



Performance of new viscosity modifying admixtures in enhancing the rheological properties of cement paste

M. Lachemi^{a,*}, K.M.A. Hossain^a, V. Lambros^a, P.-C. Nkinamubanzi^b, N. Bouzoubaa^b

^aDepartment of Civil Engineering, Ryerson University, 350 Victoria Street, Toronto, ON, Canada M5B 2K3

^bInternational Centre for Sustainable Development of Cement and Concrete (ICON), CANMET/Natural Resources Canada, 405 Rochester Street, Ottawa, ON, Canada K1A 0G1

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Abstract

The use of viscosity modifying admixtures (VMA) has proved to be very effective in stabilizing the rheological properties and consistency of self-compacting concrete (SCC). SCC is known for its excellent deformability, high resistance to segregation and use, without applying vibration, in congested reinforced concrete structures characterized by difficult casting conditions. Most of the commercial VMAs currently available in the market are costly and increase the price of such a concrete. Identification or production of new low-cost VMA is then essential. This paper presents the performance of four new polysaccharide-based VMAs in enhancing the rheological and consistency properties of cement paste. The study of the rheological properties and consistency of cement paste to screen the dosage and type of new VMA to be used in SCC is a promising approach. Investigation was carried out on cement pastes with combinations of various dosages of new VMAs and of a superplasticizer (SP) to study the influence on rheology, consistency and washout mass loss. A commercial VMA designated in this paper as “COM” was tested for comparison. The study on new VMAs is encouraging and confirms that pastes with satisfactory rheological and consistency properties comparable with or even better than commercial VMA can be developed. The combined use of proper dosages of VMA and SP is shown to clearly contribute to securing high-performance cement pastes that is highly fluid yet cohesive enough to reduce water dilution and enhance water retention. Attempt has also been made to correlate rheological properties (yield stress) to consistency (slump) of pastes.

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1. Introduction

Viscosity modifying admixtures (VMA) are water-soluble polymers that increase the viscosity of mixing water and enhance the ability of cement paste to retain its constituents in suspension. VMAs are used [1–3] to enhance the stability of self-compacting concrete (SCC). SCC is defined as a concrete that has excellent deformability and high resistance to segregation and can be filled in a heavily reinforced sections without applying vibration. SCC was developed in Japan [4] in the late 1980s, and recently, this concrete has gained wide use in many countries for different applications and structural configurations [5–9]. The use of

VMA along with adequate concentration of high-range water reducer (HRWR) can ensure high deformability and adequate workability leading to better resistance to segregation of such a concrete. Commonly used VMA in cement-based materials include polysaccharides of microbial or starch sources, cellulose derivatives and acrylic-based polymers [10].

Mixture containing VMA exhibits shear-thinning behavior whereby apparent viscosity decreases with the increase in shear rate. Such mixture (paste, mortar or concrete) is typically thixotropic where the viscosity buildup is accelerated due to the association and entanglement of polymer chains of the VMA at a low shear rate that can further inhibit flow and increase viscosity. Cellulosic and polysaccharide-based VMAs are normally used in conjunction with compatible HRWRs. The use of Welan gum, a kind of natural polysaccharide as VMA, has proved very effective in enhancing the rheological properties of grout and SCC

* Corresponding author. Tel.: +1-416-979-5000x6465; fax: +1-416-979-5122.

E-mail address: mlachemi@ryerson.ca (M. Lachemi).

[11]. It exhibits no apparent incompatibility with either melamine-based or naphthalene-based HRWRs for typical dosages of VMA and HRWR [12]. However, currently available commercial VMAs such as Welan gum are costly. Investigation is necessary to explore the potential use of new low-cost VMAs, which can be used in the concrete industry. The SCC currently available in the market is expensive due partly to higher prices of VMAs, and a low-cost product is desired to produce a competitive SCC for the construction industry.

The objective of this study was to investigate the performance of four different types of new VMAs based on various tests of rheological properties, fluidity, segregation and washout resistance of the cement pastes. A series of tests using viscometer to obtain rheological data such as yield stress and apparent and plastic viscosity along with minislump and washout tests were carried out to determine the robust mixture proportions for the cement pastes incorporating various dosages of superplasticizer (SP) and VMA. Rheological properties and consistency of cement paste play an important role in controlling the rheology and consistency of concrete. The current study on cement pastes and identification of suitable new VMAs are important and will provide the foundation of developing a cost-effective SCC with adequate rheological properties.

2. Experimental program

The experimental investigation was carried out on various cement paste mixes with four new VMAs in addition to a commercial VMA largely used in Canada and designated in this paper as “COM.” The flow of fresh paste is in the domain of fluid mechanics that deals with mass in motion, namely, a time-dependent parameter. Using static measurements to predict dynamic behavior is quite disputed. For this

Table 1
Chemical and physical properties of cement

ASTM cement type I				
Chemical analysis (%)		Physical tests		
Calcium oxide (CaO)	62.0	Specific gravity		3.17
Silica (SiO ₂)	20.3	Fineness		
Alumina (Al ₂ O ₃)	4.2	Passing 45 μ m (%)		94
Iron oxide (Fe ₂ O ₃)	3.0	Specific surface, Blaine (m ² /kg)		407
Sulfur trioxide (SO ₃)	3.5	Compressive strength (MPa; 51-mm standard mortar cubes)		
Magnesia (MgO)	2.8	7 days		32.0
Sodium oxide (Na ₂ O)	0.2	28 days		40.0
Potassium oxide (K ₂ O)	0.9	Setting time, Vicat test (min)		
Loss on ignition	2.0	Initial setting		220
		Final setting		325
		Air content of mortar (vol.%)		5.5

Table 2
Chemical and physical properties of VMA

	VMAs				
	A	B	C	D	COM
Total solids (%)	80.7	80.2–81.4	80.4–81.6	82.1	42.5
pH	4.9	4.9	4.8	4.8	7.0
Specific gravity	1.42	1.42	1.42	1.42	1.21
Viscosity (cP)					
26 °C	81,000	81,000	54,000	25,000	–
60 °C	2500	2500	1600	1000	–

reason, the Bingham model [13] was introduced to characterize the flow behavior of fresh paste, mortar and concrete by measuring the rheology data such as the yield stress, viscosity, shear stress and shear rate. The minislump test including horizontal spread measurements was used in evaluating the workability or consistency of the paste. This evaluation can be considered as time independent, as measurements are taken when the sample comes to a complete stop. In this study, both time-dependent tests such as rheology tests and time-independent tests (minislump tests) were conducted. Washout tests were also carried out in this experimental program.

2.1. Materials

2.1.1. Cement

Type 10 Portland cement (similar to ASTM type I) with specific gravity of 3.17 and Blaine fineness of 407 m²/kg was used. Chemical and physical properties are shown in Table 1.

2.1.2. Chemical admixtures

A SP composed of naphthalene formaldehyde condensates having specific gravity of 1.21 and total solid content of 40.5% was used. Four novel polysaccharide-based VMAs in liquid form classified as A–D having specific gravity of 1.42 and total solid content of about 81% were used (Table 2). All the tests in this study were carried out at room temperature. A known commercial VMA widely used in Canada and designated in this paper as “COM” was also used to perform a comparative study. The specific gravity and total solid content of COM were 1.21% and 42.5%, respectively (Table 2). The chemical composition of COM is a proprietary secret and it is composed of a combination of SP and VMA. The percentages of VMA and SP were calculated based on total solid content. New VMAs are soluble in water and dispersed homogeneously to create robust mixtures.

2.2. Fluidity tests

The minislump tests on paste were designed to study the performance of novel form of VMAs and to identify the best one that could be employed in the later stages of the

research for the development of SCC. The performance of new VMAs was compared with commercial VMA COM.

2.2.1. Minislump test procedure

The minislump test [14] was used to study the effect of VMAs on the fluidity of the paste. This test could also be used for studying the cement/SP compatibility. The test required small quantity of material and the results could be achieved quickly.

Cement was weighed in a bowl. Water, SP and VMA were weighed and mixed in a beaker. Then, cement was added to the solution and the mixture was hand mixed with a spatula for 1 min. The paste was then mechanically mixed with a blender for 2 min. Following the mixing sequence, the paste was poured into the minicone while sitting on a Plexiglas sheet and the cone was then lifted. The diameter of the spread paste was measured in two perpendicular axes and the average of the two was taken as minislump value. The paste was then placed back in the beaker and covered until the next test interval. The test was repeated to measure the slump at 5, 15, 30, 45, 60, 90 and 120 min. The tests were conducted at room temperature ranging from 22 to 25 °C.

2.2.2. Mix proportions

The proportions for the cement paste mixes identified as Series 1–4 are summarized in Table 3. The variable parameters in the mixes were VMA and SP contents. Twelve mixes using Types A–D VMA, six mixes using COM and two control mixes without VMA were used. The mixes had a constant water-to-cement ratio (W/C) of 0.45, new polysaccharide VMA content of 0.025%, 0.050% and 0.075% and COM content of 0.25%, 0.37% and 0.62%. SP content of 0.25% and 0.75% by weight of cement were used (Table 3). The content of COM and SP in the paste mixes were taken in accordance with the manufacturer-recommended dosages as used in a typical commercial SCC. The addition of SP to COM is recommended by the manufacturer although COM contains SP. The need for the addition of SP to COM was revealed from the preliminary investigation on trial mixes. Investigations on pastes with COM suggested that the addition of SP was necessary to generate slump flows comparable with those of new VMAs. Even then, higher dosages of COM were necessary in the pastes to generate slump flows comparable with those of new VMAs.

Table 3
Mix design of paste for minislump tests

	VMAs		
	A, B, C or D	COM	Control
W/C	0.45	0.45	0.45
VMA (%C)	0.025	0.25	0
	0.050	0.37	
	0.075	0.62	
SP (%)	0.25	0.25, 0.75	0.25, 0.75
Number of mixes	12	6	2

Table 4
Mix design of pastes for washout and rheological tests

	Washout tests		Rheological tests
	VMA Types A–D, COM	Control	VMA Types A–D, COM
W/C	0.45	0.45	0.45
VMA (%C)	0.025	0	0.025
	0.050		0.075
	0.075		
SP (%)	0.25, 0.5, 0.75	0.25, 0.5, 0.75	0.25
Number of mixes	45	3	10

2.3. Washout tests

The mix proportions of the cement pastes used to investigate washout mass loss are presented in Table 4. All pastes were prepared with 0.45 W/C and VMA (A–D and COM) concentrations of 0.025%, 0.05% and 0.075% by mass of cement. For each VMA dosage, three paste mixes were prepared with 0.25%, 0.50% and 0.75% of SP by mass of cement. These pastes were used to determine the effect of VMA–SP combination on washout mass loss.

The washout mass of cement paste was determined using a 750-ml paste sample that was introduced into a beaker containing an equivalent volume of water. A schematic representation of the washout test setup is shown in Fig. 1. The setup was similar to that used by Yahia [15]. The paste was first poured from a 750-ml upper beaker into a funnel, which was positioned at a given height above the water level of another 750-ml lower beaker completely filled with water. As grout fell freely in water through a fixed distance, it displaced the water and became partially diluted with it. The degree of dilution depends on the ability

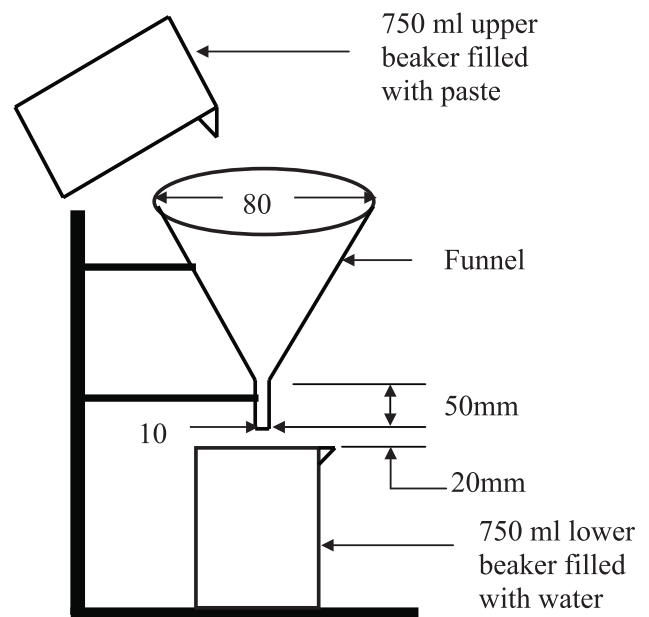


Fig. 1. Schematic representation of washout test setup.

of the grout to retain its mix water and suspended cement particles. In this process, suspended cement particles of the paste were also washed away with displaced water. The washout mass loss was determined by calculating the difference in mass of the paste before and after dropping it in water, expressed as percentage of initial mass [15]. All the tests were carried out at room temperature within the range of 22–25 °C.

2.4. Tests on rheological properties of cement pastes

Flow behavior of fresh paste is a complex rheological phenomenon that is roughly described by the Bingham model. The flow behavior of a test sample can be quantified by the measurable parameters of torque, and rotational speed with a rheometer, as demonstrated by Bingham model [13]. The purpose of using VMA in cement-based pastes is to improve the stability (bleeding) and rheological properties (viscosity, cohesion and internal friction or bond) to enhance the penetrability and flow characteristics.

2.4.1. Test procedure

Ten paste mixes shown in Table 4 were used. These included eight mixes with 0.025% and 0.075% of Types A–D VMA and two mixes using 0.025% and 0.075% of COM with W/C of 0.45. The SP content in all the paste mixes was kept constant at 0.25% by mass of cement. The mixing sequence was similar to that used in minislump test.

The rheological measurements of paste were conducted by using a commercially available digital Brookfield viscometer (Model RVDV-II) equipped with disc spindles at normal room temperature of about 22–25 °C. Tests were conducted at 5, 15 and 30 min interval following the contact between water and cement. The test samples were poured into the viscometer and the spindle was introduced in the beaker for measurement. The tests were executed stepwise at 100, 60, 50, 30, 20, 12 and 5 rpm. At each rotational speed, torque and apparent viscosity data were recorded. The results were then converted into viscosity functions such as shear stress (Pa) and shear rate (1/s) using standard procedure [17]. The linear regression analysis was carried out to determine the viscosity and yield stress as slope and intercept of the regression line drawn through the data points in shear stress versus shear rate plot.

The samples were allowed to stand and were covered after each measurement. Before starting the test for next time interval, the samples were manually stirred for 15 s.

3. Test results and discussion

3.1. Fluidity of pastes

Fig. 2 compares the influence of different types of VMA by showing typical variation of minislump diameter with

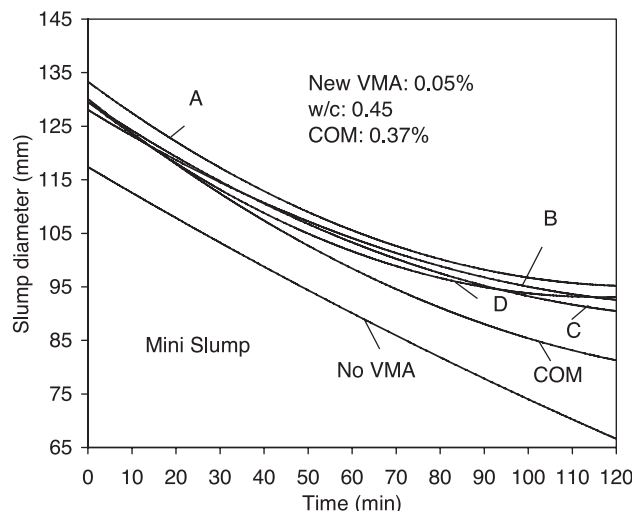


Fig. 2. Effect of type of VMA on the slump of paste (SP=0.25%).

time for pastes. The fluidity of new VMA pastes is much better than the control mix without VMA and commercial VMA COM. The minislump value (Fig. 2) in VMA pastes ranges between 127 and 132 mm at 0 min compared with 116 mm in control paste (0% VMA). To achieve similar fluidity at a particular dosage of SP (0.25% in Fig. 2), the paste needed much higher commercial VMA COM (about 0.37% compared with only 0.05% of new VMAs in Fig. 2). A number of trial paste mixes using different dosages of COM were tested to verify this conclusion. The minislump value also decreases with the increase of time. The minislump value decreases from a range of 127–132 mm to a range of 93–98 mm in new VMA pastes, from 130 to 82 mm in COM paste and from 118 to 65 mm in control paste (Fig. 2) within 2 h. This clearly indicates a better fluidity retaining capacity for pastes with the combinations of new VMA–SP than the COM–VMA and the control. The requirement of lower dosages to achieve satisfactory fluidity and better fluidity retaining capacity with time can be very useful in developing a satisfactory and cost-effective SCC with new VMAs. Fig. 3 shows the influence of dosages of Type A VMA and time on the fluidity of the paste. The slump value increases with the increase of dosages of VMA in the mixes. Similar phenomena were also observed in pastes with other new VMAs. All VMAs (with Type A marginally better) prove to be quite effective in all the paste mixes and shows better results compared with commercial VMA COM.

3.2. Washout resistance of pastes

Typical washout resistances of paste with VMAs A and B and COM are compared with those of control paste without VMA in Fig. 4. For similar dosages of VMA and SP, washout resistances of new VMAs A and B are found to be better than the commercial COM and control pastes. Washout mass losses were higher in COM pastes (ranges

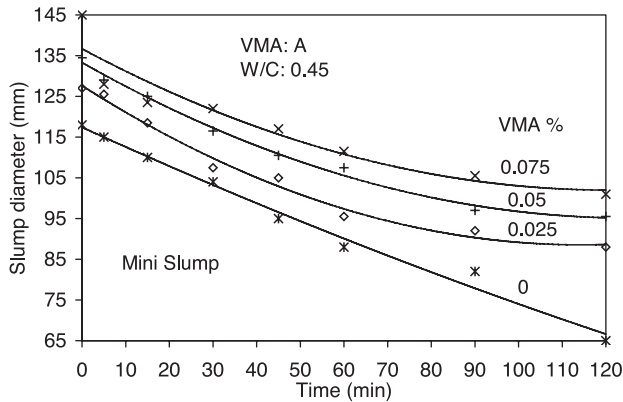


Fig. 3. Effect of VMA content on the slump diameter of paste (SP=0.25%).

between 12.6% and 14.9%) compared with VMA Types A (ranges between 6.7% and 12.4%) and B (ranges between 7.5% and 13%) pastes. The washout resistance of the control paste was similar to that of COM pastes. Improvement in washout resistance was not observed in pastes with COM compared with control pastes for the SP–VMA combinations of dosages used in this study. VMA Types A and B pastes are found to have similar washout resistances. Similar behavior was also observed in pastes with VMAs C and D. These results are similar to those obtained by Khayat and Yahia [16] in their investigation on the combination of Welan gum–HRWR. Washout resistance improves with increasing concentration of VMA coupled with a greater content of SP to maintain the desired fluidity. Therefore, by adjusting the combination of VMA–SP, a washout-resistant paste with adequate fluidity can be obtained. The increase in the dosage of SP in paste disperses the cement grains and increases the amount of free water in the system. The higher SP dosage may be the cause for no improvement in washout resistance of pastes with COM compared with control paste.

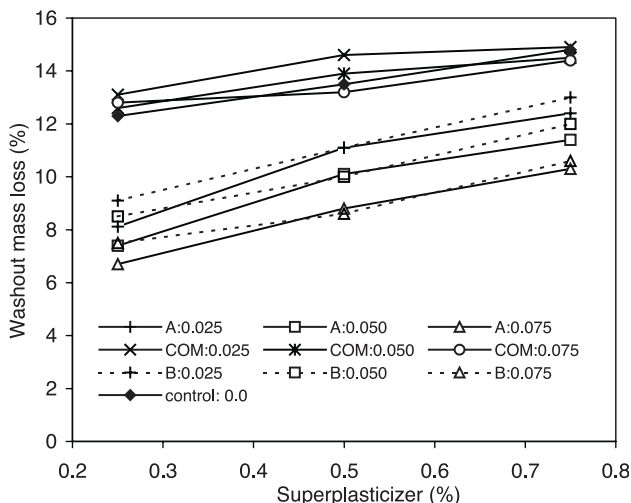


Fig. 4. Washout mass loss of paste.

As COM is a combination of SP and VMA, the actual SP content in the COM paste is higher than the pastes with new VMA. This leads to the higher washout mass loss in pastes with COM (Fig. 3). The combined addition of SP and VMA can improve both fluidity and washout resistance. The improvement in washout resistance is due to the enhancement in the degree of water retention by the VMA, whereby some of the free water made by the addition of SP can be physically adsorbed by hydrogen bonding onto polymer molecules of the VMA. Furthermore, some of the VMA polymer becomes adsorbed onto cement grains along with the imbibed water, resulting in further retention of suspended cement particles. The use of VMA increases the viscosity of the paste, which reduces the rate of sedimentation of cement grains, thus resulting in highly stable paste even at elevated fluidity levels [16].

3.3. Rheological properties of pastes

The variation of apparent viscosities with shear rate for pastes with 0.025% and 0.075% of various VMAs at 15 and 30 min is shown in Figs. 5 and 6. Apparent viscosity decreases with the increase of shear rate. The apparent viscosities of pastes with Types A and B VMA are found to be higher than those of the other VMAs including commercial COM. The apparent viscosity is also increased with the increase of the dosages of VMA from 0.025% to 0.075% as can be seen from the typical graphs shown in Figs. 7 and 8 for Types A and B VMA. For any given concentration of SP (0.25% in the current study), the increase in VMA content should increase the viscosity at both high and low shear rates, and this was observed in the current study (Figs. 7 and 8).

The apparent viscosity of paste with 0.025% of Type A VMA is increased from 1500 MPa s at 10 s^{-1} to 11,500 MPa s at 1.5 s^{-1} compared with 7000 MPa s at 10 s^{-1} and 20,500 MPa s at 1.5 s^{-1} in paste with 0.075% Type A VMA (Fig. 7). Similar behavior is also observed in pastes with Type B VMA (Fig. 8).

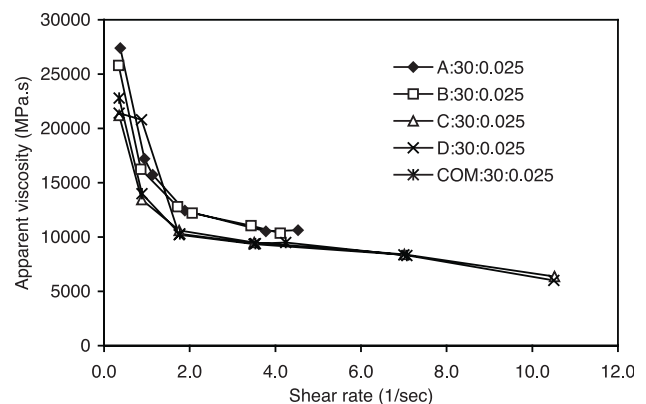


Fig. 5. Variation of apparent viscosity with types of VMA.

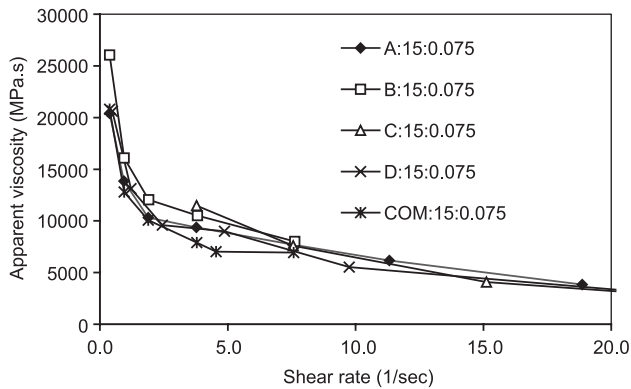


Fig. 6. Variation of apparent viscosity with types of VMA.

This can be attributed to the fact that the degree of water retention and the free water needed to lubricate the paste increase with the dosage of VMA that acts on the aqueous phase. The addition of VMA also increases the degree of pseudoplasticity or shear thinning of cement paste regardless of the concentration of SP. Pastes with VMA exhibit high apparent viscosities at low shear rates and significantly lower viscosities at greater shear rates. For the same dosage of SP, the use of VMA results in a greater apparent viscosity at low shear rate than at high shear rate (Figs. 5–8).

The increased pseudoplastic response in the presence of VMA is believed to be because the polymer chains of the VMA entangle or associate, resulting in an increase in apparent viscosity, especially at low shear rate. With the increase in shear rate, the entangled chains dislodge and align in the direction of flow, thus decreasing the resistance of the grout to undergo deformation. The apparent viscosity is then decreased with an obvious improvement in flowability at high shear rate regimes [16].

The effect of the increase in the concentration of VMA on viscosity depends on the shear rate. For a given concentration of SP, the increase in the dosage of VMA is more effective in increasing viscosity at low shear rate than that at high shear rate (Figs. 5 and 6).

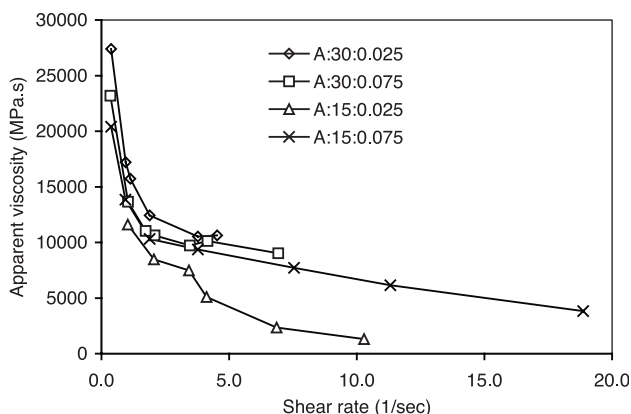


Fig. 7. Variation of apparent viscosity with time and dosages of VMA.

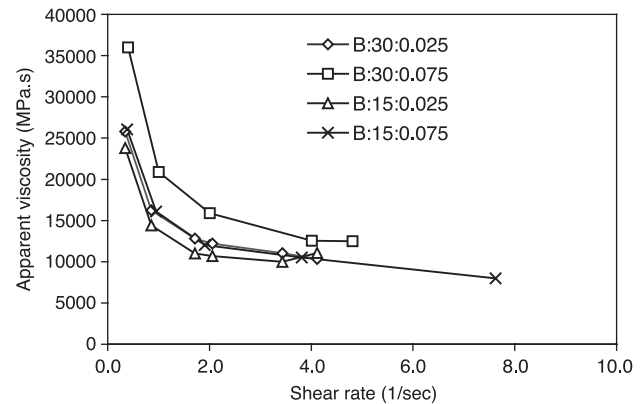


Fig. 8. Variation of apparent viscosity with time and dosages of VMA.

Fig. 9 compares the yield stress of pastes with different dosages and types of VMA. The yield stress is decreased with the increase of dosages of VMA from 0.025% to 0.075%. The yield stress, viscosity and apparent viscosity values are affected by the combination of dosages of VMA and SP. It is then important to find out the combinations of dosages of VMA and SP to secure a stable paste with required fluidity and rheological properties. This can be achieved by testing trial mixes with various combinations of dosages of SP and VMA as illustrated in this study.

Fig. 10 shows a typical variation of viscosity of paste with time (elapsed between mixing and testing) for various types of VMA. Viscosity is found to increase with the increase of elapsed time. This can be attributed to the hydration of cement with time that made the paste stiffer as time progresses. This also indicates somehow that the new VMAs are not inhibitors for the cement hydration.

The performance of Types A and B VMA is found to be better than the other VMAs for similar dosages of VMA and SP used in this study.

3.4. Correlation between rheological and consistency properties

Fig. 10 shows a typical variation of viscosity and slump diameter with time. The viscosity increases and the slump

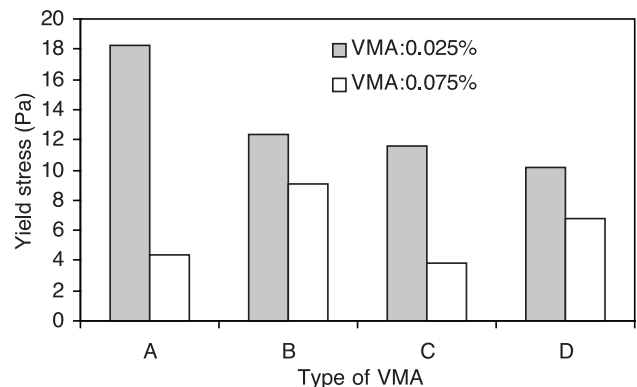


Fig. 9. Variation of yield stress with VMA concentration.

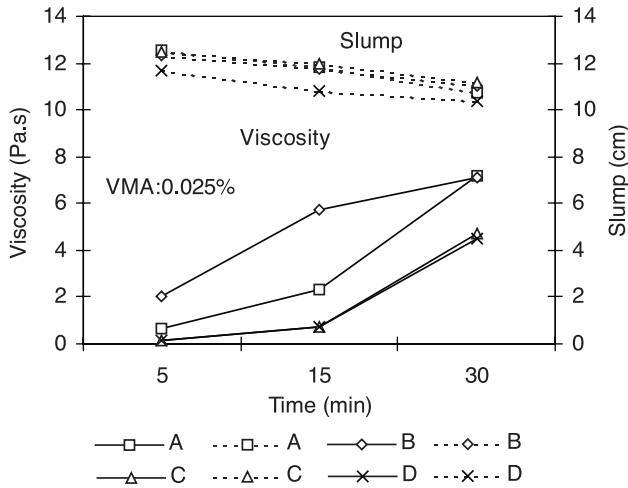


Fig. 10. Viscosity and slump as a function of time and types and dosages of VMA.

value decreases with time. It can be seen that an increase in the viscosity of the paste reduces the flowability. The increases in Types A and B VMA content from 0.025% to 0.075% can increase the viscosity in wider range than the other VMAs (Figs. 7 and 8). This justifies the suitability of Types A and B VMA and the need of finding an optimum dosage that can develop adequate viscosity to ensure an optimum flowability.

The plot of yield stress of paste versus minislump diameter (as shown in Fig. 11) shows a weak correlation. However, the trend shows that a higher yield stress corresponds to a lower spread in the minislump. Therefore, a qualitative indication of the yield stress could be obtained from the minislump. This result was expected [18] because the cement paste in a minislump test will only flow if the stress due to the weight of the paste contained in the cone is higher than the yield stress of the cement paste. Despite some of the scatter of the data

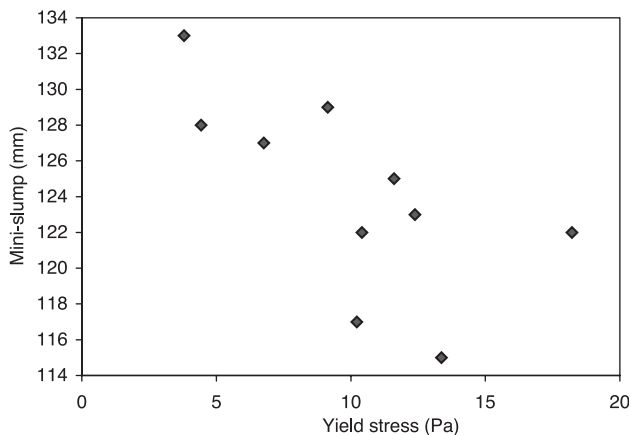


Fig. 11. Correlation between paste yield stress and minislump diameter.

shown in Fig. 11, an approximation of the yield stress could be obtained by fitting a straight line through the data.

A correlation between viscosity and yield stress is achieved as shown in Fig. 12. The trend shows a decrease in yield stress with the increase in viscosity. The data points in this co-relation were derived from pastes with 0.25% SP and having VMA content of 0.025% and 0.075%. The viscosity and yield stress of the paste were measured at 5, 15 and 30 min intervals. Therefore, the co-relation takes into account the effect of SP and VMA contents as well as the time dependence of the fluidity. These results show again that the combinations of new viscosity enhancing admixtures and SP can produce very stable and flowable pastes.

3.5. Related investigations

The influence of new VMAs on the setting time of paste was not studied. However, extensive investigations were carried out on mortars and SCC mixtures to investigate the effect of new VMAs on setting times as well as fresh and hardened properties [19,20]. The investigation on mortars showed an increase in setting times with the increase of VMA dosage. Yet, the increase in setting times with the increase of the dosage of VMA was higher in mortars with A–D VMAs compared with COM VMA. However, to achieve similar setting time, the new VMAs needed five times less VMA (only 0.075%) than commercial COM VMA (0.37%).

It is also revealed that a satisfactory SCC can be achieved by using less amount of new Type A VMA than the manufacturer-recommended dosage of commercial VMA COM and Welan gum [20]. To achieve comparable and acceptable fresh properties and better strength characteristics, SCC with new Type A VMA required 7.4 and 1.25 times lower VMA compared with commercial COM and Welan gum SCC mixtures, respectively. The use of new Type A VMA also produced cost-effective SCC [20]. However, the long-term durability characteristics of such

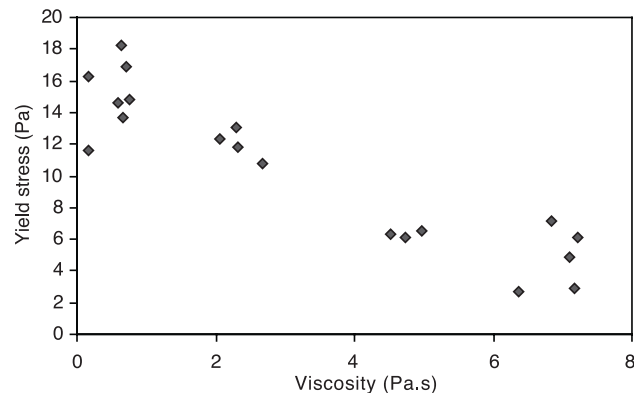


Fig. 12. Correlation between yield stress and viscosity.

SCCs with new types of VMA and their performance in structural elements are important issues and should be considered in its future application in the construction industry.

4. Conclusions

The performance of four different novel VMA designated as Types A–D compared with a commercial VMA designated as “COM” was investigated. The influence of various dosages and types of VMA in addition to the dosages of SP on fluidity, viscosity, yield stress and washout resistance of cement pastes made with a W/C of 0.45 was studied. Based on the results presented in this paper, the following conclusions can be derived:

1. Minislump value increases with the increase of dosages of VMA from 0.025% to 0.075% for a fixed dosage of SP. Minislump value decreases with the increase of elapsed time between mixing and testing, somehow indicating that the hydration of the cement pastes is not inhibited. Based on investigation, all new VMAs prove to be quite effective (with Type A marginally better) in enhancing the consistency of all the paste mixes, showing better flowability compared with commercial VMA COM.
2. The apparent viscosity of the cement paste is increased with the increase of dosages of VMA from 0.025% to 0.075%. The viscosity of Types A and B VMA is found to be higher than other new VMAs and commercial COM particularly at low shear rate of up to 5 s^{-1} . Viscosity of the pastes also increases with the increase of elapsed time between mixing of paste and testing. Based on viscosity data, Types A and B VMAs are found to be more efficient than other new VMAs and would provide better rheological properties.
3. The washout resistance is enhanced by the increase in VMA dosage and reduction in SP content. However, with a proper use of VMA–SP combination, highly flowable yet washout-resistant mixtures can be secured. Washout resistance of Type A, B, C or D VMA is found to be higher than that of commercial VMA COM for similar dosages of VMA–SP combination.
4. The minislump test results correlate roughly with the yield stress of the cement paste even if there is a scatter of data. General trend shows a decrease in yield stress of the paste with the increase in slump. This suggests that a correlation between rheological properties and consistency of paste can be achieved with sufficient test data.
5. A correlation between viscosity and minislump of paste shows a decrease in minislump with the increase in viscosity of the paste.
6. Based on the current investigation, cement pastes with 0.05% of either Type A or Type B VMA would provide better rheological properties at W/C of 0.45 and SP content of 0.25%.
7. The conclusions reached based on the tests on paste in the selection of type and dosage of new VMA (in this case, VMA Types A or B) could be used in the development of a SCC with satisfactory properties, and research is now focused in that direction. The future research will also validate the prediction of the influence of VMA on SCC rheology based on that on the rheology of cement paste.

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