Some key cement factors that control the compatibility between naphthalene-based superplasticizers and ordinary portland cements

by Pierre-Claver Nkinamubanzi, Byung-Gi Kim and Pierre-Claude Aïtcin

Synopsis: Superplasticizers improve the workability of concrete at low water-cement ratios but this workability is sometimes lost rapidly in the first hour after contact between the cement and water. This is especially the case for naphthalene-based and melamine-based superplasticizers when used with the so-called incompatible cements. It is possible to reasonably predict the rheological behavior of a given Portland cement when used with a naphthalene-based superplasticizer once the physico-chemical composition and the properties of the clinker, of the cement, and of the superplasticizer are known.

Sixteen different Portland cements, having a wide range of C_3A contents (6.0 to 11.8%) and SO_3 contents (0.09 to 2.90%), made with clinkers having a wide range of alkali contents (0.07 to 0.87% of Na_2O_{eq}), have been selected to highlight the key cement factors that control the compatibility between ordinary Portland cements and the naphthalene-based superplasticizers.

The soluble alkalis (in fact the soluble SO_4^{2-} ions from alkalis), the fineness, the C_3A content, and the type of $CaSO_4$ of the cement are among the key cement factors that control the rheological behavior of a superplasicized cement paste and concrete. An optimum amount of soluble alkalis content exists and ranges between 0.4 and 0.6% of Na_2O_{eq} in the cement. The rate of the naphthalene-based superplasticizers' adsorption on the cement particles and cement hydrates, which is influenced by these parameters, controls the loss of fluidity of the concrete. Moreover, this study has shown that concrete made with cements that have a low soluble alkalis content not only lose their slump when underdosed in superplasticizer but also present severe segregation and bleeding when the dosage of the superplasticizer is slightly higher than the saturation point. In this case the cement/superplasticizer combination is said to be **"non-robust".**

Key words: adsorption, alkalis content, alkaline sulfates, cement, clinker, compatibility, grout, high performance concrete, mini-slump, Marsh cone, naphthalene superplasticizer, segregation, slump loss, soluble alkalis, soluble sulfates, sulfonate superpalsticizer, robust combinations, robustness.

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INTRODUCTION

The use of superplasticizers in concrete having a low water to cement ratio highlights the problem of more or less rapid slump loss in the case of some combinations of cement and superplasticizers, while in other cases a high slump can be maintained during the first 60 to 90 minutes following contact between the cement and water, without any sign of segregation and bleeding. In the first case, the cement and the superplasticizer are said to be non-compatible, and in the second case, they are said to be compatible.

Compatibility problems are now well documented in the case of polynaphthalene sulfonate-based superplasticizers (PNS) which are presently the most used superplasticizers in the concrete industry. But in the literature, some cases of incompatibility involving normal water reducers based on lignosulfonates have been reported **(1)**.

When studying the rheological behavior of superplasticized grouts with PNS using a Marsh cone, it has been found that there exists a critical dosage beyond which any additional increase of PNS does not generate an increase in fluidity of the cement paste and the initial slump of concrete. This point had been called the saturation point and the PNS dosage at this point is called the saturation dosage **(2).**

When studying the evolution of flow time trough a Marsh cone as a function of superplasticizer dosage, some cements do not present any difference between their flow time at 5 minutes and at 60 minutes after contact between the cement and water, while for some other cements the flow time increases very much even when a higher dosage of PNS is used.

In some cases, the increase of PNS dosage beyond the saturation point allows concrete to maintain its high slump for a long time while in some other cases any increase of PNS dosage beyond the saturation point leads to segregation and bleeding. In the first case the combination of cement and PNS is said to be **"robust"** while in the second case, the combination can be described as **"non-robust"(3).**

The cement composition and physico-chemical properties, especially its C_3A content, its fineness, the nature of the calcium sulfate used during the clinker grinding, and the sulfatisation degree have been identified, in some studies, as important parameters that influence the compatibility between cement and polysulfonated superplasticizers **(4).** Recently, the soluble alkalis (in fact alkali sulfates) of the cement had been identified as an important parameter **(5)**. For each combination of cement/polysulfonated superplasticizer it seems that there exists an optimum content of soluble alkali (**6-8**). In cement with low alkali content the addition of a small amount of sodium sulfate improves significantly the rheological behavior of a grout and of a concrete made with such cements (**9**). The use of a PNS with a high residual sulfate content can also improve the slump retention of the concrete (**10**).

It is also known that a delayed addition or a double dosage of superplasticizer can be used to maintain an adequate rheological behavior to some cement/superplasticizer combinations. In fact, when a polysulfonated superplasticizer is introduced at the beginning of the mixing process of the concrete, it can interact with the C_3A of the cement and form an organomineral complex while it is just slightly adsorbed by ettringite when it is added later during the mixing process of the concrete (**11, 12**).

In order to understand more clearly the reason for such incompatibilities, which occur from time to time between some cements and polysulfonated superplasticizers, a systematic study had been conducted on 16 different Portland cements, having a wide range of C_3A content (1.3 to 11.8%) and SO_3 content (0.09 to 2.90%), made with clinkers having a wide range of alkali contents (0.07 to 0.87% of Na_2O_{eq}).

MATERIALS

The cements studied, and their physico-chemical properties are presented in Table 1. The clinker composition is given in Table 2. It is important to have the clinker composition to have a clear idea of the source of SO_3 in a given cement. It can be seen that the clinker SO_3 content varies from 0.09 to 2.90 %, which means that the SO_3 from calcium sulfate added to the clinker during the grinding process varies from 0.04 to 3.12 %. The alkali sulfates are much more soluble and have a higher dissolution rate than the calcium sulfates, so that they are more rapidly available in the interstitial solution of the cement pastes in first minutes after contact between the cement and water.

The superplasticizer used is a well-characterized sodium polynaphthalene sulfonate. It is known to have a high purity and other desired functionalities for this type of superplasticizer (**12**). Its molecular weight distribution is shown in Figure 1.

EXPERIMENTAL PLAN

A systematic study of the rheological behavior of grouts made with the sixteen cements has been undertaken using the mini-slump test to find the incompatible combinations and the Marsh cone test to determine the saturation dosage of PNS for each cement. The water/cement ratio of the grouts is 0.35.

Once the saturation point on grouts was obtained, concrete having a water/cement ratio of 0.30 was made to confirm and validate the results obtained on grouts. The slump of the fresh concrete was monitored during 90 minutes following contact between the water and cement, after which specimens were sampled in order to measure the mechanical properties of the hardened concrete.

Chemical analysis of alkaline and other ions present in the interstitial solution of the grout (W/C = 0,35) was performed using the ICP (Induced Coupled Plasma) analysis and the amount of PNS adsorbed on cement grains was measured by UV spectrometry.

RESULTS AND DISCUSSIONS

Tests on grouts

Rheological behavior—The mini-slump test carried out on grouts made with the 16 cements containing 1% of PNS allowed us to find the systems that did not present any compatibility problem. Figure 2 shows the rheological behavior of the 16 cements, expressed as the spread area (mini-slump) during two hours after contact between the water and cement. It is clear that cements A1, D1, and D2 do not have enough fluidity, or lose it rapidly, in the conditions under study. The chemical composition of these cements (and of the clinker used to produce them) shows that their alkali and the sulfate content of the clinker are very low. These results obtained with the mini-slump test are confirmed by the results obtained with the Marsh cone test. The variation of the flow time as a function of time is illustrated in Figures 3. The Marsh cone test shows also that the dosage of 1% used for the mini-slump test was too high in some cases so that it hid the lack of compatibility of some cements which were overdosed (cement C1).

Superplasticizer adsorption—It is now well known that naphthalene-based superplasticizers proceed mainly by adsorption on the cement grains and the cement hydrates when dispersing cement pastes (**12, 13**). The adsorption of PNS on the cement grains has been measured for the sixteen cements. For this purpose, grouts having water to cement ratios (W/C) of 0.35 were prepared and the interstitial solution extracted by applying pressure. The PNS content of the solution was determined by UV spectrometry (**12**). Table 3 shows the results obtained for the 16 cements. The affinity between the cement grains and the superplasticizer leads to a consumption of the latter from the interstitial solution by adsorption. This phenomenon results in a loss of fluidity if there is not enough superplasticizer remaining in the solution to ensure a good fluidity of the cement grain and the cement hydrates (**11, 12**). This is the case of cements with low alkali and alkaline sulfates. The superplasticizer in the mixing water acts as a sulfate ions provider and interacts with the C_3A instead of performing its dispersing role. It is clear from Table 3 that cements A1, C1, D1, and D2 that have the lower alkali content exhibit a strong adsorption of PNS. More than 75% of initial PNS is consumed within the first five minutes following contact between the cement and the mixing water. In the case of the cements having a high alkali content, more than 50% of the PNS remains in the interstitial solution. Figure 4 presents the relationship between the adsorbed PNS and the alkali (and alkaline sulfates) content of the sixteen cements. It is clear that the amount of PNS adsorbed on the cement grains decreases quasi-linearly when the alkali and alkaline sulfates content increase in the cements.

These results also show that cements having a good rheological behavior (i.e. no fluidity loss) have an alkali content between 0.4% and 0.6% (that is to say about 150 mmol/l). Figure 5 presents the relationship between the fluidity of the cement pastes and the adsorption of the PNS. The fluidity of the grouts obtained by the mini-slump test decreases for high PNS adsorption, i.e. for cements having low soluble alkaline sulfate contents. When sodium sulfate is added to cements A1 and D1, which have a low alkali content, the PNS adsorption decreases but an optimum is not observed (Fig.6).

Tests on concrete

Tests on grouts allow a preliminary analysis of compatibility problems between the cements and the superplasticizer but cannot insure a faultless rheological behavior of the concrete made with the same cement and the same superplasticizer. Validation of the results obtained on grout is then necessary on concrete.

The concrete composition used in this study is given in Table 4. The superplasticizer dosage varies with the cement used, as the saturation dosage changes with the cement. The sand used was a natural siliceous sand having a fineness modulus of 2.50, a SSD specific gravity of 2.65 and an absorption of 1.20%, while the coarse aggregate was a crushed metamorphic limestone having a SSD specific gravity of 2.71 and an absorption of 0.35%. The superplasticizer, diluted in 1/3 of the mixing water, is added 90 seconds after the contact between the cement and the first 2/3 of the mixing water. The initial slump targeted was 200 ± 20 mm. The slump of the concrete was monitored during 90 minutes when possible.

The properties of the fresh concrete and the mechanical behavior of the hardened concrete are summarized in Table 5. For the cements having a high alkali content, we did not experience too many problems achieving a good workability of the concrete during 90 minutes and the superplasticizer dosage was adjusted until the desired workability was achieved (Figure 7). The results obtained with the concrete usually correspond to those obtained with the grout, with sometimes a small adjustment.

In the case of the cements having a low alkali content, the situation is very different. It was possible to achieve a good initial slump when an adequate superplasticizer dosage was used, but the loss of slump was very rapid in some cases (Figure 8). When the dosage was slightly increased beyond the saturation point, a strong segregation and bleeding occurred in most of these low alkali cements. The concrete made with this kind of cement shows a lack of "**robustness**" with the PNS used. It will not be easy on the building site to produce, with this kind of combination of cement and superplasticizer, a high performance concrete having a very low water cement ratio. The delayed addition and double dosage of the superplasticizer (half at the beginning of the concrete mixing and the other half after 5 minutes) did not correct this lack of **robustness**. Figure 9 presents the slump loss of concrete made with some of the sixteen cements during the 90 minutes following contact between the cement and water. The low soluble alkali cements are the ones that lose rapidly their slump. Using the ratio of soluble SO_{3sol} and C_3A calculated for the sixteen cements as a function of their total alkali content, it has been possible to classify the cements in three groups (Figure 10): **compatible and robust** for high soluble SO_3 and high alkali content cements, **less compatible and less robust** for medium soluble sulfate and alkali content cements and **incompatible or non-robust** for low soluble sulfate and low alkali content cements. Six other cements (K*i*) tested in an other study have been added in the figure to strengthen this relationship.

CONCLUSION

The careful selection of the cements used in this study allowed to highlight the importance of the soluble alkali and alkaline sulfate content of a cement when it is used to produce a high performance concrete with a sulfonate-based superplasticizer. Although the fineness of the cement and the aluminate phase content play a major role on the superplasticizer dosage of the concrete, the soluble alkaline sulfates content has to be optimized. The optimum soluble alkali content was between 0.4 and 0.6% in this study. The strong adsorption of the sulfonate-based superplasticizer by the cements having a low alkaline sulfate results in a drastic slump loss of the concrete. The superplasticizer adsorption decreases quasi-linearly when the soluble sulfate content (and the soluble alkali content) increases. The concrete made with cements having a low soluble alkali content not only lose their slump when underdosed, but also present severe segregation and bleeding when slightly overdosed. This is not the case of the cements that have a suitable soluble alkali content. The amount of rapidly soluble alkali sulfate was found to be a key parameter in the **"robustness"** of the cements and the sulfonate-based superplasticizer combinations.

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Table 1 – Bogue composition and alkali content of the cements

Cement	**Na_2O**	**K_2O**	**Na_2O_{eq}**	**Na_2O_{sol}**	**C_3S**	**C_3A**	**C_4AF**	**SO_3**	**S.D.****
A1	0.30	0.69	0.75	0.25	57	8.3	9.6	3.52	41
A4	0.14	0.80	0.67	0.44	55	7.3	8.9	3.46	106
A6		0.64	0.42	0.41	66	6.9	10.4	3.56	96
A7	0.10	0.92	0.71	0.62	47	7.0	10.7	3.45	141
B3*	0.16	1.05	0.85	0.48	53	6.7	8.7	3.32	129
B4*	0.16	1.05	0.85	0.45	54	6.8	9.0	3.15	129
B8		0.77		0.41	52	8.0	9.6	2.94	443
B10	0.17	0.70	0.63	0.46	59	8.7	8.6	3.50	147
C1	0.16	0.16	0.22	0.22	66	9.4	5.5	2.93	286
C3	0.19	1.05	0.58	0.58	66	8.0	7.7	3.66	124
C4	0.05	0.98	0.47	0.47	53	6.2	9.6	3.51	256
C6	0.22	0.53	0.42	0.42	57	10.0	5.8	3.33	161
D1	0.02	0.15	0.04	0.04	61	6.8	11.3	2.74	691
D2	0.03	0.06	0.03	0.03	64	11.5	0.9	2.78	100
D3	0.19	0.48	0.55	0.55	55	7.7	9.1	2.79	47
D4	0.09	1.19	0.60	0.60	54	10.5	7.0	3.49	67

* The two cements differ only by their calcium sulfate form; cement B3 : high gypsum. high anhydrite. low hemihydrate; cement B4:low gypsum. high anhydrite. high hemihydrate.

** Sulfatisation degree

Table 2 – Chemical analysis of the clinkers

	Chemical analysis of the clinkers (% oxides)													
	L.O	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	Na_2Oéq	C_3S	C_2S	C_3A	C_4AF
A_1		20.66	5.55	3.54	66.28	0.9	0.69	0.3	0.4	0.75	70.5	6.1	8.7	10.8
A_4		20.54	4.83	3.02	63.84	4.79	1.04	0.19	0.87	0.87	56.1	16.7	7.7	9.2
A_6		21.10	4.98	3.6	67.34	1.25	0.64	0.11	0.52	0.53	63.7	12.2	7.1	10.9
A_7		21.07	5.12	3.7	66.03	1.26	0.92	0.1	1.28	0.71	57.8	16.9	7.3	11.3
B_3		21.41	4.62	2.92	65.89	1.71	1.05	0.16	1.42	0.85	66.0	11.6	7.3	8.9
B_4		21.41	4.62	2.92	65.89	1.71	1.05	0.16	1.42	0.85	66.0	11.6	7.3	8.9
B_8		20.30	5.15	3.07	64.50	1.50	0.77	—	2.90	0.51	69.3	6.0	8.5	9.3
B_{10}		20.70	5.30	3.15	65.82	2.13	0.70	0.17	1.20	0.63	64.4	10.8	8.7	9.6
C_1	0.15	22.11	4.98	2.25	68.51	0.80	0.16	0.16	0.98	0.27	68.31	11.7	9.4	6.84
C_3	0.30	20.96	5.44	2.95	66.39	0.78	1.05	0.19	1.41	0.88	62.46	12.43	9.43	8.95
C_4		21.25	4.73	2.76	65.97	1.71	0.98	0.05	2.30	0.70	60.93	14.83	7.86	8.4
C_6	0.11	22.25	5.05	1.86	66.96	1.09	0.53	0.22	1.18	0.57	60.55	17.84	10.24	5.65
D_1	0.73	21.58	4.87	3.95	67.32	0.51	0.15	0.02	1.06	0.12	71.6	7.9	6.2	12.0
D_2	0.95	23.84	4.65	0.33	69.53	0.49	0.06	0.03	0.09	0.07	70.1	15.5	11.8	1.0
D_3	0.82	21.96	5.13	3.02	64.55	3.42	0.48	0.19	0.31	0.51	57.0	20.0	8.5	9.2
D_4	0.80	21.46	5.47	2.31	66.29	0.98	1.19	0.09	0.76	0.87	66.7	11.2	10.6	7.0

Table 3 – Amount of PNS adsorbed on the cements: The dosage of PNS is 1%

Cements	**NaO_{sol} (%)**	**% PNS_{ads} 5 min**	**% PNS_{ads} 60 min**	**Blaine (m^2/kg)**	**% ads/Blaine 5 min**	**% ads/Blaine 60 min**
A1	0.25	85	87	570	0.15	0.15
A4	0.44	61	68	515	0.12	0.13
A6	0.41	55	60	445	0.12	0.13
A7	0.62	54	58	480	0.11	0.12
B3	0.48	53	57	435	0.12	0.13
B4	0.45	51	54	435	0.12	0.12
B8	0.41	57	59	295	0.19	0.20
B10	0.46	54	62	370	0.15	0.17
C1	0.22	74	78	415	0.18	0.19
C3	0.58	42	51	415	0.10	0.12
C4	0.47	49	51	395	0.12	0.13
C6	0.42	54	57	360	0.15	0.16
D1	0.04	88	91	330	0.27	0.28
D2	0.03	93	94	460	0.20	0.20
D3	0.55	45	45	360	0.13	0.13
D4	0.60	48	51	385	0.12	0.13

Table 4 – Concrete composition

W/C	0.30	
Water	140	kg/ m^3
Cement	470	
Fine aggregate	790	
Coarse aggregate	1050	
PNS*	5.5 to 14	l/m^3

* The PNS dosage depends on the type of cement

Table 5 – Summary of the main results on concrete: W/C = 0.35

Cement	**Optimum dosage**	**Slump (mm)**			**Comp. strength (MPa)**	
		Initial	*60 min*	*90 min*	*24 h*	*28 d*
A_1	0.8	250	180	160	55	93
A_4	1.25	240	225	200	54	85
A_6	1.0	240	200	160	54	87
A_7	1.0	230	230	230	57	79
B_3	0.8	230	180	150	50	78
B_4	0.8	200	180	150	49	73
B_8	0.8	220	220	220	44	72
B_{10}	0.8	220	110	80	51	74
C_1	0.6	210	120	*	49	70
C_3	0.6	230	210	200	46	74
C_4	0.6	200	160	100	47	65
C_6	0.8	230	210	200	51	72
D_1	0.7	210	60	*	37	69
D_2	0.7	230	190	150	53	79
D_3	0.8	200	170	140	45	64
D_4	0.8	240	230	230	45	69

* could not be measured

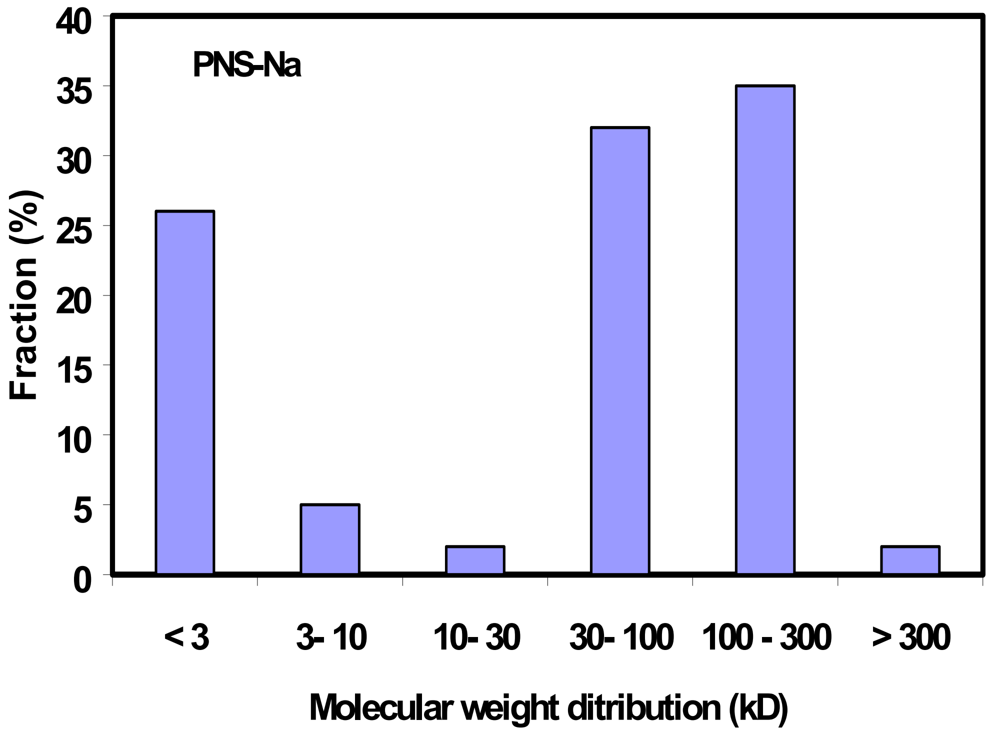


Fig. 1 – Molecular weight distribution of the superplasticizer used in this study

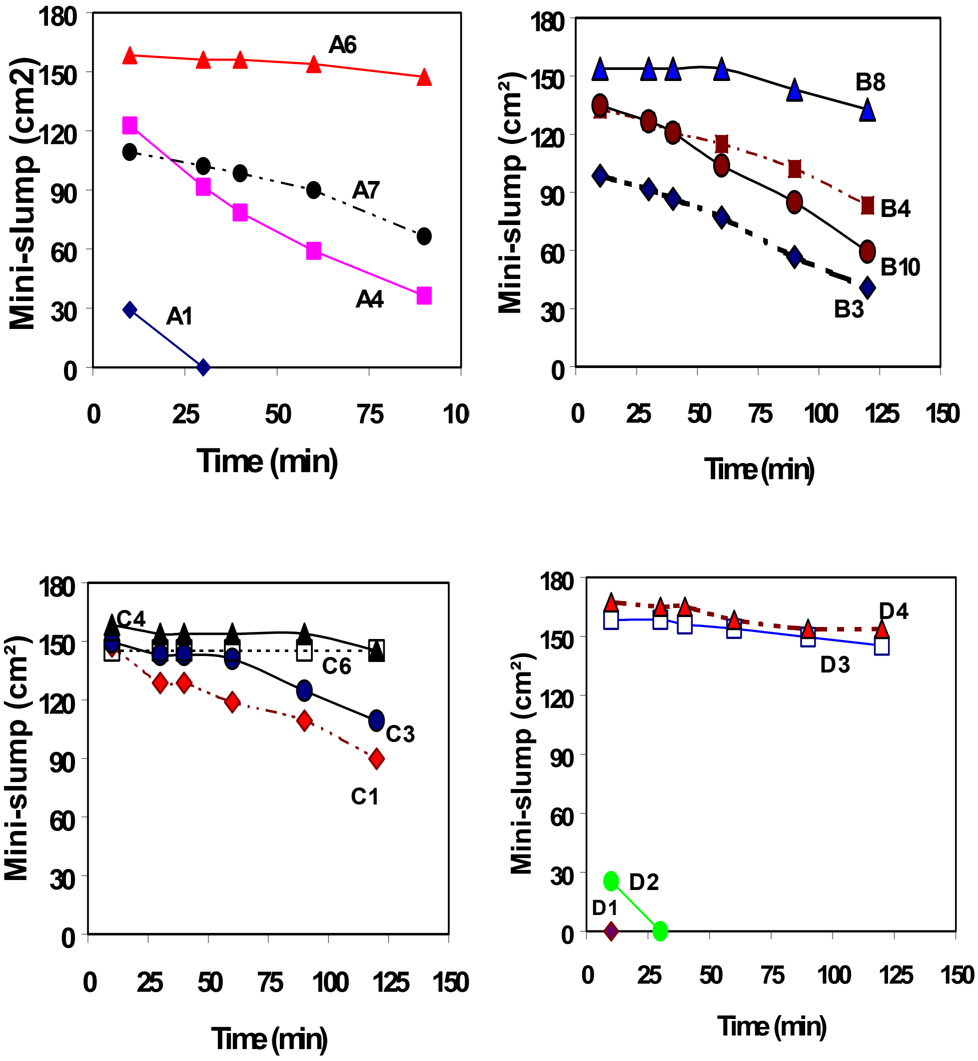


Fig. 2 – Rheological behavior of grouts of the sixteen cements

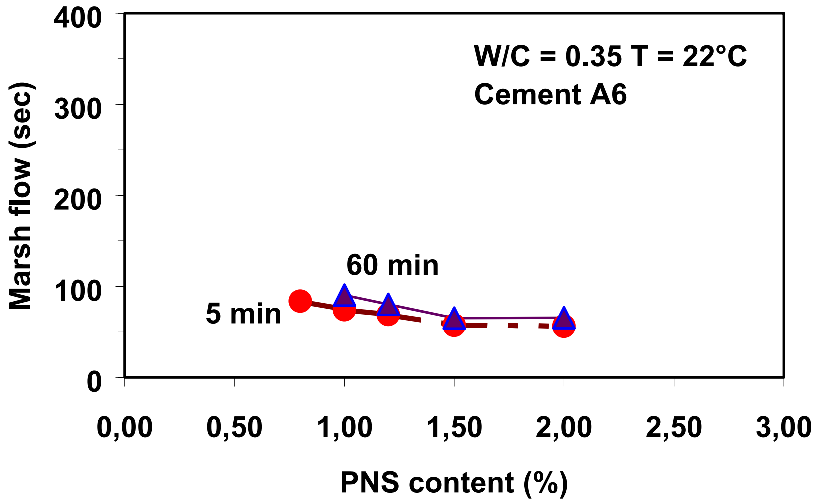


Fig. 3.a – Example of a compatible combination. The flow time is low and it does not vary between 5 and 60 minutes. This combination is also robust: a small variation of PNS dosage does not influence the flow behavior of the grout.

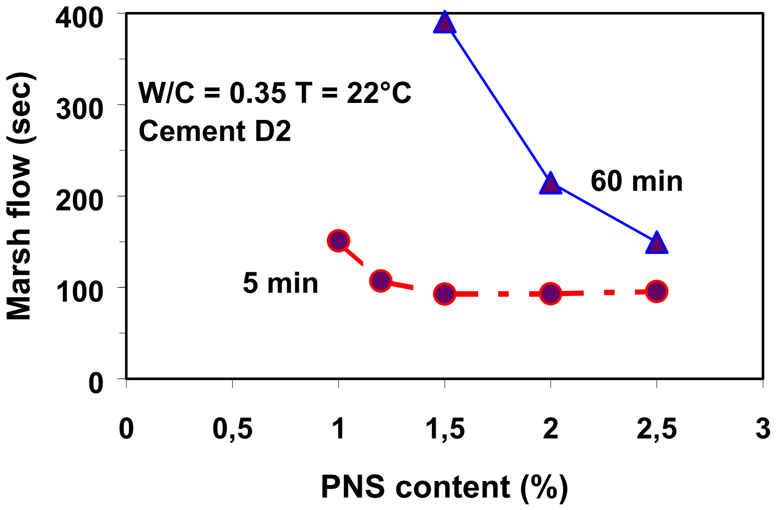


Fig. 3.b – Example of a non-compatible combination: the flowability of the grout is lost between 5 and 60 minutes.

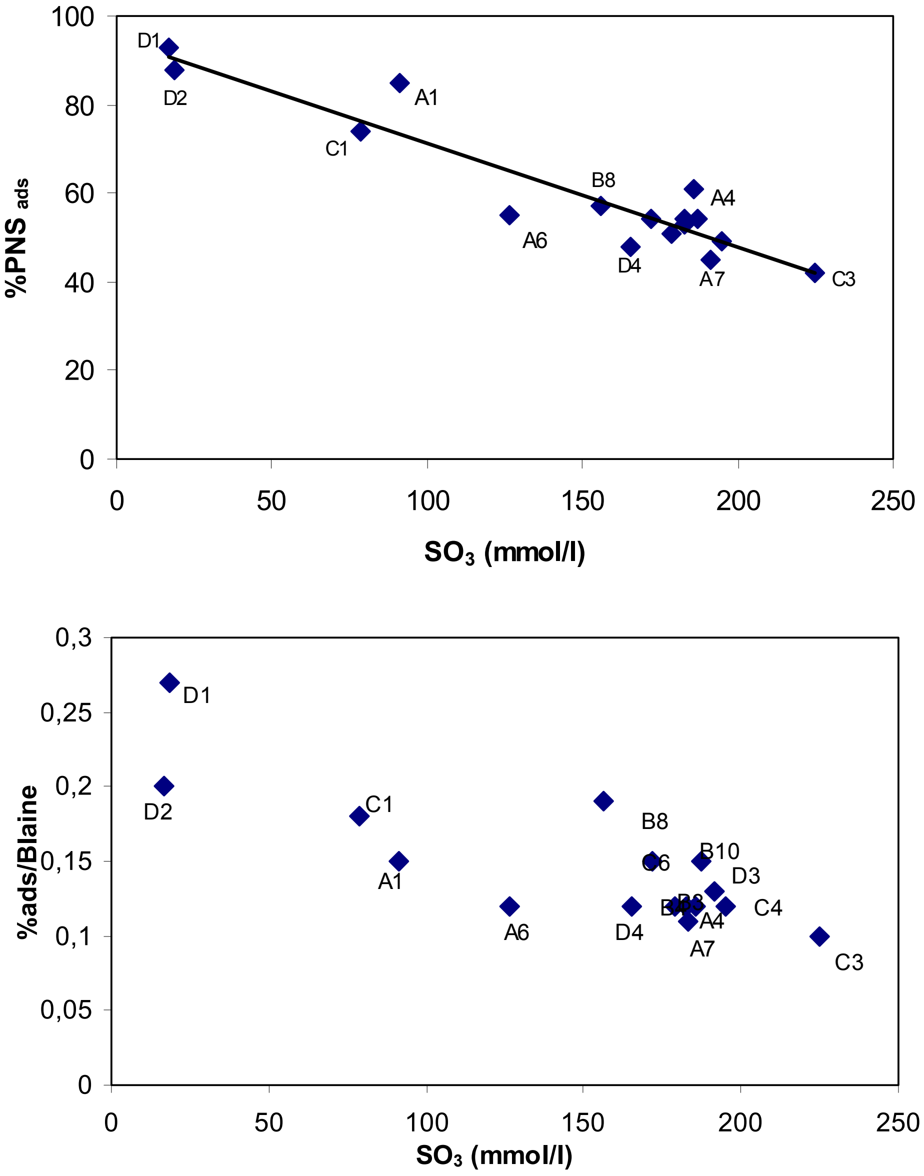


Fig. 4 – Adsorption of PNS as a function of the soluble sulfates of the cement

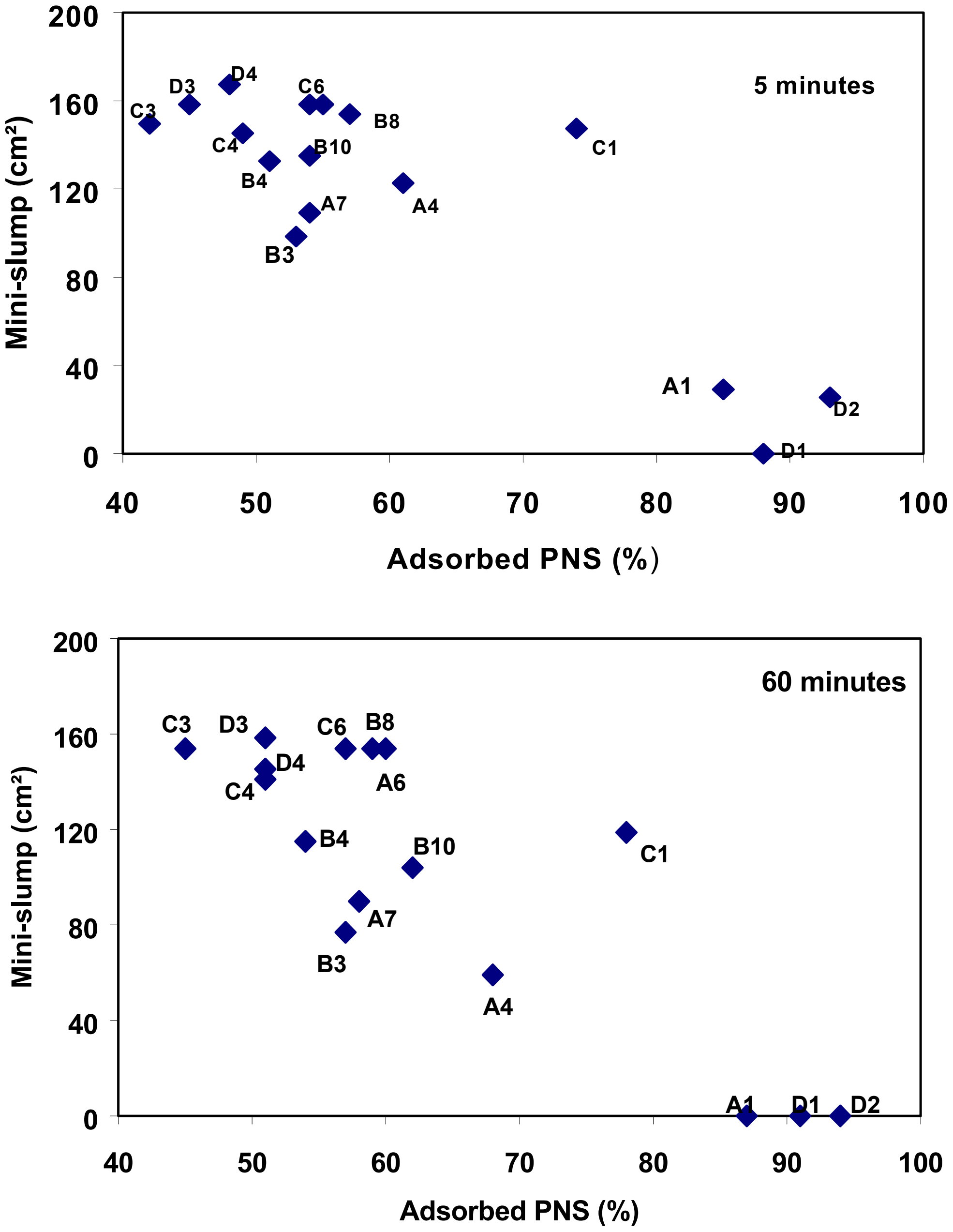
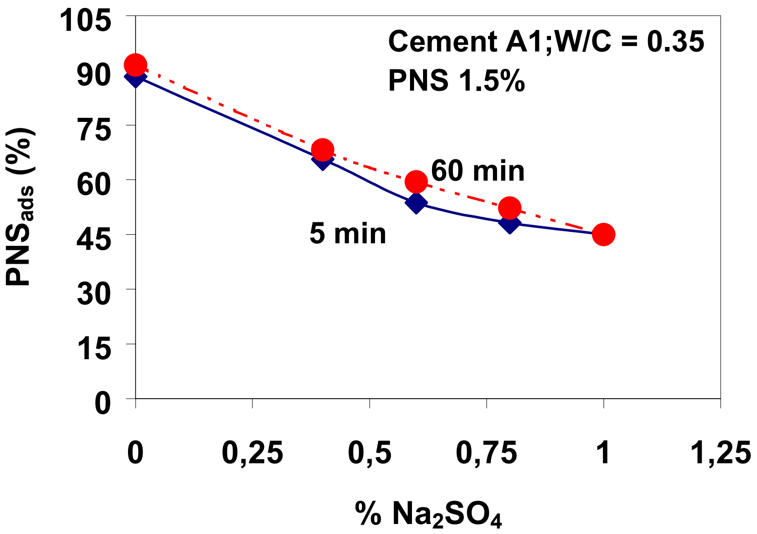


Fig. 5 – Relationship between the PNS adsorption and the rheological behavior of the grouts



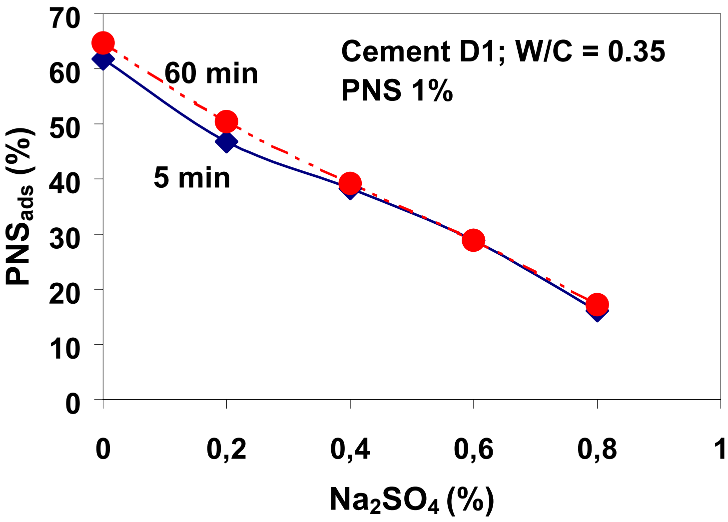


Fig. 6 – Effect of the addition of Na_2SO_4 on the PNS adsorption by cement pastes.

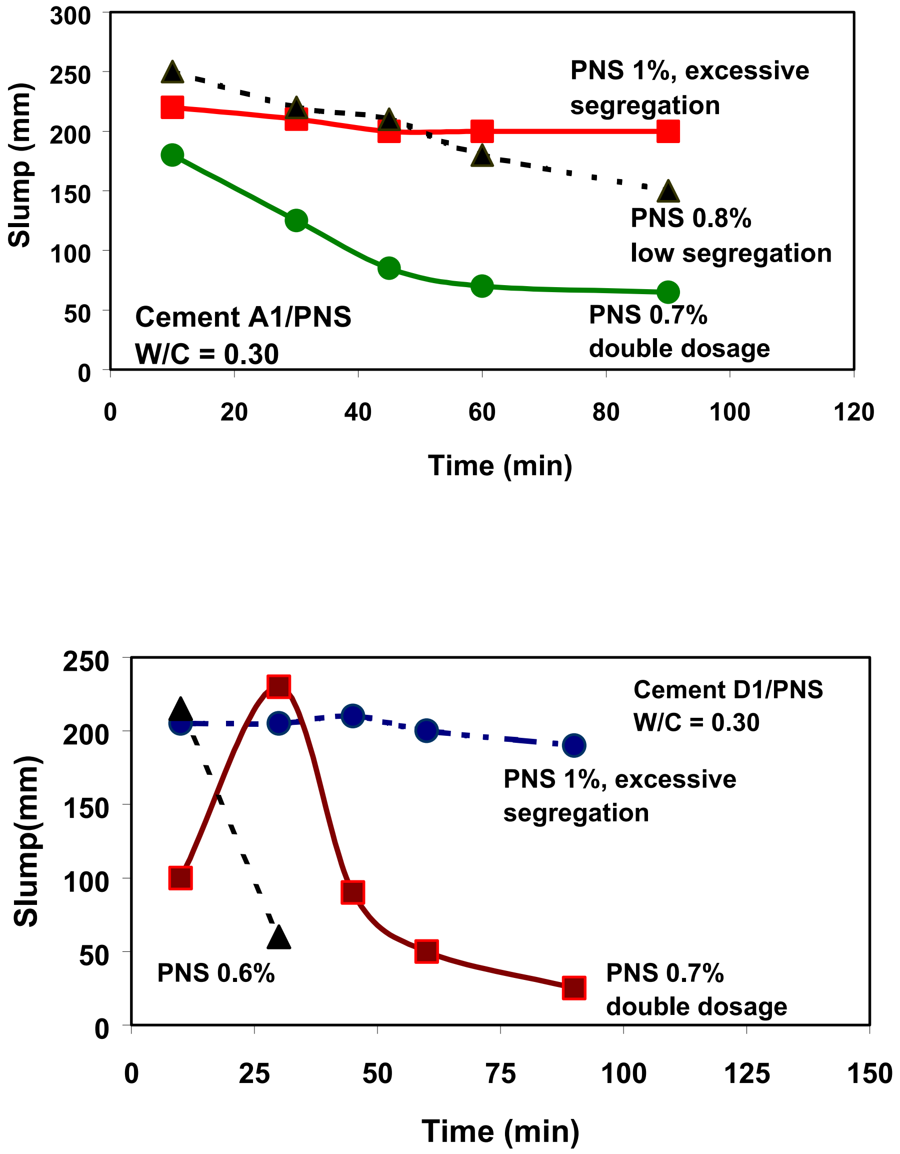


Fig. 7 – Examples of incompatible cement/PNS combinations. The lack of robustness is also illustrated.

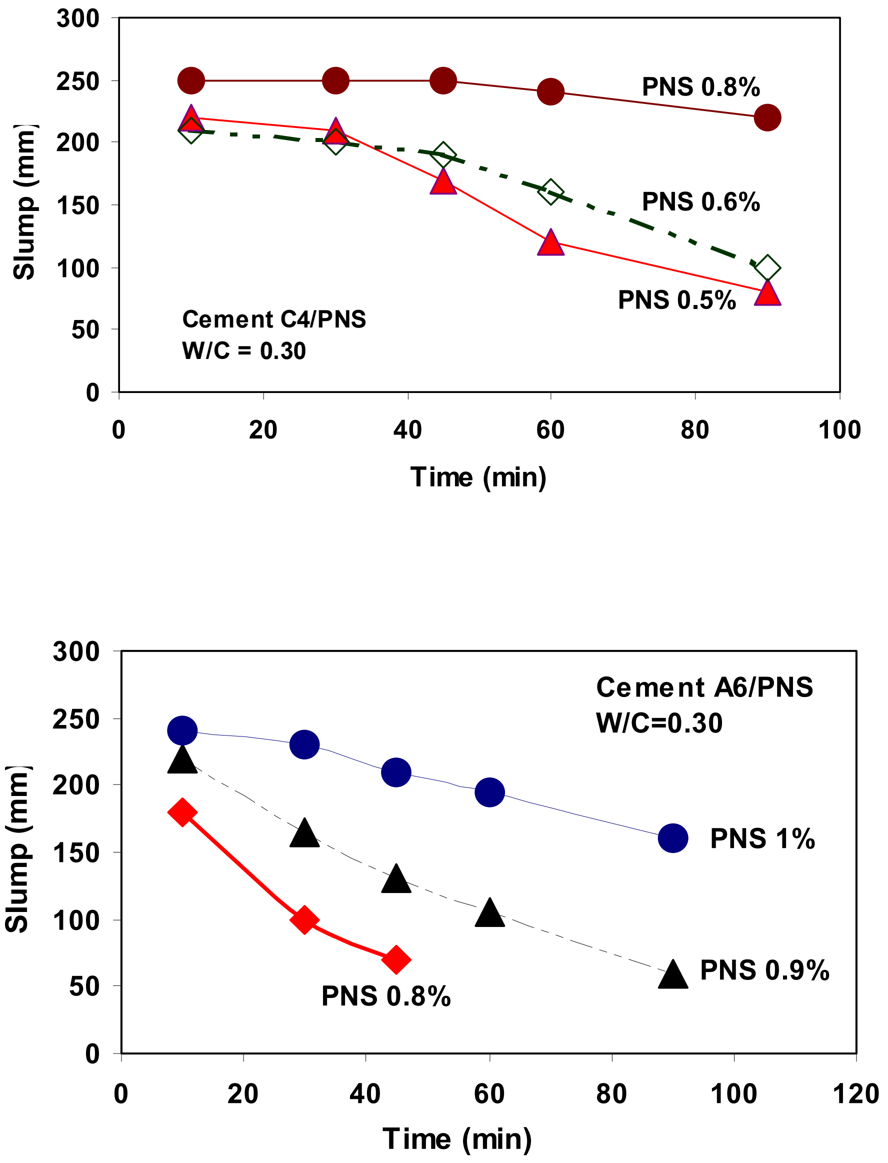


Fig. 8 –Examples of slump monitoring of compatible cement/PNS combinations

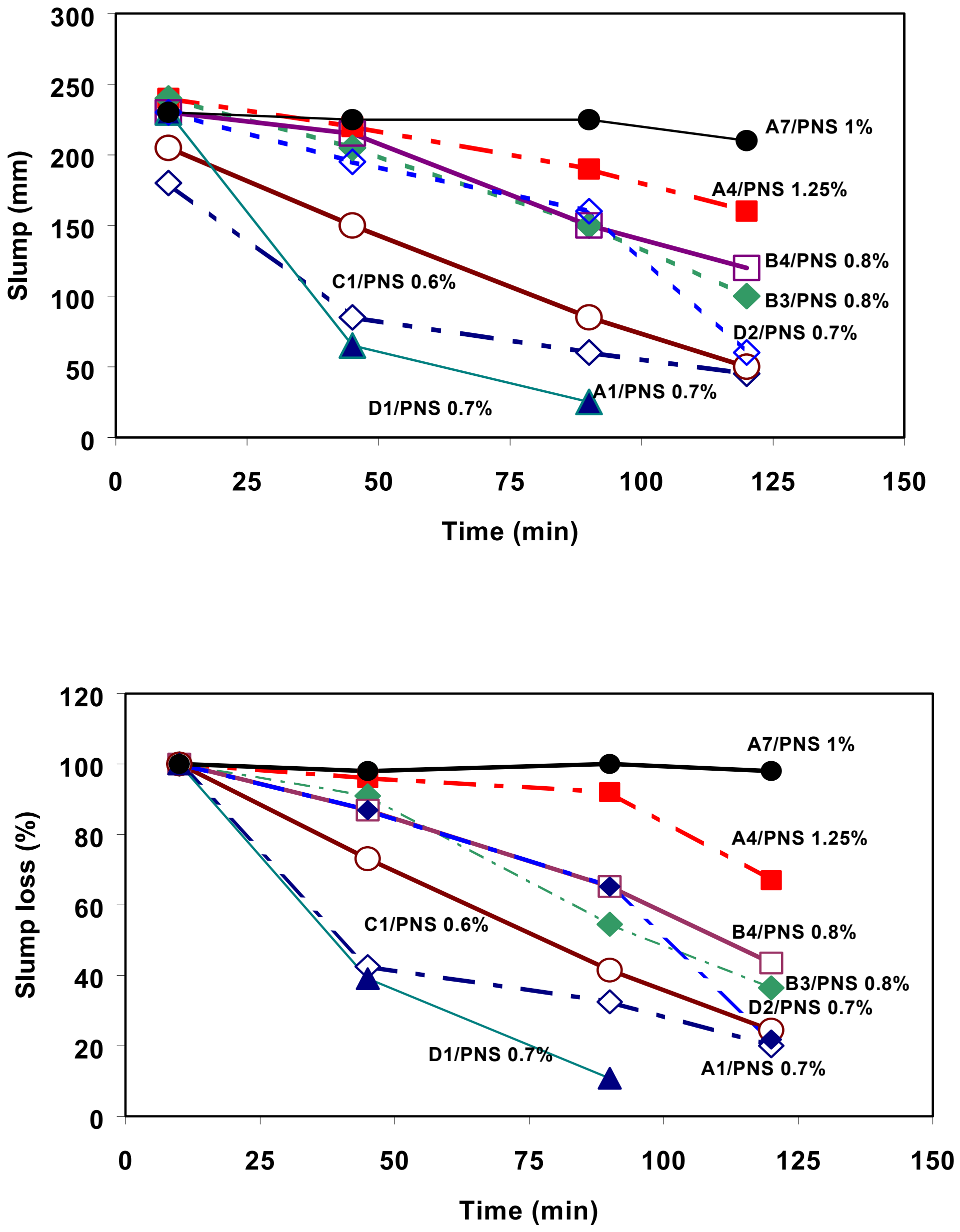


Fig. 9 – Comparison of the rheological behavior of concrete made with some of the sixteen cements at their PNS saturation points

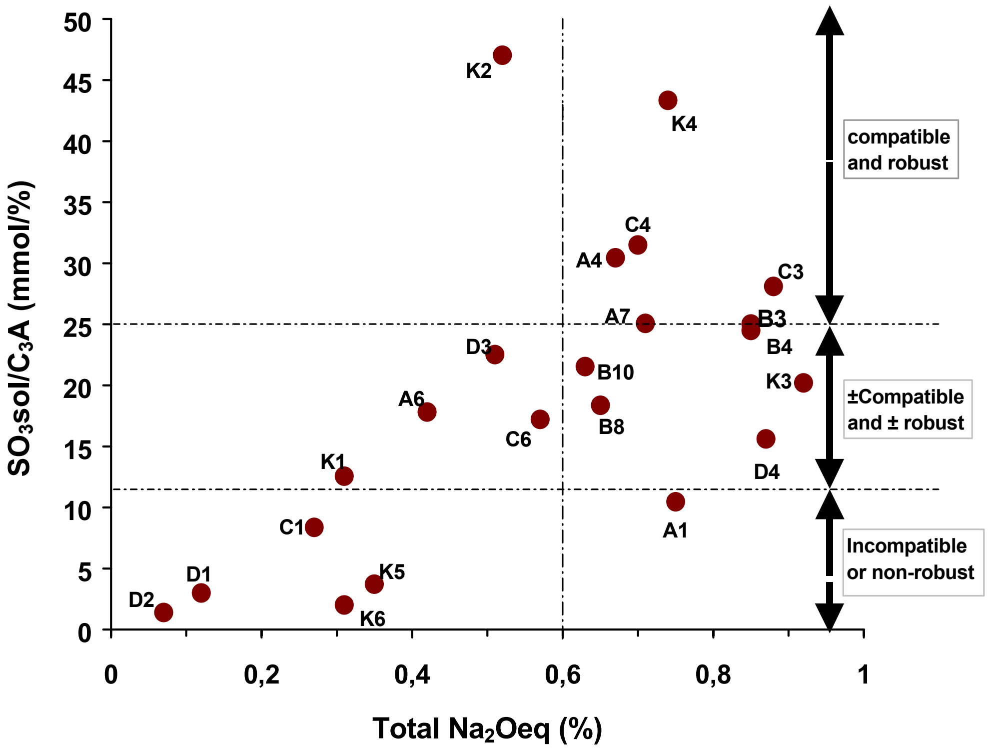


Fig. 10 – Compatibility and robustness of cements as a function of their Na_2O_{eq} content and their SO_3 sol./C_3A.