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Performance characteristics of cement grouts made with various combinations of high-range water reducer and cellulose-based viscosity modifier

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Abstract

Cellulose-based viscosity-modifying admixtures (VMA) are used to increase the viscosity of cement-based systems, hence, reducing the risk of material separation during handling and transport and thereafter until the onset of hardening. To ensure proper fluidity such admixtures are incorporated along with high-range water reducers (HRWRs). The ability of the VMA to ensure the required rheological properties depends on the type and interaction with the incorporated HRWR. Good understanding of such interaction is essential to ensure adequate performance. Limited knowledge is available on the effect of cellulose-based VMA and HRWR on physico-chemical characteristics and cement hydration.

The performance of grouts made with 0.40 water/cement (w/c) ratio containing a liquid-based cellulose material was investigated for mixtures made with polynaphtalene sulfonate (PNS) and polymelamine sulfonate (PMS) HRWR. The grouts are tested for fluidity, rheological properties, stability, setting and rate of hydration. The grouts were also tested for strength and pore-size distribution, and microstructural characteristics.

This paper summarizes the results of the study regarding the influence of the type and dosage of HRWR on key characteristics of grouts made with the cellulose-based VMA.

Keywords: Cement; High-range water reducers; Hydration; Rheology; Stability; Viscosity modifier

1. Introduction

Grout mixtures used for anchorage sealing, injection grouting, and for filling post-tensioning ducts are proportioned to be highly flowable to facilitate injectability and penetrability. Such grouts should be stable to minimize the risk of material separation and secure homogenous hardened properties and adequate durability. Viscosity-modifying admixtures (VMAs) are often incorporated with high-range water reducers (HRWRs) to enhance fluidity. The combinations of viscosity agent and HRWR result in highly flowable yet cohesive grouts that can flow into place with minimum risk of material separation, hence developing adequate performance of the hardened grout [1–14].

Cellulose-based VMA, such as hydropropyl methylcellulose (HPMC) is a high molar-mass polymer that fixes some of the water molecules to the periphery of chains and thereby increasing the viscosity of the solution. Adjacent polymer chains can also develop attractive bonds among one another blocking the motion of water and increasing further the apparent viscosity, especially at low rates of shear. Mixing energy can easily destroy such bond, and the polymer chains can be aligned in the direction of flow, which results in a reduction in viscosity, hence leading to a shear-thinning or pseudoplastic behavior [2].

Limited information is available about the interaction between cellulose-based VMA, such as HPMC, and various types of HRWR. This study targets the evaluation of such interaction between a liquid-based cellulose VMA and two different types of HRWR; a polynaphthalene sulfonate (PNS) and a polymelamine sulfonate (PMS). An experimental program was undertaken to evaluate the effect of the concentration of a liquid-based cellulose VMA on fresh and

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Table 1
Composition of grout mixtures

Mixture	Fluidity	VMA (ml/100 kg C)	HRWR (%)
PNS-L	low	–	0.5
PNS-H	high	–	1.0
VMA + PNS-L	low	600	0.8
VMA + PNS-H	high	300	1.1
VMA + PMS-L	low	600	1.0
VMA + PMS-H	high	300	1.8
PMS-L	low	–	0.6
PMS-H	high	–	1.1

Low fluidity—minislump diameter of 120 ± 5 mm.
High fluidity—minislump diameter of 140 ± 5 mm.

hardened properties of neat cement grout. The grout was prepared with 0.40 water/cement (w/c) ratio. The dosage rates of the chemical admixtures were determined by fixing the dosage of the VMA while varying the concentration of the HRWR to maintain two levels of initial consistency measured by minislump flow. Control mixtures consisted of grouts made without any VMA. The evaluated grouts were divided into two groups. In the first group were mixtures with relatively low fluidity (Group L) where medium dosage of the liquid-based VMA was used (600 ml/100 kg of cement), and the second group of grouts had higher fluidity levels (Group H) where a relatively low concentration of VMA was used (300 ml/100 kg of cement). The dosage rates of the various HRWR were adjusted to obtain initial minislump spreads of 120 ± 5 mm and 140 ± 5 mm for the mixtures in Groups L and H fluidity, respectively.

As shown in Table 1, eight mixtures were investigated, four in each fluidity group, as follows: two grouts containing PNS and no VMA, two grouts containing PNS and VMA, two grouts containing PMS and VMA, and finally two mixtures with PMS and no VMA. A grout mixture made without any chemical admixture was prepared to evaluate some key properties. The grouts were tested to evaluate fluidity, rheological properties, stability, cement hydration, as well as the development in compressive strength, pore-size distribution, and microstructure.

2. Materials and test methods

A Type 10 CSA-CAN A5 cement was used. The chemical and physical characteristics of the cement are given in Table 2. A liquid-based cellulose VMA was used in this study. The dosage rates of the VMA were set to 300 and 600 ml/100 kg of cement which correspond to relatively low to medium dosages necessary to control bleeding and washout loss. A polynaphthalene sulfonate HRWR (PNS) containing 42% solids mass with 1.21 specific gravity was used. A polymelamine sulfonate HRWR (PMS) containing 40% of solids and 1.2 specific gravity was also employed.

All mixtures were prepared in 4-l batches mixed using a high shear mixer rotating at 3000 rpm. The mixing water was cooled to 12 °C to compensate for heat generation resulting from the mixing action. The water and admixtures were first added and followed by the cement that was introduced into the bowl of the mixer rotating at low speed over 1 min. The grout was then mixed for 2 min.

Temperatures of freshly mixed grouts ranged from 20 to 23 °C. Grouts were tested for consistency using the minislump and Marsh cone 5 min after the initial contact of cement with water. Rheological properties were then evaluated followed by the forced bleeding and washout testing. The loss of consistency was also determined by evaluating the minislump and 60 min Marsh cone flow time. Between 5 and 60 min, the grout was poured into a sealed container to prevent evaporation. The grout remained undisturbed, except for manual mixing for 30 s, prior to the 60-min testing to ensure homogeneous suspension. The same batches were used to determine setting time and to cast samples for porosity testing. Identical batches were prepared for measuring heat flux and compressive strength development.

The minislump test involves measuring of the spread of grout poured in a special cone resting on a flat Plexiglas plate. The minislump cone has an upper diameter of 19 mm, a lower diameter of 38 mm and a height of 57 mm, all of which are proportional to the concrete slump cone [7]. A modified Marsh cone with a capacity of 1.2 l and an orifice diameter of 4.56 mm was used. The time needed to fill a graduated cylinder with grout at 100-ml intervals was noted. A flow time for 700 ml is reported and was on the order of 20 s for water at 20 °C. Unlike the minislump, the shear rate involved in the Marsh cone test was higher and closer to rates encountered during mixing, pumping, and grout injection.

Table 2
Chemical and physical properties of cement

Chemical composition	Mass (%)
SiO_2	21.1
Al_2O_3	4.4
Fe_2O_3	2.6
CaO	62.0
MgO	2.78
SO_3	2.71
K_2O	0.68
Na_2O	0.25
Na_2O eq.	0.70
LOI	2.72
Bogue composition	**Mass (%)**
C_3S	51
C_2S	22
C_3A	7
C_4AF	8
Physical properties	
Blaine fineness	350 cm^2/g
Vicat initial setting time	150 min
Vicat final setting time	260 min
3-Day compressive strength	17 MPa
28-Day compressive strength	36 MPa

Rheological properties were evaluated using a coaxial cylinder viscometer. Viscosity was measured at 11 rotational speeds varying between 1 and 600 rpm, corresponding to 1.7 and 1020 s^{-1}. The shear stress was noted after 20 s of rotation at each speed. The apparent viscosity is expressed as the ratio between the shear stress (τ) and the shear rate (γ) at any given shear rate. The yield value and plastic viscosity are derived by regression analysis of the shear stress–shear rate data obtained on the ascending curve, by assuming a polynomial response and suppressing the second order value which is insignificant. The polynomial law ensures a better fit than the traditional linear response [8].

The washout resistance was determined by pouring 500 ml of grout into a beaker containing an equivalent volume of water. Grout flows from a funnel positioned at a given height above a beaker filled with water. The grout displaces the water along with washed out cement. The washout loss is determined by calculating the difference in mass of the grout sample before and after dropping it in water, expressed as a percentage of initial mass [8].

The resistance of the grout to forced bleeding (also referred to as pressure loss) was determined using a Baroid filter. The test consists in measuring the volume of water infiltrated through fine filters under sustained a pressure of 0.55 MPa. The filters are capable of retaining 99.7% of solid particles with diameters exceeding 0.3 μm. The bleed water was monitored over 10 min and is expressed as a percentage of the mix water.

The setting time was measured using the Vicat needle test (ASTM C 953). A semiadiabatic calorimetric analysis was used to evaluate the influence of the various admixtures on the kinetics of cement hydration in the first 24 h of hydration. Approximately 120 g of grout was poured into a plastic bottle placed in a Dewar vase filled with water at 20 °C. The Dewar vase was placed into a jacket filled with water whose temperature was varied to match that of the grout over the 24-h test period. The system was calibrated prior to carrying out each test to ensure that heat exchange between the vase and surrounding environment was limited to 0.25 °C. The temperature of the grout was measured at 5-min intervals using a probe at the center of the grout. Electrical conductivity was measured with a commercial conductimeter using a diluted sample of 2.0 w/c.

Mercury intrusion porosimetry was used to determine pore-size distribution at 28 and 91 days. Core samples measuring 20 mm in diameter and 50 mm in length were used. The grout samples were water-cured until the time of testing. The cores were immersed in acetone for 6 h and oven dried at 60 °C. The mercury intrusion porosimetry apparatus permits the gradual increase in pressure to 150 MPa, enabling the intrusion of pores with apparent pore diameters of 5 nm to 5 μm.

Compressive strength was evaluated using 50-mm mortar cubes. The mortar samples were prepared using grout mixture evaluated above by mixing the grout with standard Ottawa sand (ASTM C 109) to obtain a sand to cement ratio of 2.75 by mass. Mortar samples were tested instead of grout samples to reduce variations in strength that are often encountered when testing grout samples. The cubes were demolded after 24 h and stored in lime-saturated water at 20 ± 2 °C until testing at 7 and 28 days. Grout samples were prepared to conduct scanning electron microscopy. The microscope was also equipped with an energy dispersive X-ray spectrometer (EDXS). Freshly fractured grout surfaces were observed at 7 and 28, and 91 days.

3. Test results and discussion

3.1. Minislump and flow time consistency

The results of the minislump consistency of the grouts at 5 min are shown in Table 3. The grouts had the targeted minislump spreads of 120 ± 5 or 140 ± 5 mm.

As expected, the incorporation of VMA increased the HRWR demand by 40% for two grouts made with PMS HRWR and the mixture with PNS with low fluidity (L Group). For the mixture prepared with PNS and VMA, in Group H (high fluidity), the demand in PNS increased by 10% only, as indicated in Table 1. This increase of HRWR demand is due to the action of VMA, which take water molecules into its structure and we need add more HRWR to achieve the same fluidity.

The mixture with the lower fluidity level prepared with PNS and no VMA exhibited limited loss in minislump (109 mm after 60 min). This loss is lower than that obtained for the grout prepared with the non-VMA grout and PMS HRWR (minislump of 77 mm after 60 min; Table 3) showing a better dispersant efficiency of the PNS HRWR compared to that of the PMS HRWR. A similar trend was observed for the grout mixtures containing medium dosages of the VMA (600 ml/100 kg of cement). The VMA mixture with low fluidity made with PNS HRWR had a minislump spread of 115 mm at 60 min, while that containing the PMS HRWR had a minislump value of 107 mm at 60 min.

The Marsh cone flow time values were quite different within each fluidity group determined from the minislump consistency. As summarized in Table 3, the flow time values were much greater for the two grouts prepared with VMA-PMS compared to the other tested mixtures. At 60 min, the Marsh cone flow time of the low fluidity grout containing PNS HRWR did not change considerably compared to 5 min (58 to 64 s). The addition of VMA at medium dosage (600 ml/100 kg of cement) did not change this trend, the flow time increased from 47 to 55 s. For the higher fluidity mixtures prepared with PMS HRWR, an opposite trend was observed; even for the non-VMA grout which presented an increase in flow time from 39 to 71 s. The incorporation of VMA at 300 ml/100 kg of cement increased the flow time from 132 s at 5 min to 225 s at 60 min. This again indicates the higher dispersion efficiency of the PNS HRWR compared to that of the PMS HRWR.

Table 3
Characteristics of tested grout mixtures

Fluidity		PNS		VMA + PNS		VMA + PMS		PMS	
		low	high	low	high	low	high	low	high
SP (% C)		0.5	1.0	0.8	1.1	1.2	1.8	0.6	1.1
VMA (ml/100 kg C)		–	–	600	300	600	300	–	–
Density		1.99	1.97	1.96	1.92	1.96	1.98	2.00	1.99
Temperature (°C)		20.3	16.2	23.1	20.4	23.5	22.6	22.2	22.5
Minislump (mm)	5 min	120	145	124	137	121	140	125	144
	60 min	109	–	115	–	107	114	77	125
Flow time (s)	5 min	57.5	39	47	44	225	132	44	39
	60 min	64	–	55	–	–	225	–	71
Yield value at 5 min (Pa)		2.5	0.7	1.8	1.4	24.9	10.0	14.4	1.6
Plastic viscosity at 5 min (Pa·s)		0.07	0.06	0.07	0.08	0.16	0.15	0.09	0.07
Apparent viscosity (Pa·s)	5.1 s^{-1}	0.60	0.20	0.40	0.40	2.00	1.00	1.90	0.30
	510 s^{-1}	0.08	0.06	0.08	0.07	0.30	0.22	0.14	0.08
Forced bleeding (ml)		35	7	19.5	10	4	6	34	18
Washout loss (%)		12.3	13.1	11.7	11.2	7.5	11.1	10.9	10.7
Setting time (h)	initial	7.3	11.25	8.50	10.65	11.70	12.15	7.10	9.3
	final	8.75	12.90	10.00	12.25	13.25	14.00	8.50	10.75
Compressive strength (MPa)	7 days	31	27	26	26	29	28	31	32
	28 days	36	34	32	31	38	34	40	41
Total pore volume (mm^3/g)	28 days	152	161	166	162	144	140	144	137
	91 days	136	134	157	146	143	143	127	120
Large pore volume (mm^3/g)	28 days	120	90	110	110	80	80	80	90
	91 days	90	60	100	100	100	100	70	70

3.2. Rheological properties

The grouts with 120 ± 5 mm initial minislump spreads had higher apparent viscosities at both low and high shear rates than the higher fluidity grouts (Table 3). The two grouts prepared with PNS and no VMA had lower apparent viscosity values than corresponding non-VMA grouts prepared with PMS. The increase in VMA dosage in grouts containing PNS HRWR did not considerably change the apparent viscosity at low and high shear rates in the two fluidity groups. In the case of grouts prepared with PMS, an opposite trend was observed. In the H Group, the addition of VMA at low dosages of 300 ml/100 kg of cement increased the apparent viscosity from 0.3 to 1.0 Pa·s at the low shear rate of 5.1 s^{-1}, and from 0.08 to 0.22 Pa·s at high shear rate of 510 s^{-1}. For the low fluidity grouts, the apparent viscosity at low shear rate was nearly the same to grouts made with 600 ml/100 kg of cement. At high shear rates, the viscosity increased from 0.14 to 0.3 Pa·s.

The increase in VMA dosage led to increase in both yield value and plastic viscosity at low and high shear rates. The yield value is related to the minimal shear stress needed to overcome internal resistance to flow. Grouts prepared with PMS exhibited higher yield value and plastic viscosity values than those prepared with PNS even those made without any VMA, which showed higher thixotropic effect of the PMS HRWR that those of the PNS HRWR. The use of cellulose-based VMA in the grouts with PMS also results in significant increase of rheological parameters that in grouts made with PNS. For example, in the high-fluidity grouts, the incorporation of 300 ml/100 kg of cement of VMA increased the yield value from 1.6 to 10 Pa for the grouts made with PMS, and from 0.7 to 1.4 Pa for those made with PNS. For the same grouts, the plastic viscosity increased from 0.07 to 0.15 Pa·s for the PMS HRWR mixtures and from 0.06 to 0.08 Pa·s for the PNS HRWR grouts. In the case of grouts of the relatively low fluidity, the trend was the same. The yield value and plastic viscosity values were similar in the case of the non-VMA grouts and those made with 600 ml/100 kg of cement of VMA for mixtures made with PNS HRWR.

A similar study carried out to evaluate the interactions of polysaccharide-based welan gum VMA and PNS HRWR in grouts made with 0.40 w/c [6]. The cement and PNS HRWR were the same as those described below for this study. The increase in the dosage of welan gum was showed to result in a proportional increase in yield value and plastic viscosity. For a mixture prepared with 0.025% of welan gum by mass of cement and 1.6% of PNS the yield value was 4 Pa and the plastic viscosity was 0.08 Pa·s. These values increased to 9 Pa and 0.11 Pa·s, respectively, when the VMA content increased to 0.055%. These values were 16 Pa and 0.14 Pa·s at 0.075% VMA content.

3.3. Stability

The results of forced bleeding are presented in Table 3. All grouts made with VMA exhibited significantly lower bleeding compared to those with no VMA. For example, in the case of the grouts prepared with PMS, the addition of VMA at 300 ml/100 kg of cement decreased the forced bleeding by 5%. The increase of VMA dosage to 600 ml/100 kg of cement led to 18% reduction. In the case of mixtures made with PNS, the incorporation of 300 and 600

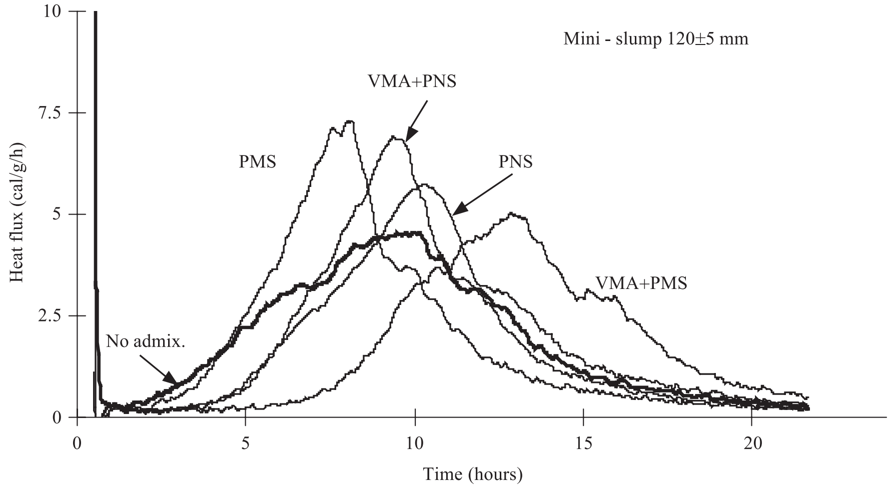

Fig. 1. Heat flux of low fluidity grouts.

ml/100 kg of cement of VMA resulted in 0 and 9% reduction in bleeding, respectively.

It is interesting to note that the grouts having minislump consistency of 140 ± 5 mm had less forced bleeding than those with 120 ± 5 mm minislump consistency. This is due to the greater dispersion of the HRWR on cement particles that can result in greater packing of the cement particles against the draining surface of the compacted cake. On the other side, the adsorption of water molecules onto VMA polymer chains decreases the amount of free water available for bleeding.

For the low fluidity grouts containing PMS, the use of VMA at medium dosage is shown to decrease washout loss from 10.9% to 7.5%. In the case of study carried out with welan gum VMA [6] a similar trend was obtained where the increase in VMA dosage led to higher resistance to forced bleeding and washout.

The improved washout resistance of those grouts is due partially to greater degree of water retention of the VMA polymers by hydrogen bond. Some of the polymers can also be adsorbed on reactive site of the cement particles. This forms a polymer network, and cement particles are suspended in this network and protected form dilution when the grout is poured in water. On the other hand, the incorporation of the same content of cellulose-based VMA in grouts made with PNS HRWR did not have the same effect. This demonstrates the importance of proper selection of efficient type and content of VMA and HRWR in these highly flowable and stable cement-based systems.

3.4. Setting time

The results of the initial and final setting time are presented in Table 3. The reference non-VMA mixtures containing lower contents of HRWR present shorter initial setting times than the VMA grouts. In the low fluidity grouts, the initial setting time was 7.3 h for the grout made with PNS HRWR and 7.1 h for that made with PMS

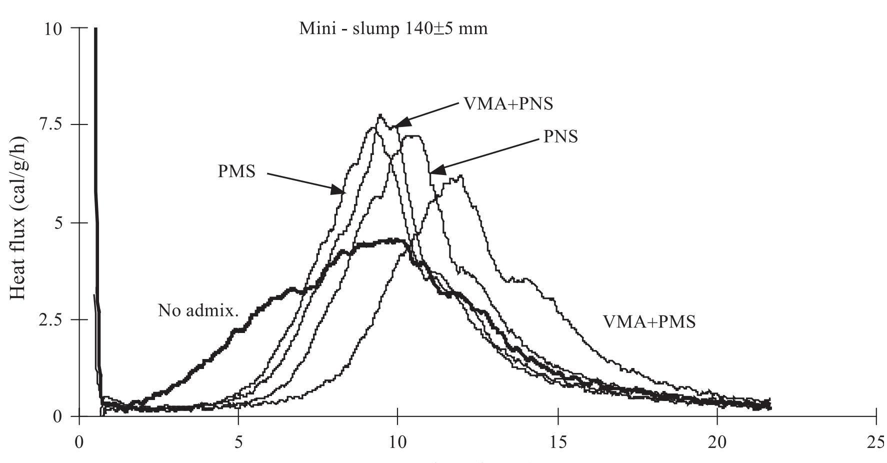

Fig. 2. Heat flux of high-fluidity grouts.

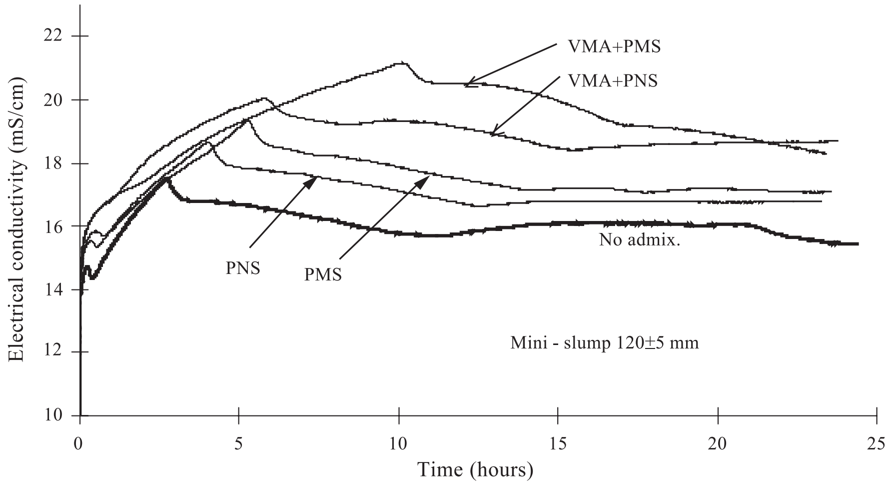

Fig. 3. Electrical conductivity of low fluidity grouts.

HRWR. In the high-fluidity group where higher dosage rates of the HRWR was incorporated, the initial setting time for the non-VMA grouts containing PNS and PMS HRWR were 11.3 and 9.3 h, respectively. Grout mixtures containing VMA necessitated greater HRWR demand, thus resulting in longer delay in setting.

Among the grouts containing VMA, the longest initial setting was obtained with the grouts made with PMS (11.7 and 12.2 h for mixtures with low and high fluidity, respectively).

In comparison, the grout containing 600 ml/100 kg of cement of VMA and 1.0% of PMS to that containing 1.1% PMS and no VMA, it can be noted that the initial setting times are slightly different (11.7 and 9.3 h, respectively), despite the similar dosage of HRWR. Therefore, there is also a retarding effect of the cellulose VMA when combined with the PMS HRWR. In the case of the PNS HRWR, the opposite trend was observed. The grout containing 300 ml/100 kg of cement of VMA and 1.1% of PNS HRWR had a shorter initial setting time than the non-VMA grout made with 1.0% PNS HRWR (10.7 vs. 11.3 h). Therefore, the setting time depends on the dosage of VMA, as well as the dosage and type of HRWR. The retarding effect of VMA on setting time can be related to the adsorption of VMA polymer chains onto cement particles and the interaction between the polymer and cement hydration products, which can cause a retarding effect on the hydration kinetics.

For the final setting time, the similar tendencies were obtained. The reference grouts had shorter final setting times than the grouts containing VMA and greater content of HRWR. Differences between final setting times are smaller in high-fluidity grouts than in the low fluidity grouts, ranging from 10.8 h (grouts containing the PMS HRWR only) to 14 h (VMA grouts containing the PMS HRWR). In the high-fluidity grouts, the longest setting time was obtained with the grout made with the highest content of HRWR (VMA grout with 1.8% PMS HRWR).

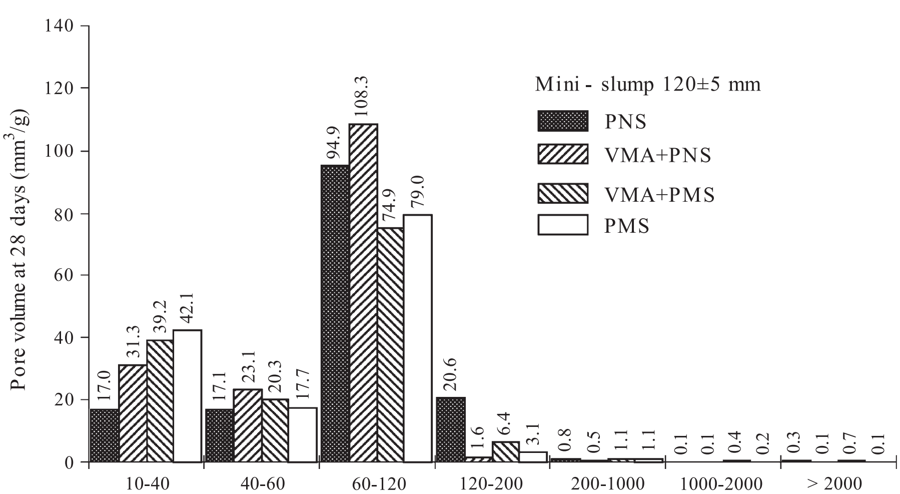

Fig. 4. Pore distribution of low fluidity grouts at 28 days.

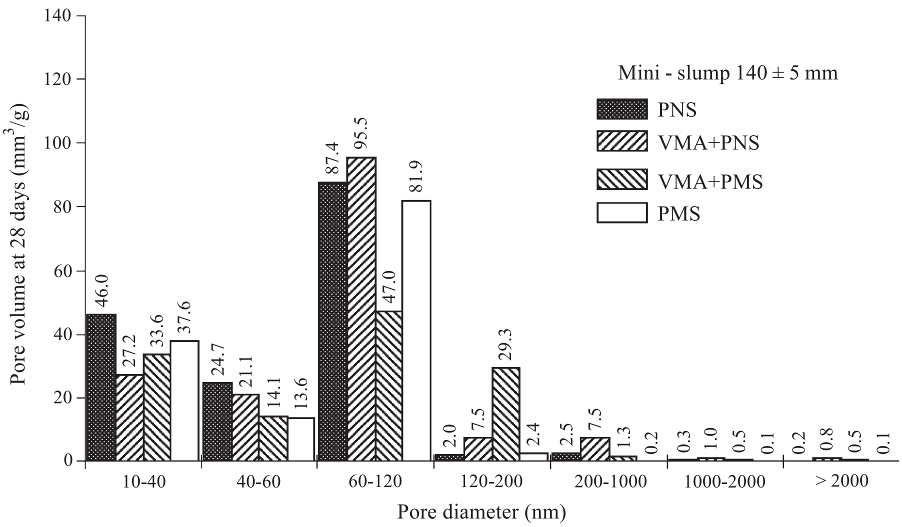

Fig. 5. Pore distribution of high-fluidity grouts at 28 days.

3.5. Adiabatic calorimetry

The heat flux curves illustrated in Figs. 1 and 2 show the differences existing between the lengths of the dormant periods and the intensities of hydration during the first 24 h. The control grout made without any admixture had the lowest rate of increase in heat flux and the shortest dormant period. The differences are more significant in the low fluidity grouts than in the higher fluidity ones.

The retarding effect of the HRWR is more pronounced in non-VMA grouts made with PNS HRWR than those with PMS HRWR. The two non-VMA grouts containing the PMS presented the shortest dormant periods. The VMA grouts containing PNS exhibited shorter dormant periods than non-VMA grouts made with only PNS. A similar accelerating effect of the VMA-PNS combination was noted from the measurement of the setting time. On the other hand, the two VMA grouts containing PMS HRWR had the longest dormant periods and the lowest heat flux values for both fluidity groups. These two mixtures also presented the slowest ascent of flux curves. This could correspond to slower degree of portlandite precipitation at the beginning of the acceleration period [15].

The slow precipitation of portlandite in the grouts could be caused by the incorporation of VMA that can interfere with the dissolution of the anhydrous phases of cement, the formation of nuclei, and growth of already formed crystals.

3.6. Electrical conductivity

The results of electrical conductivity tests of the low fluidity grouts are presented in Fig. 3. After the first contact

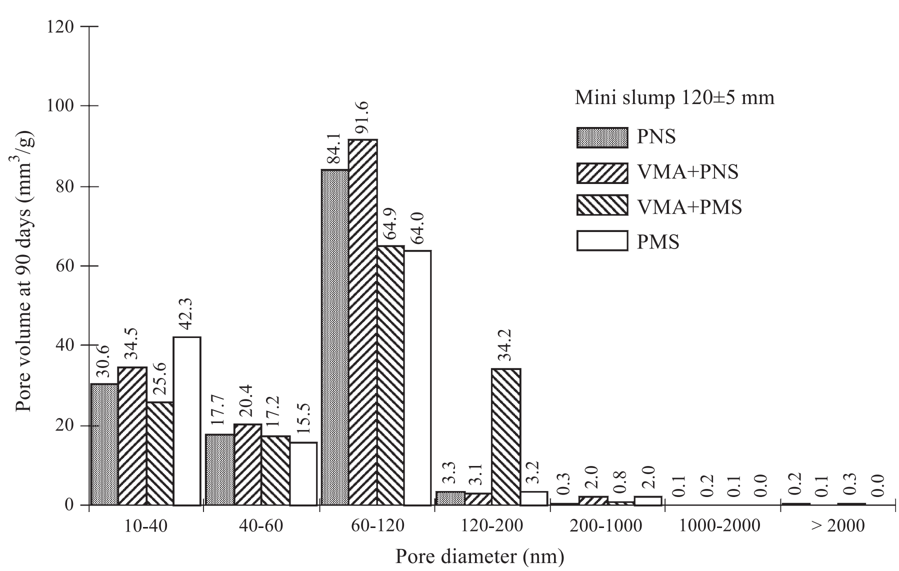

Fig. 6. Pore distribution of low fluidity grouts at 91 days.

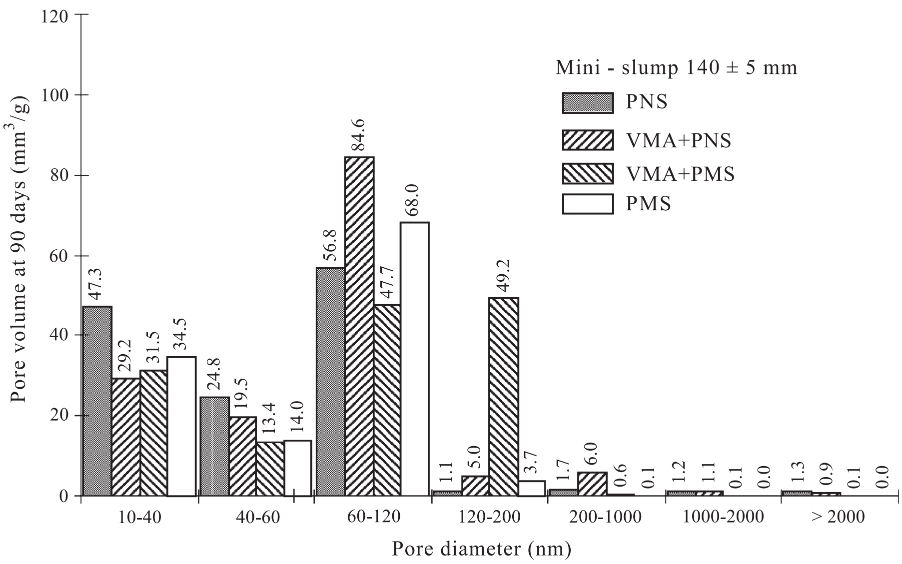

Fig. 7. Pore distribution of high-fluidity grouts at 91 days.

between water and cement, the electrical conductivity increases until a peak value that represents the beginning of portlandite precipitation. Similar results were obtained for grouts with the higher fluidity level containing the lower VMA dosages. The grout made without any chemical admixture had the lowest ionic activity in the solution. This was followed by the non-VMA grouts containing HRWR. The two non-VMA grouts with higher fluidity exhibited greater (not shown here) electrical conductivities than those with the lower fluidity. This may be due to the better dispersion of cement particle, which permits better dissolution of the cement grains and higher ionic activity in the solution.

The two grouts made with VMA containing PMS HRWR presented the most delayed points of portlandite precipitation, which confirms the results obtained from adiabatic calorimetry. These two grouts maintained the highest electrical conductivity after the beginning of portlandite precipitation. The ionic activity in the solution was high, but precipitation was very slow; the slope after the peak was very low, almost inexistent. The presence of the network of the VMA polymers could be related to the slow degree of precipitation.

3.7. Mercury porosity

The total pore volume and volume of large pores are presented in Table 3. The pore-size distribution of the grouts with low and high fluidity levels are plotted in Figs. 4–7. At 28 and 91 days of age, the grouts containing the non-VMA mixtures with the PNS HRWR had higher total porosity than those with the PMS HRWR. The two grouts containing PNS and no VMA had similar cumulative pore volumes at 28 and 91 days. However, lower fluidity grout contained higher volume of large pores with apparent diameters greater than 60 nm (120 mm^3/g) compared to the higher fluidity mixtures (90 mm^3/g). In the grouts containing PMS HRWR prepared without any VMA, the differences between total volume of large pores and pore distribution are not so pronounced.

In comparing the non-VMA with the corresponding VMA grouts, the total pore volume at 28 days of the VMA grouts was similar to the reference grouts. Only the low fluidity grout containing VMA and PNS was more porous than the corresponding reference grout. At 91 days, all of the VMA grouts exhibited greater total porosity than the corresponding grouts made without any VMA.

At 28 and 91 days of hydration, the VMA grouts made with PMS had greater volumes of capillary pores with apparent diameters between 120 and 200 μm compared to the grouts made with PMS and no VMA. The same grouts exhibited similar or lower concentration of pores with apparent diameters of 60 to 90 μm than the corresponding

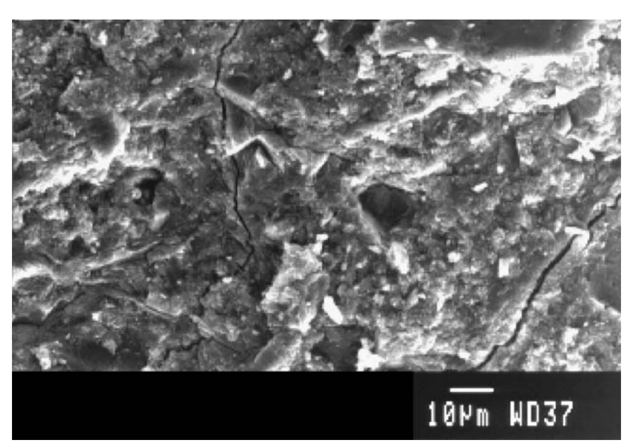

Fig. 8. Microstructure of cement paste prepared with PMS containing no VMA at 7 days.

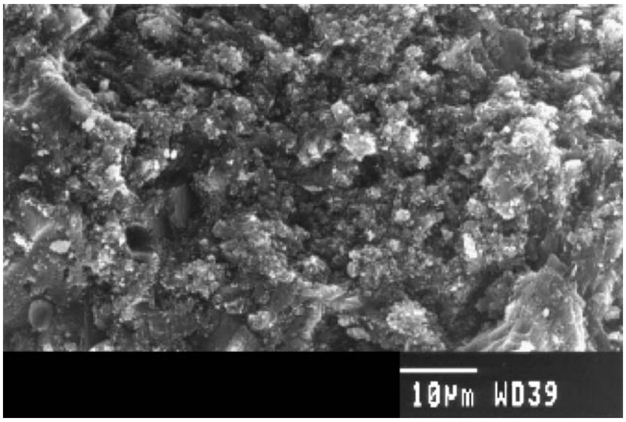

Fig. 9. Microstructure of cement paste prepared with PMS containing VMA at 7 days.

grouts containing only PMS. The VMA mixtures containing PNS had higher pore volumes ranging between 60 and 120 μm compared to the reference non-VMA grouts made with PNS HRWR.

3.8. Compressive strength

As presented in Table 3, the compressive strength values varied between 26 and 31 MPa at 7 days and 27 and 38 MPa at 28 days. The compressive strength of mortar mixtures having the same w/c depends mainly on the pore volume, pore-size distribution, and degree of hydration. It is important to underline that, in our study, the porosity was tested on cement paste, and the compressive strength was tested on mortar samples. The highest compressive strengths at 7 and 28 days were obtained with the reference mixtures containing PMS HRWR, for both fluidity groups. These strength values were 31 and 32 MPa for the mixtures with the relatively low and high fluidity levels, respectively, at 7 days and 40 and 41 MPa, respectively at 28 days. These grouts had the lowest total pore volumes and low volume of large pores at 28 days that have marked influence on strength.

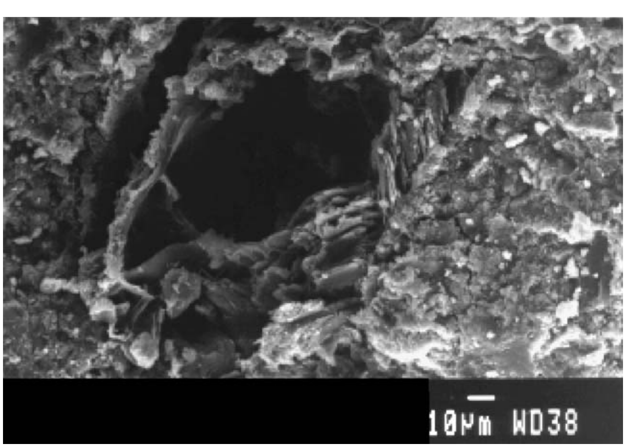

Fig. 10. Organic gel in cement paste containing VMA and PNS at 7 days.

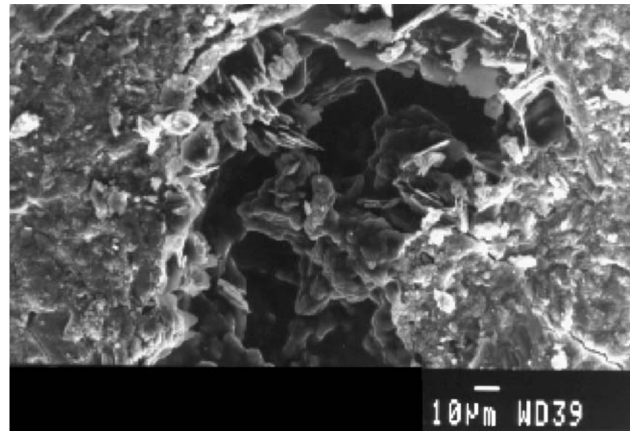

Fig. 11. Organic gel in cement paste containing VMA and PNS at 91 days.

The VMA mixtures had 6 to 16% lower strengths than the corresponding non-VMA mortars. The two VMA mortars containing PNS HRWR developed the lowest compressive strengths. Corresponding grout mixtures had the highest pore volumes and considerably greater volume of large pores at 28 days, which has adverse effect on strength.

The mortars containing the cellulose-based VMA and PMS HRWR had higher compressive strengths than mixtures with VMA and PNS HRWR. At the same time, in the respective cement pastes, the volume of large pores was lower in the paste made with VMA and PMS HRWR.

3.9. Observations of cement paste using scanning electron microscope (SEM)

At 7 days, the no VMA grouts were observed to have dense structure with compact Type III C-S-H gel, indicating more advanced hydration (Fig. 8). At same age, the structure of the pastes containing VMA was rather heterogeneous; some of the C-S-H gel was of Type III and in other cases in the form of fibrous network, indicating less advanced hydration (Fig. 9).

In large capillary pores of the VMA grouts containing PNS, organic gel was observed at 7, 28, and 91 days (Figs. 10 and 11). This may be the consequence of an interaction of the two admixtures or the formation of interaction product between the admixtures and cement hydration products.

4. Conclusions

Based on the above results of mixtures prepared with 0.40 w/c and liquid-based cellulose VMA and two HRWR types, the following conclusions can be drawn:

1. For the same consistency, the incorporation of VMA was shown to increase the HRWR demand between 10 and 40% with either PNS- or PMS-based HRWR.
2. The slump loss at 60 min was higher for grouts prepared with PMS-based HRWR than those with PNS

HRWR. The increase in VMA dosage was shown to increase the yield stress and plastic viscosity. Mixtures prepared with PMS-based HRWR exhibited higher yield value and viscosity that those prepared with PNS-based HRWR.

3. Grouts incorporating VMA exhibited significantly lower forced bleeding compared to no VMA mixtures.
4. The effect of combined addition of HRWR and VMA led to an increase of HRWR demand, and longer setting time. The highest retarding effect was observed for the grout made with the VMA-PMS combination.
5. Grouts prepared with the liquid-based cellulose VMA exhibited larger porosity than those without VMA. The low fluidity grouts made with PNS HRWR and VMA had higher volumes of relatively large pores compared to those made without any VMA. In the case of grouts prepared with PMS HRWR, such differences were not so pronounced.
6. The mortars containing VMA developed 6% to 16% lower compressive strength than those without any VMA. However, the compressive strength of mixtures made with PMS was higher that corresponding mixtures made with PNS. This was partially due to the lower capillary porosity.
7. The C-S-H gel structure was more dense and compact in cement paste made without any VMA, indicating more advanced cement hydration that in the case of VMA mixtures where the structure was rather heterogeneous.

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