Cement and Concrete Research 41 (2011) 292–300

Contents lists available at ScienceDirect

Cement and Concrete Research

journal homepage: http://ees.elsevier.com/CEMCON/default.asp

Influence of cement grouts composition on the rheological behaviour

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ARTICLE INFO

Article history:
Received 14 April 2010
Accepted 23 November 2010

Keywords:
Cement D
Admixture D
Rheology A
Grout
Herschel–Bulkley

ABSTRACT

The influence of water to cement ratio, HRWRA content and viscosity modifying admixture content on the rheological characteristics of cement grouts considered as Herschel–Bulkley fluids is studied experimentally. Results show that cement grouts without chemical admixtures and cement grouts containing only a viscosity modifying admixture present a shear-thinning behaviour with an approximately constant value for the exponent n of Herschel–Bulkley model. On the contrary, grouts containing a HRWRA content near the saturation point exhibit quasi Binghamian behaviour. The Herschel–Bulkley model describes properly the rheological behaviour of cement grouts without chemical admixtures. It can be applied correctly to grouts containing HRWRA and/or viscosity modifying admixture in a shear rate range comprised between 4 s^{-1} and 100 s^{-1} and can be used to predict satisfactorily the Marsh cone flow time of cement grouts of widely varying compositions.

1. Introduction

Cement-based grouts are widely used for filling post-tensioning ducts to protect wire strands against corrosion and to transfer stresses between the strands and concrete. To facilitate pumping and to provide full voids penetration in the duct and proper coating of steel surface, grout mixtures should be fluid enough. Moreover, after grouting, grout mixtures should not present sedimentation of cement particles or bleeding of free water filling the upper parts of post-tensioning ducts to ensure corrosion protection, stress redistribution and freeze–thaw resistance [1]. High fluidity and stability of cement grouts are also the main requested properties for diverse grouting applications including anchorage, ground treatment, rock or soil permeability reduction, concrete repair and oil well completion [2]. To ensure high fluidity, stability and adequate mechanical properties, chemical admixtures such as High Range Water Reducing Admixtures (HRWRA) and viscosity modifying admixtures (VMA) are used separately or together in grout mixes with the aim to obtain appropriate properties [3]. The systematic study of the influence of these admixtures on the rheological behaviour of cement grouts is therefore of great importance in order to improve grouting results.

Many studies on the rheological behaviour of cement-based grouts have shown that these materials are viscoplastic fluids presenting a yield stress, which must be overcome by the shear stress so that the flow takes place [2,4,5]. The rheological behaviour of the grouts can be shear-thinning type [6–8] or shear-thickening type [9,10], depending on many parameters such as solid concentration (water/cement ratio), interaction between particles, size and shape of grains … [11]. It can be described satisfactorily by the Herschel–Bulkley model [11–13] characterized by three parameters: yield stress τ_0, consistency K and exponent n which relates, in the case of simple shear, the shear stress τ to the shear rate $\dot{\gamma}$ by the following relation (Eq. (1)):

$$\begin{cases} \dot{\gamma} = 0, & \text{if } \tau < \tau_0 \\ \tau = \tau_0 + K\dot{\gamma}^n, & \text{if } \tau \geq \tau_0 \end{cases}. \quad (1)$$

When $n = 1$, the Herschel–Bulkley model is reduced to the Bingham model which is also used in the literature to simulate the rheological behaviour of cement grouts [5,8]. However, the Bingham model cannot take into account the nonlinearity (curvature) of flow curve of the grout, which is generally significant at low shear rate [14]. It overestimates the yield stress for the shear-thinning fluids [14] or underestimates, even gives a negative yield stress, for the shear-thickening fluids [15].

Other analytical models (Casson, de Kee, ...) have also been proposed in the literature to describe the rheological behaviour of cement grouts [16]. However, these models have no further application in the flow description of cement grouts. Until now, only the Bingham [17] and the Herschel–Bulkley models [18,19] have been used to predict the Marsh cone flow time allowing to verify the validity of these models to describe the flow of cement grouts.

The purpose of this paper is to study the influence of water to cement ratio, HRWRA and VMA contents on the rheological characteristics of cement grouts considered as Herschel–Bulkley fluids and to verify the

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doi:10.1016/j.cemconres.2010.11.015

Table 1
Chemical and physical properties of cement.

Chemical composition	Mass (%)
CaO	64.0
SiO_2	20.3
Al_2O_3	5.2
Fe_2O_3	3.1
MgO	0.9
K_2O	0.8
Na_2O	0.21
Na_2O eq.	0.74
SO_3	3.1
CO_2	0.8
S^2	0.01
Cl^-	0.02
LOI	1.7
Free CaO	0.7
Insoluble	0.4
Main cement components	**Mass (%)**
Portland clinker	92
Calcareous filler	2
Gypsum	6
Bogue composition of the clinker	**Mass (%)**
C3S	57
C2S	24
C3A	9
C4AF	10
Physical properties of cement	
Density	3.11 kg/m^3
Blaine specific surface area	430 m^2/kg
Main particle diameter	20.5 µm
Water demand for standard paste	29%
Initial setting time	150 min
Final setting time	295 min
Compressive strength (28 days)	65.5 MPa

effectiveness of this model for the prediction of the Marsh cone flow time of grouts.

2. Materials and experimental methods

2.1. Materials

A Portland Cement (CEM I 52.5N CP2 according to the European Standard EN 197.1) has been used for all the grouts. Table 1 presents its main chemical and physical properties.

The HRWRA is a new generation modified polycarboxylate product, which allows improving and maintaining the flowability of the grout. The viscosity modifying admixture is a polysaccharide suspension in vegetable oil containing no water. Tables 2 and 3 present their main properties.

2.2. Grouts composition

Four series of cement grouts, representing 81 different mixtures have been studied. Series 1 was used to examine the effect of *w*/*c* of grouts without any admixtures; series 2 was used to study the influence of HRWRA; series 3 was used to examine the influence of VMA; and series 4 was chosen to study the combined effect of HRWRA and VMA. Table 4 presents the compositions of all the grouts.

Table 2
Properties of HRWRA.

Density at 20 °C	1.060 ± 0.020 kg/m^3
pH	6.5 ± 1.0
Dry content	20.0% ± 1.0%
Cl^-	< 0.10%
Na_2O eq.	≤ 1.5%
Recommended content	0.2% to 3.0%

Table 3
Properties of viscosity modifying agent.

Density at 20 °C	0.930 ± 0.030 kg/m^3
pH	9.5 ± 1.0
Recommended content	0.1% to 0.5%

2.3. Experimental procedures

Grout mixing procedure is an important factor affecting the rheological properties. The better the grout is mixed, the more the number and size of cement particle agglomerates decrease, the more the grout is fluid [20]. With the purpose of obtaining high dispersion of the cement particles in water, a high speed–high power propeller mixer and the following procedure were used. Water and chemical admixtures were poured in the mixer's cup, then cement was introduced into the water over a period of 2 min at a rotation velocity 1200 rpm, then the mixture (1.5 l approximately) was mixed for a total of 15 min in three stages: 6 min at 1800 rpm, 3 min at 1500 rpm and finally 6 min at 1800 rpm. Preliminary experiments showed that a longer mixing time did not improve significantly the fluidity of the grout, the Marsh cone flow time of the grout having reached a minimum constant value.

The Marsh cone was initially filled with 1 l of grout. Then the flow time of the first 0.5 l of grout flowing out a nozzle of 8 mm in diameter was measured at 18 min after the introduction of cement into water (1 min after the mixing). The density of the grouts was also measured.

The rheological behaviour of the cement grout was then characterized by a coaxial cylinders rheometer (Haake RS150) with an external fixed cylinder of radius $R_2 = 21.7$ mm and an inner rotating cylinder of radius $R_1 = 15.8$ mm and height of $H = 55$ mm. To decrease the influence of slip which occurs between the grout and the smooth surface of the inner cylinder, especially at low shear rate and

Table 4
Different series of grout mixtures.

Series 1		
w/*c*	0.42, 0.43, 0.44, 0.45, 0.46, 0.48, 0.50, 0.52, 0.55, 0.60	
Series 2		
w/*c*	HRWRA dry content	
0.45	0.00%; 0.05%; 0.10%; 0.20%; 0.30%; 0.40%; 0.50%	
0.40	0.05%; 0.10%; 0.15%; 0.20%; 0.30%; 0.40%; 0.50%	
0.35	0.20%; 0.25%; 0.30%; 0.35%; 0.40%; 0.50%	
Series 3		
w/*c*	VMA content	
0.45	0.00%; 0.05%; 0.10%; 0.15%; 0.20%; 0.25%; 0.30%	
0.50	0.00%; 0.10%; 0.20%; 0.30%; 0.40%; 0.50%	
0.55	0.00%; 0.20%; 0.40%; 0.60%; 0.80%; 1.00%	
Series 4		
w/*c*	HRWRA Dry content	VMA content
0.50	0.00%; 0.05%; 0.10%; 0.20%; 0.30%; 0.40%; 0.50%	0.50%
0.45	0.00%; 0.05%; 0.10%; 0.20%; 0.30%; 0.40%; 0.50%	0.25%
0.45	0.25%	0.00%; 0.20%; 0.40%; 0.60%; 0.80%; 1.00%
0.40	0.30%	0.00%; 0.10%; 0.20%; 0.30%; 0.40%; 0.50%
0.35	0.40%	0.00%; 0.05%; 0.10%; 0.15%; 0.20%; 0.25%

at high solid concentration [21,22], the surface of the inner cylinder was made rough by sanding. The diameter of the sanded cylinder was then measured and introduced into the measurement software of rheometer. In order to homogenize the sample, to decrease the hysteresis effect and to give a reproducible reference state to the grout with high dispersion of the cement particles in water, the grout once placed into the rheometer was pre-sheared for 3 min (30 s to increase from 0 s^{-1} to 500 s^{-1}, 120 s at 500 s^{-1}, 30 s to decrease from 500 s^{-1} to 0 s^{-1}) at 21 min after the introduction of cement into water. It was then left at rest for 1 min so that some interparticle bonds were restored. The ascending flow curve and then the descending flow curve were finally measured in steady mode: 15 stages during 5 min for each flow curve over a range of shear rate between 0.1 s^{-1} and 100 s^{-1}. In the present study, the descending flow curves were used for the determination of the rheological characteristics of the studied cement grouts (as in [13,20]). In order to obtain results as precise as possible, especially in our case where the gap between the two cylinders is relatively large, the rheological characteristics of the grouts were determined by regression of the M–Ω experimental curve which must obey the following theoretical relation (Eq. (2)):

$$\begin{cases} \Omega = 0 & \text{if} \quad R_0 \leq R_1 \\ \Omega = \int_{R_1}^{R_0} \left[\frac{M}{2\pi H r^2 K} - \frac{\tau_0}{K}\right]^{\frac{1}{n}} \frac{dr}{r} & \text{if} \quad R_1 \leq R_0 \leq R_2 \\ \Omega = \int_{R_1}^{R_2} \left[\frac{M}{2\pi H r^2 K} - \frac{\tau_0}{K}\right]^{\frac{1}{n}} \frac{dr}{r} & \text{if} \quad R_0 \geq R_2 \end{cases} \tag{2}$$

where M and Ω are respectively the torque and angular velocity of the rotating cylinder, $\tau(r) = M/(2\pi r^2 H)$ is the shear stress on the cylindrical surface of radius r ($R_1 \leq r \leq R_2$), $R_0 = \sqrt{\frac{M}{2\pi H \tau_0}}$ is the radius of the unsheared part of the grout. Theoretical values of M–Ω can therefore be computed with Eq. (2), from trial values of (τ_0,K,n), and compared to the M–Ω experimental curve. Minimizing the difference between experimental and theoretical M–Ω curves allows therefore the determination of (τ_0,K,n).

All our experimental measurements were carried out in a room at a constant temperature (22 ± 1 °C), however, it has to be noticed that the temperature of the grout during mixing and rheometer measurements was not controlled.

3. Results and discussion

3.1. Influence of the w/c ratio

Fig. 1a presents three examples of flow curves obtained with grouts containing only cement and water and Fig. 1b to d the evolution of the three rheological parameters of the Herschel–Bulkley model of all the grouts containing no chemical admixtures (series 1) as a function of the w/c ratio.

Fig. 1a shows that the rheological behaviour of these grouts is strongly shear-thinning, which has already been reported in the literature [6–8]. The fitting between the experimental results (points) and the theoretical relation (solid lines according to Eq. (2)) is very close; maximum difference is indeed always lower than 10%. The Herschel–Bulkley model therefore gives a very satisfying description of the rheological behaviour of grouts without chemical admixtures, whatever the water to cement ratio.

The exponent n obtained by regression analysis of the experimental results (Fig. 1b) varies in a narrow range (±15%) around a mean value (n = 0.431) and is hardly affected by the w/c ratio of the mixture, as if the pseudoplastic character of the mixtures is not influenced by the variation of the cement concentration.

The variation of K with the w/c ratio is sparser (Fig. 1c). Globally, K decreases with an increase in w/c, but the fluctuation of n around its mean value does not allow distinguishing a clear relation between K and w/c. We have therefore included on this figure the K values obtained by regression analysis when the exponent n is fixed at its mean value and only τ_0 and K are fitted to the experimental results. The variation of K with w/c can in that case be described satisfactorily by Eq. (3) (solid line in the figure). Taking n = 0.431, the difference between the experimental values of M–Ω and the simulated ones (with the Herschel–Bulkley model) increases a little, but this difference still remains lower than 10%.

$$K = 0.001\,\exp(20.15\,\Phi). \tag{3}$$

Φ being the solid concentration of cement in the grout ($\Phi = \rho_w c/(\rho_w c + \rho_c w)$ with ρ_w and ρ_c density of water and cement)

The variation of τ_0 with w/c (Fig. 1d) is more regular than that of K. The values obtained by regression analysis are different from those obtained when n = 0.431 (regression analysis on only the two parameters τ_0 and K) however, the two sets of points can be approximated satisfactorily by the following equation:

$$\tau_0 = 0.001\,\exp(22.75\,\Phi). \tag{4}$$

The exponential variations of K and τ_0 as a function of w/c can be explained by the increase in the number of interactions between cement grains and fluid phase and between cement grains themselves as the solid concentration of cement increases.

3.2. Influence of HRWRA content

Fig. 2a presents three examples of flow curves obtained with grouts containing HRWRA. The HRWRA content is larger than the saturation values (determined from measures of the Marsh cone flow time) for the grouts with w/c ratio equal to 0.45 and 0.40 and lower for grout with w/c ratio equal to 0.35.

At very low angular velocity (corresponding to a shear rate of about 2 s^{-1}) the measured torque presents a minimum value, which can be observed on Fig. 2a especially for low w/c values. For shear rates lower than that corresponding to the minimum torque value, the stress increases when decreasing the shear-rate. This type of flow curves has already been reported in the literature [23–26] and is generally attributed to shear localisation into a limited region near the moving surface of the inner cylinder particularly when the gap in the rheometer is important and the yield stress of the mixture is high. The shear localisation has been observed by Magnetic Resonance Imaging technique in the case of dense granular pastes [27]. In our case, this particular rheological behaviour is induced by the high HRWRA content in the mixture and is not observed for grouts without chemical admixtures. So, the hypothesis of shear localisation doesn't seem to be credible if we take into account that the yield stress of the mixtures containing high amounts of HRWRA (Fig. 2d) is lower than that of the mixtures without chemical admixture (Fig. 1d). In any way, this particular rheological behaviour for very low shear rates cannot be described properly with the Herschel–Bulkley model. Therefore, we have used in our regression, only the experimental values obtained for shear rates larger than or equal to 4 s^{-1}. This choice has been motivated by the fact that, at very low shear rate, the steady state in the rheometer is sometimes difficult to obtain in a reasonable time and also because, excluding shear rate values lower than 4 s^{-1} allows us to describe satisfactorily the rheological behaviour of the major part of the flow curve with the Herschel–Bulkley model for all the studied grouts. Indeed, the fit of series 2 with the model for shear rates larger than 4 s^{-1} is very good.

Fig. 2b to d presents the evolution of the three rheological characteristics as a function of HRWRA content of all the grouts of the

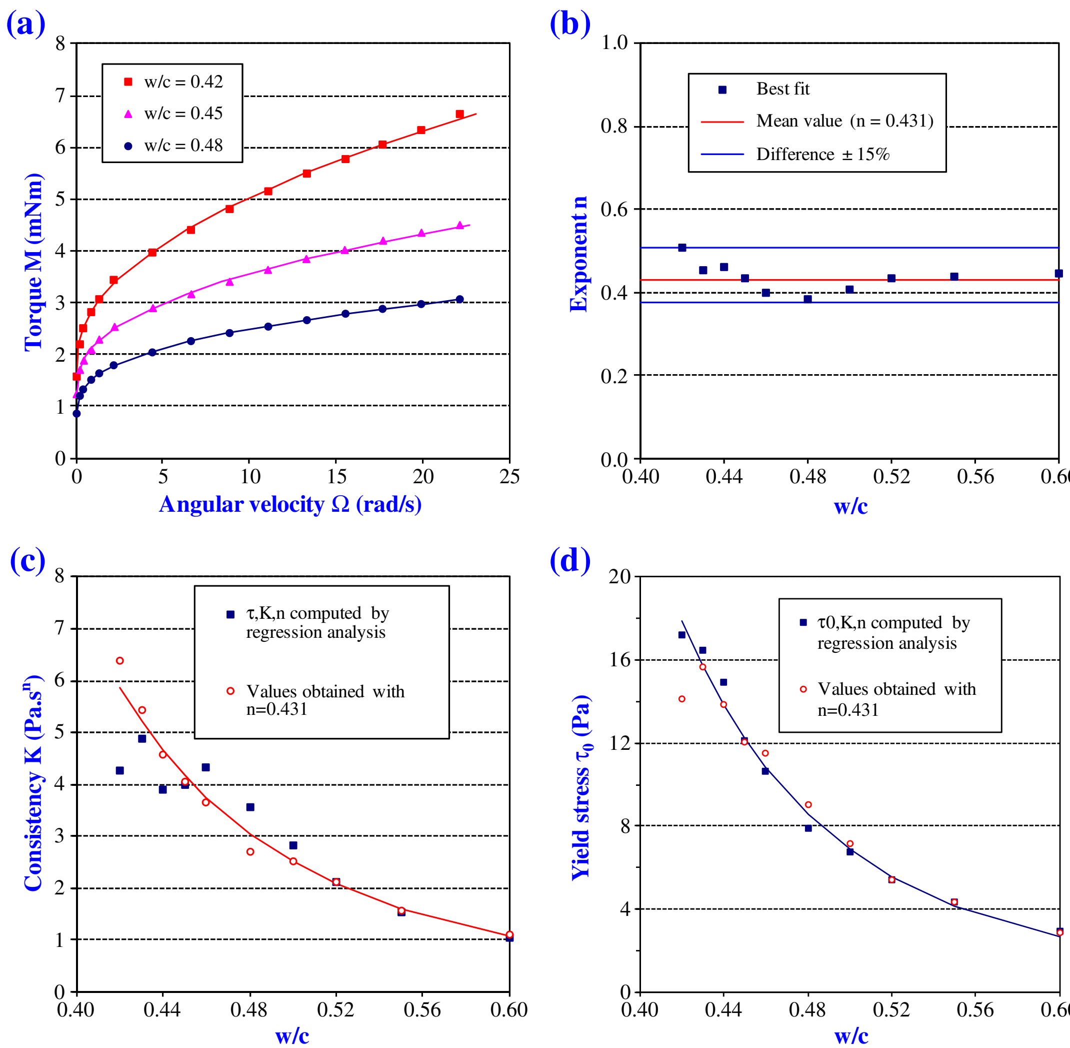

Fig. 1. Influence of w/c on the flow curves and on the rheological characteristics (τ_0, K, n) of cement grouts without chemical admixtures (series 1).

series 2. Whatever the w/c ratio, the exponent n increases with an increase in HRWRA content. This increase is very fast for low HRWRA contents and stabilizes progressively for contents larger than the HRWRA saturation value determined by the Marsh cone flow time. This result shows that the rheological behaviour of the grouts changes from a shear-thinning type to a shear-thickening type at high HRWRA content, which is in agreement with [10]. For HRWRA contents close to the saturation value, the n value is larger than but close to one, showing that the rheological behaviour of these cement grouts can be described satisfactorily with the Bingham model. These results are in good agreement with Jayasree and Gettu [28].

The consistency K and the yield stress τ_0 decrease monotonically with the HRWRA content (Fig. 2c and d); this decrease is very fast for low HRWRA contents and slows down progressively as the saturation point is reached. This result can be explained by the deflocculating of cement agglomerates, which is very efficient when a small amount of HRWRA is introduced. Deflocculating allows the release of part of the water, which is contained into flocculated agglomerates. It is worth noting that the variation of K and n are strongly correlated for these grouts. Indeed, Fig. 3 shows that K varies exponentially with n, the variation being almost parallel for the three w/c ratios studied. This exponential decrease in K is large enough to compensate the increase in n so that the flow time of the grout decreases with an increase in HRWRA content. On the other hand, the yield stress becomes almost null for large HRWRA contents for the grouts with $w/c = 0.40$ and 0.45. However, it has to be noticed that the yield stress values indicated here do not correspond exactly to the real yield stress values. As mentioned above, at very low shear rate, the rheological behaviour of these grouts cannot be properly described with the Herschel–Bulkley model. However, Fig. 2a shows that, for the grout with the larger w/c ratio, the yield stress is quasi-null, allowing us to consider as quasi-Newtonian the grouts with high w/c ratio and high HRWRA content.

3.3. Influence of VMA content

Fig. 4 presents three examples of flow curves obtained with grouts containing large contents of VMA (Fig. 4a) and the variation of the three rheological characteristics of the grouts as a function of VMA content (Fig. 4b to d).

Fig. 4a shows that, for the grouts with $w/c = 0.45$ and 0.50, the flow curve can be split into two parts. The first one, corresponding to shear rates lower than 6 s^{-1}, presents a slope significantly larger than that obtained for larger shear rates, the slope change between the two parts being abrupt. We have also observed that this phenomenon is more pronounced as the VMA content increases and as the w/c ratio decreases. Grouts containing a large VMA content have therefore a pronounced shear-thinning rheological behaviour. This behaviour can

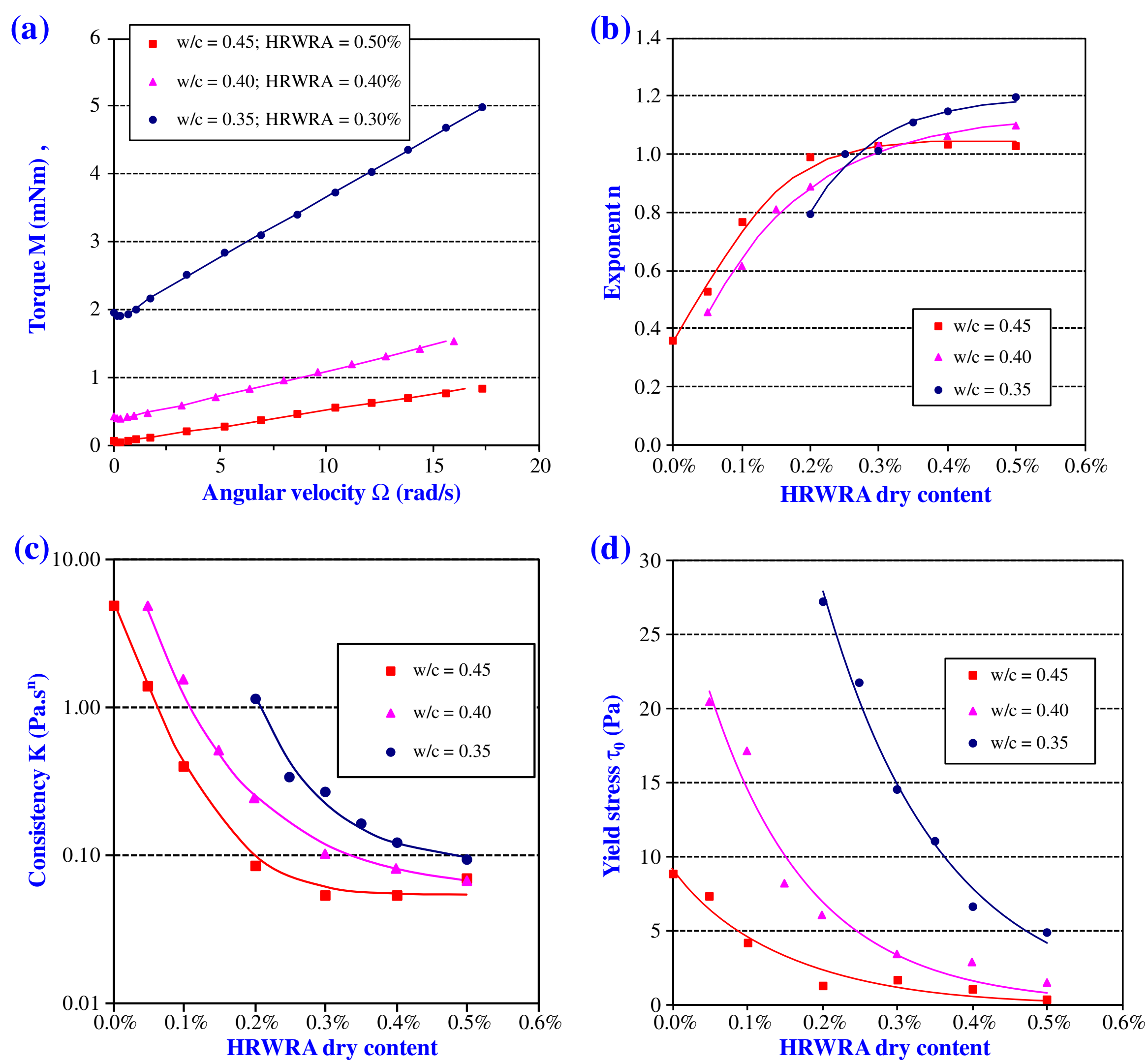

Fig. 2. Influence of HRWRA dry content on the flow curves and on the rheological characteristics (τ_0, K, n) of cement grouts containing HRWRA (series 2).

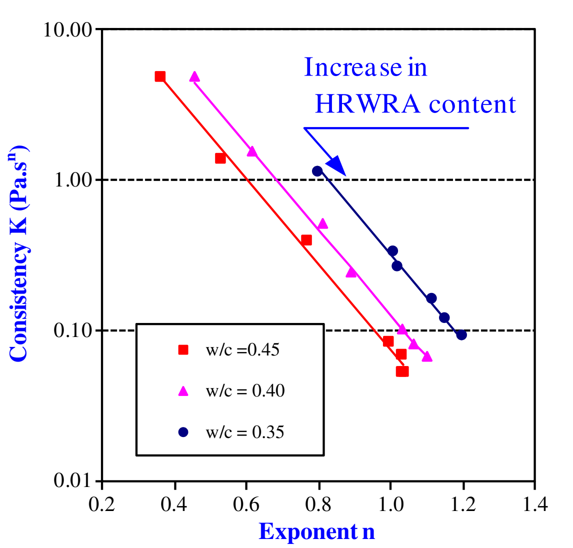

Fig. 3. Variation of K as a function of n for cements grouts containing increasing amounts of HRWRA (series 2).

be explained by the tangle of long VMA polymer molecules at low shear rate resulting in high viscosity. At larger shear rate, long molecules align along the flow direction, which decreases the grout viscosity [7,29]. This behaviour reduces static segregation and bleeding and allows in the same time obtaining a sufficient fluidity during the flow at high shear rate. However, this slope change cannot be described properly with the Herschel–Bulkley model. Once again, the measured values for shear rates comprised between 4 s^{-1} and 100 s^{-1} have been used for determining τ_0, K, n.

Fig. 4b presents the variation of exponent n as a function of VMA content for the three studied w/c ratios. n varies around a mean value equal to 0.462 ($\pm$20%) for all the studied grouts, showing that the pseudoplastic behaviour in the shear rate range 4 s^{-1} – 100 s^{-1} is almost constant. This shows that, above a shear rate equal to 4 s^{-1}, the greater part of the polymer molecules is aligned along the flow direction and does not influence anymore the shear-thinning behaviour of the grout which reaches the pseudoplastic character of the mixtures without chemical admixtures where the mean value of n was constant equal to 0.431.

Fig. 4c and d presents the variation of K and τ_0 as a function of VMA content. K and τ_0 were firstly determined by regression analysis of the flow curves (solid symbols) and secondly by fixing n to its mean value ($n = 0.462$, empty symbols). In both cases, K and τ_0 increase with an increase in VMA content even if the variation of K is sparser than that of τ_0 which seems linear with an increasing slope for low w/c ratios.

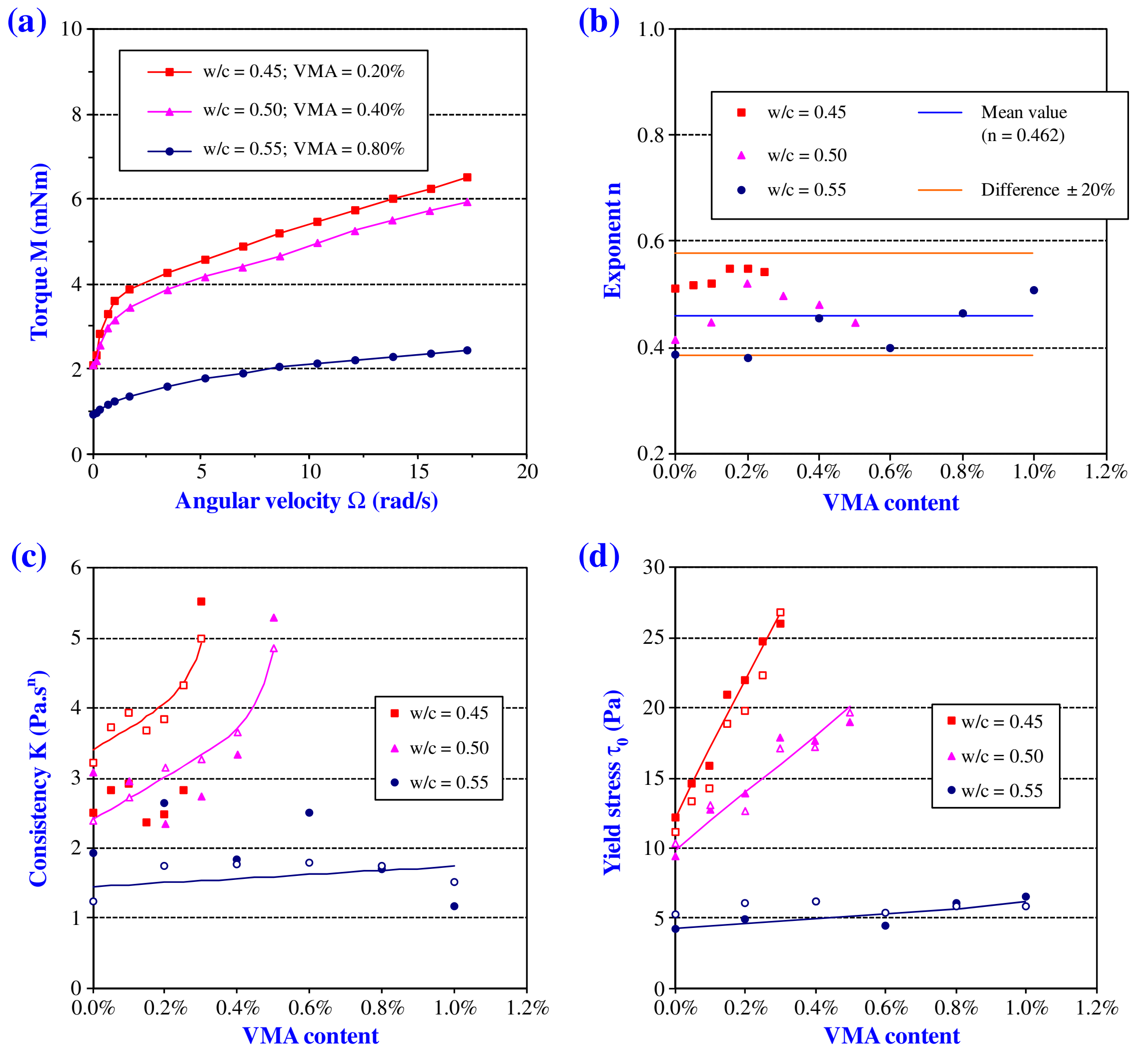

Fig. 4. Influence of VMA content on the flow curves and on the rheological characteristics (τ_0, K, n) of cement grouts containing VMA (series 3).

Note however that the values obtained for τ_0 with the Herschel–Bulkley model do not correspond exactly to the yield stress of the grouts because this model does not allow capturing the slope change of the flow curve for shear rates lower than 4 s^{-1}. In any case, the long polymer molecules of the VMA interact with each other due to attractive Van der Waals forces or to the formation of hydrogen bridges and that all the more the *w/c* ratio is low. This reduces the mobility of interstitial water molecules and increases the viscosity and the yield stress of the grouts [7,29].

3.4. Influence of HRWRA and VMA content

The flow curves obtained with cement grouts containing both HRWRA and VMA (series 4, Fig. 5a and 5b) differ generally from those obtained when only HRWRA (series 2) or only VMA (series 3) are present in the grouts. Firstly, when the grout contains a large amount of VMA, the slope change that has been observed for grouts containing only VMA (Section 3.3, Fig. 4a) does not occur anymore when HRWRA is introduced into the grout. Secondly, for the grouts containing a large amount of HRWRA, the particular rheological behaviour characterized by a minimum Torque value at low angular velocity observed for grouts containing only HRWRA (Section 3.2, Fig. 2a) is no more observed when a large content of VMA is used (Fig. 5a). However, this particular behaviour is systematically obtained for grouts with $w/c = 0.35$ containing HRWRA = 0.40%; whatever the VMA content used (Fig. 5b).

Fig. 6 presents the variations of exponent n as a function of HRWRA or VMA for the different grouts studied. It shows that, for a given VMA content, the incorporation of HRWRA increases the exponent n (Fig. 6a). This increase is however less fast than that observed for grouts containing only HRWRA. For example, the grout with $w/c = 0.45$ and containing only HRWRA (VMA = 0% on Fig. 6a) becomes rheo-thickening ($n > 1$) for a HRWRA dry content equal to 0.2% whereas the grout with $w/c = 0.45$ and VMA content = 0.25% never becomes rheo-thickening for the HRWRA content studied. The variation of n as a function of VMA content when HRWRA is fixed is more complicated and depends closely on the *w/c* ratio of the mixture (Fig. 6b). n is almost constant for the grout having a $w/c = 0.35$ with HRWRA = 0.40% whereas n decreases for the two other grouts having a higher *w/c*. This shows that the decrease in exponent n is not related to the progressive alignment of the long polymeric molecules of the VMA in the flow direction because, if it was the case, n should also decrease for low *w/c*. The decrease in n could be due to a decrease in the amount of HRWRA that can be adsorbed on the surface of cement grains due to the presence of the VMA. However, this hypothesis still has to be validated, for example in changing the order of introduction of admixtures into the grout.

Fig. 7 presents the variation of K with both HRWRA and VMA contents. The results show that for a given VMA content, K decreases with an increase in HRWRA content (Fig. 7a) this decrease being

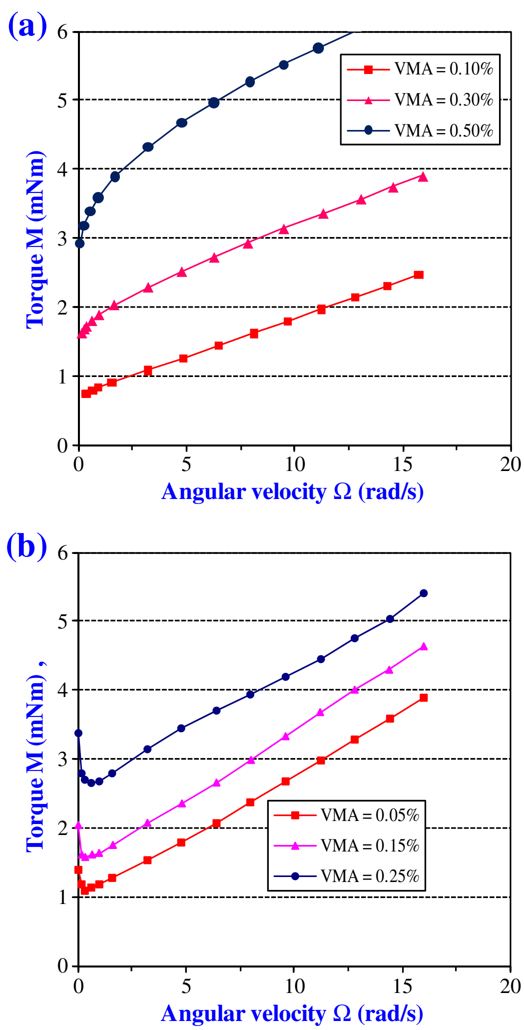

Fig. 5. Flow curves obtained with grouts containing HRWRA and varying contents of VMA (a: $w/c = 0.40$, HRWRA = 0.30%, b: $w/c = 0.35$, HRWRA = 0.40%).

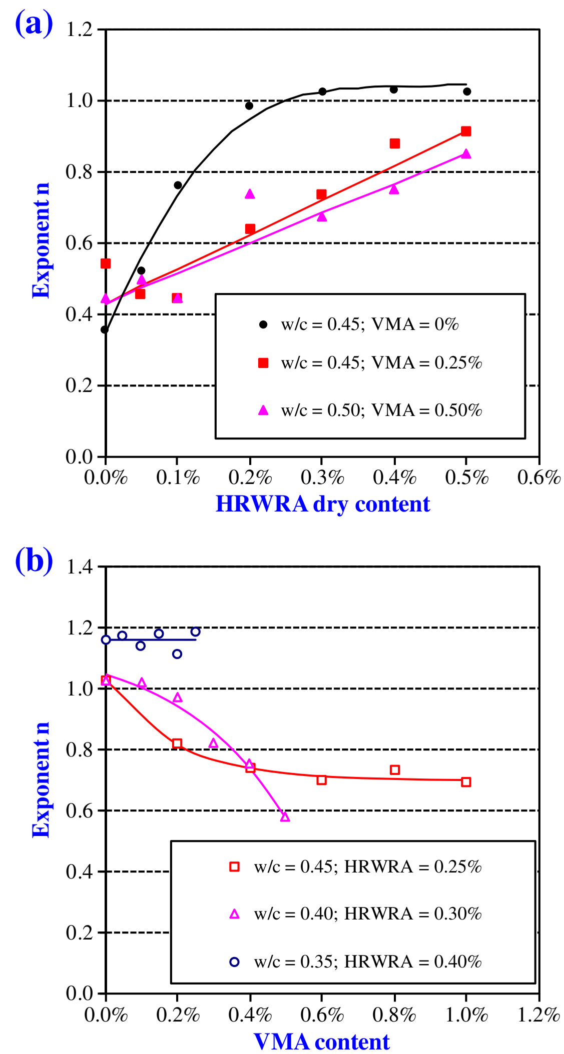

Fig. 6. Exponent n as a function of HRWRA dry content for grouts containing VMA (a) and as a function of VMA content for grouts containing HRWRA (b).

slower than that of grouts containing only HRWRA. On the other hand, for a given HRWRA content K increases with an increase in VMA content, except for $w/c = 0.35$ for which K remains approximately constant (Fig. 7b). In both cases, the variation of K is strongly correlated with that of the exponent n, as in the case of grouts containing only HRWRA (Fig. 3).

Fig. 8 presents the variation of yield stress τ_0 as a function of HRWRA dry content for constant amounts of VMA and as a function of VMA for constant amounts of HRWRA. Fig. 8a shows that the yield stress decreases more and more slowly as the HRWRA content increases, similarly to what has been observed for grouts without VMA. This is once again due to the decrease in HRWRA adsorption on cement particles surface as the saturation point is reached. On Fig. 8b, it can be observed that for a given HRWRA content, the yield stress increases more and more rapidly as the VMA content increases and as the w/c ratio decreases. For large w/c ratios, this variation is similar to what has been observed for grouts containing only VMA (Fig. 4d).

4. Prediction of Marsh cone flow time from the rheological characteristics

In a previous paper [19], a semi-analytical relation has been established between the Marsh cone flow time t and the rheological characteristics of Herschel–Bulkley fluids (τ_0, K, n) (Eq. (5)). This relation takes into account the Marsh cone geometry the mean velocity of the grout through the nozzle and introduces two correction coefficients (α and β) corresponding respectively to the loss of pressure at the entry of the nozzle and to the non-verification of Poisseuille type flow through the nozzle because of its short length.

$$t = -\int_{H_1}^{H_2} \beta \frac{(R + H_t \tan\phi)^2}{\overline{V}R^2} dH_t. \quad (5)$$

Where

$\overline{V}$ is the mean velocity at the nozzle end,
R is the radius of the nozzle
ϕ is the angle between the cone generating lines,
H_1, H_2, and H_t are the height of fluid at initial, final and current time

The Marsh cone flow time of all the cement grouts studied in this work has been determined according to Eq. (5) using the rheological characteristics determined previously for the 4 series of grouts. The aim is to verify the validity of the Herschel–Bulkley model to describe the flow of cement grouts, particularly in the case of grouts containing chemical admixtures for which the rheological behaviour at low shear rates ($<4\ s^{-1}$) diverges from the Herschel–Bulkley model. Fig. 9 presents a comparison between the measured and computed flow times of these grouts. The agreement between computed and measured

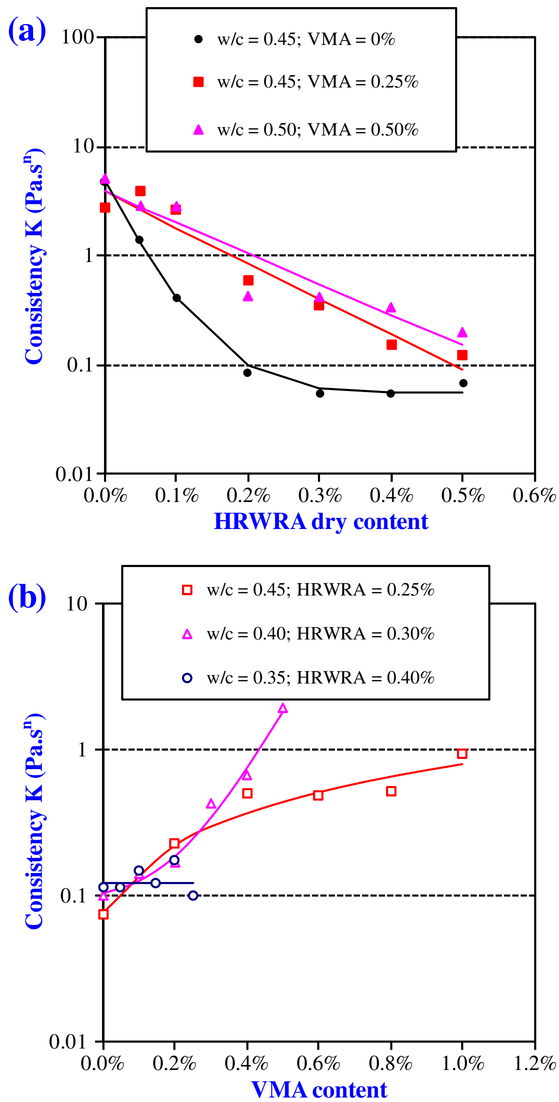

Fig. 7. Variation of K as a function of HRWRA dry content (a) for cement grouts containing VMA and as a function of VMA content (b) for cement grouts containing HRWRA.

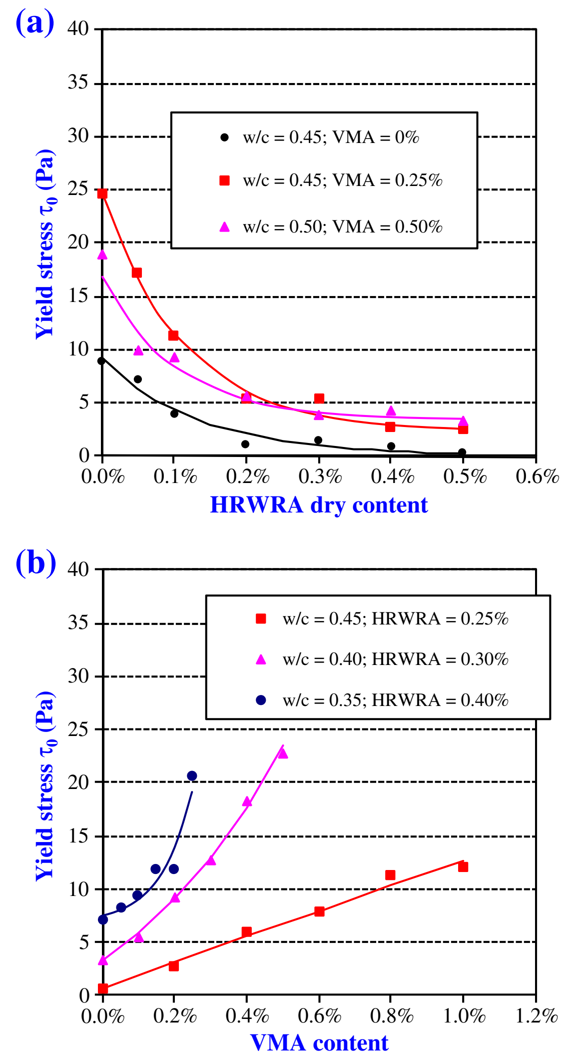

Fig. 8. variation of τ_0 as a function of HRWRA dry content (a) and of VMA content (b) for the grouts of series 4.

flow times is good, the error being always lower than 25%. This allows us to conclude firstly that Eq. (5) can be used to predict with satisfactory accuracy the Marsh cone flow time of a large variety of cement grouts, and secondly that the Herschel–Bulkey model can be used to describe the rheological behaviour of grouts despite the divergence at low shear rates concerning grouts with chemical admixtures. In fact, during the flow, high shear rates are present near the wall of the nozzle which determines the mean velocity of the grout flowing out, whereas low shear rates encountered in the central part of the nozzle have a very limited influence on it.

5. Conclusion

The results presented in this paper allow drawing the following conclusions:

1. The rheological behaviour of grouts composed of only cement and water can be simulated satisfactorily with the Herschel–Bulkley model for shear rates lower than 100 s^{-1}. This behaviour is clearly shear-thinning type, the n exponent being almost independent of w/c ($n \approx 0.431$) for all the studied grouts. Moreover, two simple equations, relying exponentially the consistency K and the yield stress τ_0 to cement concentration have been identified. For the cement and the experimental mixing procedure used in this study, the rheological behaviour of these cement grouts can be predicted on the basis of w/c only. However, one type of cement has been used in this work so it is not possible, at this stage, to extend the previous relations to other cement types.

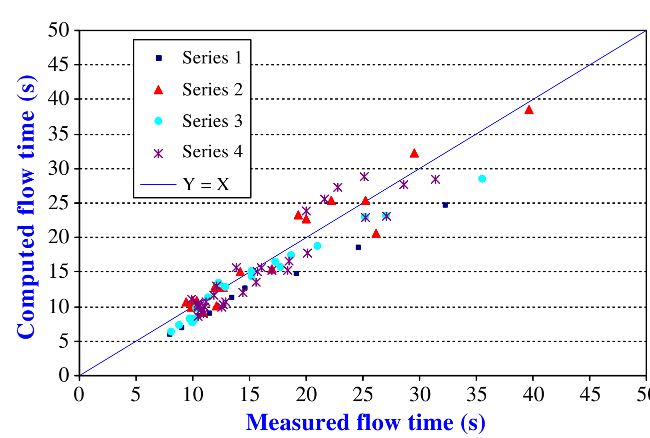

Fig. 9. Comparison between measured and computed Marsh cone flow time for the 4 studied series of cement grouts.

2. The rheological behaviour of cement grouts containing HRWRA can also be described satisfactorily with the Herschel–Bulkley model in the range of shear rate comprised between $4\ s^{-1}$ and $100\ s^{-1}$. For cement grouts containing large amounts of HRWRA, the Herschel–Bulkley model is not applicable at lower shear rates because the Torque presents a minimum value at about $\gamma = 2\ s^{-1}$. The rheological behaviour of cement grouts containing HRWRA evolves from a shear-thinning type to a shear-thickening type with an increase in the HRWRA content. This evolution is fast at low HRWRA contents and more and more slower as the HRWRA content reaches the saturation value. The yield stress decreases exponentially with the HRWRA content while the consistency K decreases exponentially with an increase in exponent n. The exponent n of cement grouts containing a high HRWRA content (close to the saturation value) is larger but close to one, showing that the behaviour of these grouts could be described satisfactorily with the Bingham model.
3. Cement grouts containing only VMA present a particular behaviour at low shear rate. The slope of M–Ω curve is larger at low shear rates ($\leq 6\ s^{-1}$) than at larger ones. This behaviour can be explained by the tangle of long VMA polymer molecules at low shear rate resulting in high viscosity. At larger shear rates, long molecules align along the flow direction, which decreases the grout viscosity [7,25]. This particular behaviour cannot be described properly with the Herschel–Bulkley model. However, as for HRWRA cement grouts, the Herschel–Bulkley model simulates correctly the rheological behaviour of these grouts in the range of shear rate considered in our study ($4\ s^{-1}$ and $100\ s^{-1}$). The exponent n is almost independent of VMA content ($n \approx 0.462$) while yield stress and consistency K increase more and more rapidly as the VMA content increases.
4. For cement grouts containing both HRWRA and VMA, the Herschel–Bulkley model cannot describe properly the whole rheological behaviour. However, in the range of shear rate comprised between $4\ s^{-1}$ and $100\ s^{-1}$, this model applies correctly and the obtained results are generally qualitatively similar to those obtained for cement grouts containing only one chemical admixture. In both cases (fixed VMA content and variable HRWRA amount or fixed HRWRA content and variable VMA amount), the consistency K decreases exponentially with exponent n increase.
5. Finally, despite the significant differences obtained at low shear rate between the experimental results and the Hershel–Bulkley model, the Marsh cone flow time of all the studied grouts can be predicted with reasonable accuracy (error lower than 25%) with a semi-analytical relation, previously established [19].

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