 0.01 in absolute value terms) with respect to experimental results for both methods. This observation confirms the acceptability of the value 4.8.

In the present study and for the sake of homogeneity, a single mixing cycle has been selected for the entire series of tests on normal pastes, whereas Lecomte *et al.* [3] had called for different mixing times for pastes whether with or without an admixture, i.e. :

- without an admixture : after 90 seconds of slow mixing, the mixer was stopped and the tank scraped with the blade for 15 seconds, before another 90 seconds of slow mixing ;
- with an admixture : the mixing cycle was applied two times in a row.

Following an analysis of test results, it can be concluded that this difference in mixing protocol only exerts a minor impact on water demand measurements inasmuch as the same packing index value is derived in both instances. The mixing protocols proposed by Lecomte *et al.* however offer a double advantage, namely :

Table 5 :

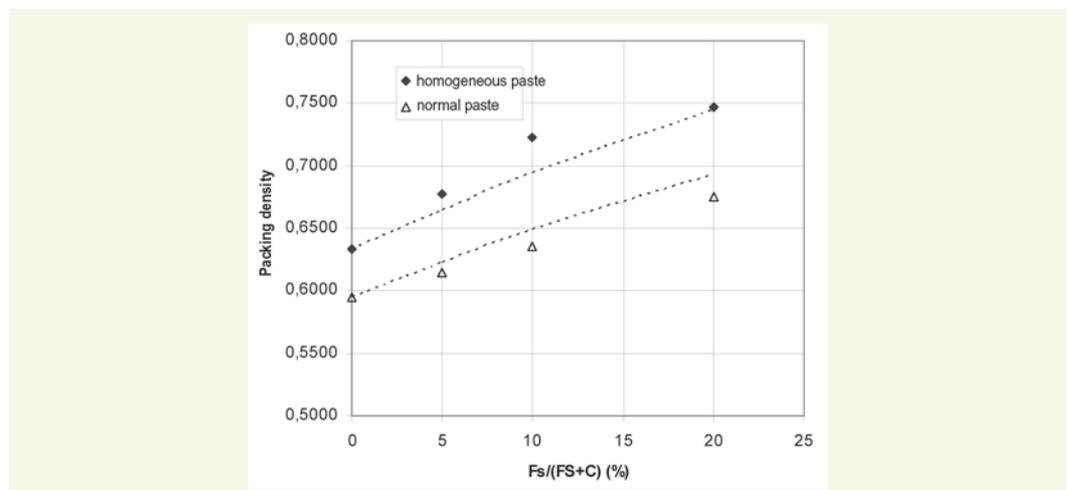
Results from packing density measurements for the Saint-Pierre-la-Cour mix + SEPR silica fume

FS/(C +FS) (%)	Homogeneous paste	Normal paste
0	0.6333*	0.5948*
5	0.6780	0.6147
10	0.7228	0.6353
20	0.7467	0.6752

* values extrapolated from measurements conducted at both 5% and 10% silica fume

Figure 6 :

Experimental and simulated packing density values : The points represent the experimental packing density values, and the dashed lines the simulations performed using CPM with the B values of cement and silica fume calibrated from tests on homogeneous pastes. Calculations were conducted using $K = 6.7$ and 4.8 for tests on homogeneous and normal pastes, respectively.



- for measurements conducted on cement without admixture, the proposed protocol is that of Standard EN NF 196-3, and the water demand values are typically available from the cement supplier ;
- adopting a double mixing cycle for the mixes with additives does in general seem to generate more regular curves than those obtained herein (see Fig. 3). Furthermore, for some cements and water content values very close to the water demand, a single mixing cycle does not yield a homogeneous paste.

CONCLUSION

The pertinence of using the water demand of a paste with normal consistency, as intended in Standard EN 196-3, to determine the packing density of powders has been confirmed in this study. Without admixture, the selected mixing cycle is the one specified in the standard. For measurements including admixture, the cycle gets repeated a second time. This test produces results consistent with those derived from the water demand test used to obtain a homogeneous paste ; which was the reference test up until now. The packing index value of 4.8 proposed by Lecomte et al. has also been confirmed. This new method offers the advantage of being less subjective than the previous one and relies upon an actual existing standard; nonetheless, it does require more time to complete. The protocol adopted in this study will thus be proposed as an alternative method for measuring the water demand of powders in Version 3 of the *BetonlabPro* software.

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