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Shear-thickening behavior of high-performance cement grouts — Influencing mix-design parameters

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ABSTRACT

Rheology of concrete is of great importance to its flow performance, placement and consolidation. A full understanding of fresh concrete flow behavior can be achieved through a good understanding of paste rheology. Cement pastes exhibit a complex rheological behavior affected by several physical and chemical factors, including water-to-cement ratio (w/c), high-range water-reducer (HRWR) type and dosage, and cement characteristics. An experimental investigation was carried out to evaluate the effect of w/c, HRWR–cement combinations, and supplementary cementitious materials (SCM) on the pseudoplastic behavior of high-performance cement grouts. Grout mixtures proportioned with w/c of 0.30, 0.33, 0.36, and 0.40, various cement–HRWR combinations, and cement substitutions by 8% silica fume were investigated. The incorporation of HRWR can lower the yield stress of mixtures, thus enhancing deformability, while silica fume improves mechanical and durability performances.

High-performance structural grouts are shown to exhibit shear-thickening behavior at low w/c and shear-thinning behavior at relatively higher w/c. Mixtures made with polycarboxylate HRWR acting by steric effect exhibited greater shear-thickening behavior compared to those made with polynaphthalene sulfonate-based HRWR acting by electrostatic effect. The paper discusses the effect of mixture parameters on non–linear rheological behavior of various grout mixtures prepared with different w/c, HRWR–cement combinations, and silica fume.

1. Introduction

Fresh concrete is a solid suspension of aggregate in cement paste. Investigations of cement paste rheology are motivated by the need to understand flow properties of concrete to secure good flow performance, placement, and consolidation. In addition, rheology can be used as a design parameter of high-performance concrete to tailor appropriate flow curves that satisfy the various requirements, including pumping pressure and distance, free fall of concrete into formwork, required stability level after placement and consolidation during the dormant period. Development of self-consolidating concrete (SCC), characterized by high deformability to facilitate casting and ensure adequate mechanical and structural performance, is an example of the tremendous benefit of using rheology as a mix design tool. The incorporation of HRWR in SCC to reduce the yield stress can result in enhancing deformability of mixtures [1,2].

Cement paste may be considered as a flocculent system due to the effects of Van der Waals attraction and hydrodynamic forces, as well as chemical reactions, resulting in a complex rheological behavior affected by several physical and chemical factors [3–6]. The flow behavior of cement paste is dominated by its solid concentration, size distribution of cement grains, chemical composition of cement, temperature, mixing energy, and the presence of HRWR. Such behavior is further complicated by shear history, the use of low w/c, and in some cases, supplementary cementitious materials (SCM) incorporated to enhance fresh and hardened properties. Rheological behavior of low w/c mixtures, is greatly influenced by cement–HRWR interaction and dispersing mechanism of HRWR.

The dispersing efficiency of an HRWR is mainly related to the chemical effects that are inherent functions of the reactive nature of cement particles. This may include preferential adsorption, chemisorptions, and chemical reactions to form new hydrate phases [7,8]. The dispersing mechanisms induced by an HRWR can be a combination of both electrostatic and steric repulsive forces [7,9–13]. Anderson has suggested that high molecular weight polymers lead to additional short-range forces, while low molecular weight polymers usually exhibit lower fluidity and poor fluidity-retention with time [14,15]. Uchikawa et al. reported that electrostatic forces play a major role in the dispersing mechanism of polynaphthalene sulfonate condensate (PNS) superplasticizers, while steric forces are dominant for a copolymer of acrylic acid with acrylic ester [16].

High-performance cement grouts incorporating a new generation of HRWR and viscosity modifying admixtures (VMA) exhibit a higher degree of pseudoplastic properties compared to conventional grouts [17–25]. Structural cement grouts proportioned with 0.40 w/c, different VMA–HRWR combinations, and various SCM, including silica fume, blast-furnace slag, and fly ash, exhibited a shear thinning behavior even

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at higher shear rates up to 600 s^{-1} [17–21]. Jones and Taylor investigated the flow curves of cement paste using a cone and plate viscometer at shear rate up to 150 s^{-1} and reported that cement paste was shear-thickening material at w/c below 0.40, a shear-thinning material above 0.40, and a Bingham material at 0.40 [22]. Experimental data generated on 78 mixtures (concrete and mortar) made with and without admixtures exhibited shear-thickening behavior using a plane-plane rheometer [26]. Recently, it has been reported that the use of some HRWR types resulted in shear-thickening response of cement paste. Rheological flow curves of 0.32 w/c pastes made with three different types of HRWR, including a melamine resin, lignosulfonate, and a polyacrylic-based HRWR were determined using a coaxial rheometer, and test results showed that when used above a certain critical dosage, the polyacrylic-based HRWR resulted in shear-thickening response [27]. Cement pastes proportioned with water-to-powder ratio between 0.22 and 0.26 exhibited shear-thickening behaviors when using HRWR mainly acting by electrostatic effect. It is also reported that mineral admixtures, such as metakaolin, increase the shear-thickening response, while silica fume reduces it [28]. The authors explained this behavior by the presence of superplasticizer as a physical component of the paste, which could increase the disorder of the system at higher shear rate. Hoffman conducted rheological and light diffraction measurements and reported the existence of critical shear rate where the suspension changes from an ordered state to a disordered one [29]. Hydrolyzed polyacrylamide solutions containing 20 g/L of NaCl exhibit shear-thinning behavior at shear rates lower than 27.4 s^{-1} and shear thickening at higher values [30]. On the other hand, Brady and Bossis carried out dynamic simulation and showed that shear-thickening may be due to the formation of a transient cluster that obstructs the flow [31].

The objective of this study is to evaluate the effect of w/c, HRWR and cement–HRWR combinations, and silica fume on rheological flow curves of high-performance grouts. This can therefore enable a good understanding of the effects of changes in mixture proportioning and material properties on rheology of cement-based materials. An attempt is made to correlate the variation of pseudoplastic and shear–thickening responses to w/c and packing density of the mixtures by means of incorporating silica fume.

2. Experimental program

Experiments were carried out using two different types of cement, various water-to-cement ratios (w/c) of 0.30, 0.33, 0.36, and 0.40, and a PNS and polycarboxylate ether (PCE) HRWR. The two cement types were a Portland cement (PC) and a low heat cement (LHC). Silica fume was used at a partial replacement rate of 8% in some mixtures. For all the w/c and cement–HRWR combinations, the HRWR saturation dosages were first determined. This approach consisted in determining the optimum HRWR dosage to ensure proper dispersion of the powder material beyond which no further significant improvement in fluidity can be achieved. Rheology of various well-dispersed grout mixtures was then determined using a coaxial cylinders viscometer.

2.1. Materials and mixture proportioning

Ordinary and low heat cement types complying with ASTM C150 Type I and IV cements, respectively, and a densified silica fume were used. The chemical and physical properties of these materials are given in Table 1. The silica fume has a mean diameter (D_{50}) of 0.065 μm, which is approximately 200 times lower than that of Portland cement.

A polynaphthalene-based HRWR (PNS) with solid content of 42% and specific gravity of 1.21, as well as a polycarboxylate-type HRWR (PC) with solid content of 42% and specific gravity of 1.09 were used in this investigation. Water in the HRWR was accounted for to maintain constant w/c.

Table 1
Chemical and physical properties of cements and silica fume.

	Portland cement (PC)	Low heat cement (LHC)	Silica fume (SF)
SiO_2	20.9	25.4	93.6
Al_2O_3	4.5	3.5	0.3
Fe_2O_3	2.9	3.5	0.5
CaO	64.9	62.5	0.3
MgO	1.5	1.1	0.5
SO_3	2.0	2.2	–
Na_2O eq.	0.62	0.24	1.4
Free CaO	1.1	–	–
C	–	–	1.9
LOI	2.5	2.2	2.8
Specific gravity	3.16	3.15	2.22
Blaine fineness, m^2/kg	331	347	20 000
B.E.T. surface area, m^2/kg	–	–	17 300
Mean apparent diameter (μm)	16	18	0.065
% passing 45 μm	90	92	100
C_3S (%)	60.5	24.0	–
C_2S (%)	14.1	56.0	–
C_3A (%)	7.0	3.0	–
C_4AF (%)	8.8	10.0	–

2.2. Test methods

All grouts were mixed in 6-L batches using a high-shear mixer rotating at approximately 1500 rpm. Such a high-shear mixing regime is used to ensure complete dispersion of cement particles and homogeneous suspension. The temperature of mixing water was controlled to keep the temperature of grouts at 23 ± 1 °C. Following the end of mixing, all grouts had constant temperatures of 23 ± 2 °C. The mixing sequence consisted of mixing water and HRWR for 20 s. The cement was then introduced gradually during 60 s while the mixer was turned on, and the mixture was mixed for a total time of 120 s. After a rest period of 60 s, the mixing was resumed for 60 s.

The saturation dosage of HRWR was determined for all w/c and HRWR–cement combinations. The saturation dosage for a given cement–HRWR combination consists of the optimum dosage of HRWR beyond which there is no improvement in fluidity. Fluidity was measured using a mini-slump cone having an upper diameter of 19 mm, a lower diameter of 38 mm, and a height of 57 mm [32,33]. The test consists in measuring the flow diameter of a given volume of grout (38 ml) placed in the mini-cone on a Plexiglas plate. Research results have shown that there exists a good correlation between spread diameter of the grout, determined using the mini-slump, and its yield stress [33]. Saturation dosages were then used to prepare various grout mixtures. Rheological flow curves of the grouts were determined using a coaxial cylinders viscometer with smooth surfaces (Chandler Engineering Model 3500). The diameters of rotor and bob are 1.8415 and 1.7245 cm, respectively, providing a shear gap size of 1.17 mm and a radii ratio of 0.9365. The bob has a height of 3.8 cm. The test protocol consists of shearing the paste at a high shear rate of 240 s^{-1} during 60 s to ensure complete structural breakdown of the structure. The flow curve was then determined by decreasing the shear rate from 240 s^{-1} to 1.7 s^{-1} during 90 s. The rheological measurements were completed between 10 and 13 min following initial contact of cement with water.

3. Test results and discussion

3.1. Saturation dosages of HRWR

HRWR saturation dosages depend on a number of factors, including the specific surface area and chemical composition of cement, especially the C_3S/C_2S, C_3A/C_4AF and $C_3A/CaSO_4$ ratios, alkalis content, and w/c of the mixture [8,34]. The HRWR saturation dosages of the investigated mixtures are summarized in Table 2.

Table 2
Saturation dosages of HRWR determined on various mixtures.

	Portland cement (PC)		Low heat cement (LHC)	
	PNS (%)	PCE (%)	PNS (%)	PCE (%)
w/c = 0.30	0.43	0.20	0.45	0.14
w/c = 0.33	0.40	0.16	0.45	0.12
w/c = 0.36	0.34	0.12	0.35	0.08
w/c = 0.40	0.28	0.07	0.26	0.06

When combined with PNS HRWR type, Portland cement (PC) required relatively lower HRWR dosages to ensure a given dispersed system than low heat cement (LHC), regardless of the w/c in use. However, LHC requires a lower dosage of HRWR than PC when PCE HRWR is used. For a given cement type and w/c ratio, the optimum PCE HRWR dosages are lower than those of PNS type. The dispersing efficiency of HRWR is mainly related to its dispersing mechanisms, the chemical effects that are inherently functions of the reactive nature of cement particles and their interaction with HRWR [8,11,34].

Furthermore, test results indicated that grout mixtures incorporating PCE-type HRWR showed higher initial fluidity than those made with PNS, regardless of the w/c and cement type. For example, grouts prepared with PCE HRWR achieved mini-slump values of 120 ± 10 mm measured 10 min after initial contact between water and cement. Furthermore, better fluidity retention (up to 90 min) was observed with grouts incorporating PCE HRWR than those made with PNS.

3.2. Effect of w/c and HRWR type on flow curve of cement grouts

Rheology of cement-based materials is affected by the w/c, HRWR type and dosage, and the characteristics of the cement in use. Rheological flow curves were determined on different mixtures made with various w/c and two different HRWR types (PCE and PNS). The investigated grouts were prepared using Portland cement (PC) and corresponding optimum HRWR dosages given in Table 2. The Herschel–Bulkley model was used to fit the shear stress–shear rate data. This model is given by: $\tau = \tau_0 + k\gamma^n$, where τ_0 is the yield stress, k is the consistency, and n is the characteristic of the mixture's behavior. The mixture is shear-thinning when $n < 1$ and shear-thickening when $n > 1$. Flow curves of the investigated mixtures containing PCE and PNS HRWR types are summarized in Figs. 1 and 2, respectively, and the values of n and the mean square error (MSE) are given for each curve.

Higher shear-thickening response is observed with low w/c mixtures, regardless of HRWR type. However, as the w/c increases the shear-thickening response decreases and may completely disappear for higher w/c, regardless of HRWR type. For example, the pseudoplastic index of a mixture made with PCE HRWR decreases

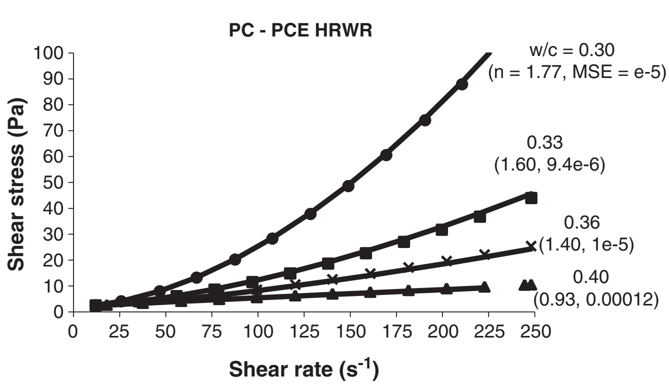

Fig. 1. Flow curves of mixtures made with Portland cement, various w/c, and PCE HRWR.

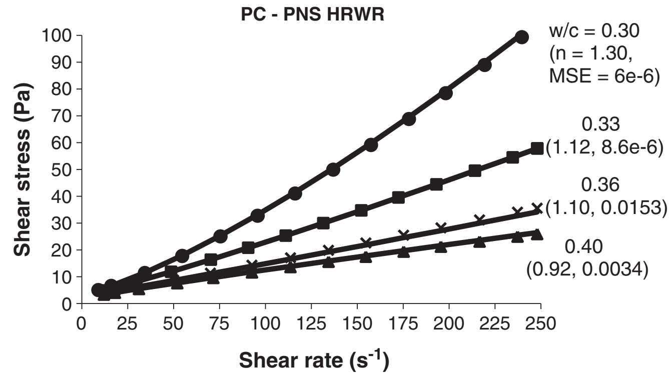

Fig. 2. Flow curves of mixtures made with Portland cement, various w/c, and PNS HRWR.

from 1.77 to 1.60 and 1.40 when the w/c increases from 0.30 to 0.33 and 0.36, respectively. Further increase in w/c to 0.40 resulted in shear-thinning behavior ($n = 0.93$).

The use of PCE HRWR acting by steric effect resulted in higher shear-thickening responses than those made with PNS, especially for lower w/c. For example, 0.30 and 0.33 w/c mixtures made with PCE HRWR showed a shear-thickening behavior with n values of 1.77 and 1.60, respectively. In the case of PNS HRWR, the power indexes are 1.30 and 1.12, respectively. For mixtures proportioned with 0.36 w/c, the use of PCE and PNS resulted in shear-thickening indexes of 1.40 and 1.10, respectively. Mixtures proportioned with relatively higher w/c of 0.40 exhibited comparable shear-thinning behavior (n values of 0.93 and 0.92), regardless of HRWR type.

3.3. Effect of cement type on pseudoplastic behavior of cement grouts

Flow curves of grout mixtures prepared with low heat cement (LHC) and incorporating PCE and PNS HRWR types are presented in Figs. 3 and 4, respectively.

For a given w/c and HRWR type, test results showed that the use of Portland cement (PC) resulted in greater shear-thickening behavior compared to low heat cement, regardless of HRWR type. For example, for 0.30 w/c mixtures and PCE HRWR, a shear-thickening index of 1.23 is obtained when LHC is employed. However, in the case of mixtures made with PC, higher shear-thickening response ($n = 1.77$) is observed. On the other hand, for mixtures made with LHC, the shear-thickening behavior is observed with a w/c of 0.30. For higher w/c, Bingham and shear-thinning behavior ($n \le 1$) is observed, regardless of HRWR type.

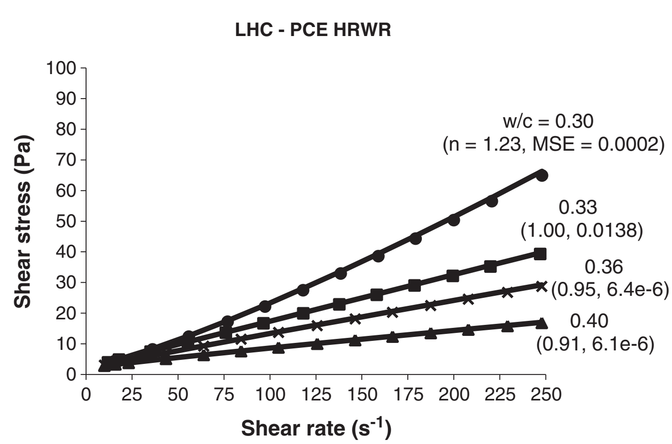

Fig. 3. Flow curves of mixtures made with low heat cement, various w/c, and PCE HRWR.

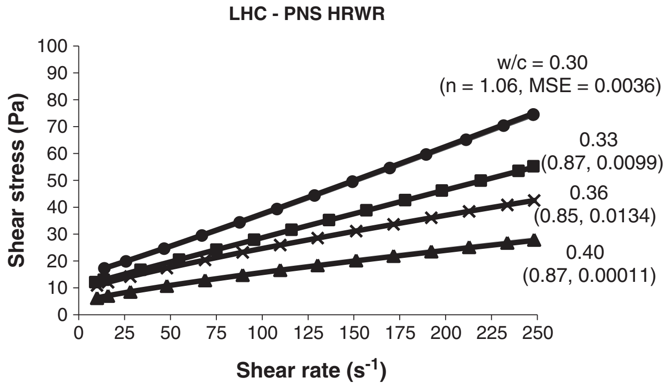

Fig. 4. Flow curves of mixtures made with low heat cement, various w/c, and PNS HRWR.

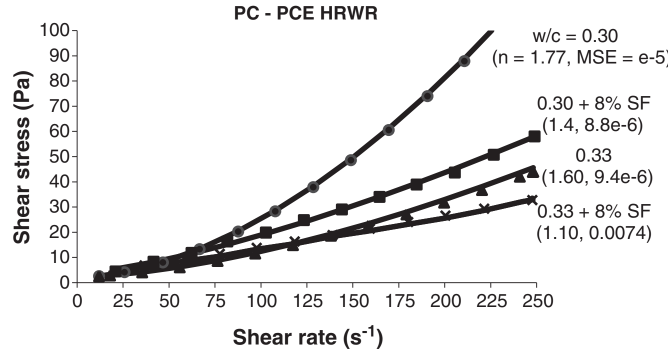

Fig. 5. Flow curves of mixtures made with Portland cement, 8% silica fume, and PCE HRWR.

For 0.30 w/c mixtures, the use of PCE HRWR resulted in greater shear-thickening behavior compared to mixtures made with PNS-type HRWR. For example, mixtures made with PCE HRWR exhibited a shear thickening behavior with n value of 1.23, while those made with PNS showed a power index of 1.06. The increase in w/c to 0.33, 0.36, and 0.40 resulted, in general, in shear-thinning behavior ($n<1$), regardless of HRWR type in use.

Based on the results obtained, it can be stated that the shear-thickening response is most likely to be predominant with highly concentrated cement suspension and reactivity. The presence of HRWR, especially the type acting by steric effect, also seems to promote the shear-thickening response. This can be explained by the fact that the presence of polymer chains into solution can cause a disorder state, especially at high shear rate. Indeed, under shearing, and depending on the elasticity of polymer chains, the presence of polymer can promote cluster formation, which results in greater viscosity of the system [27,35–37]. On the other hand, the adsorbed polymer may be desorbed when the shear rate increases, thus increasing polymer concentration into the interstitial solution, which may lead to an increase in the viscosity of the solution with shear rate. The occurrence of this phenomenon remains to be clarified.

3.4. Effect of silica fume

The partial replacement of cement by finer particles, such as silica fume, can provide useful information in understanding the shear-thickening behavior observed with grouts made with PCE HRWR and low w/c of 0.30 and 0.33. Rheological behavior of mixtures proportioned with 0.30 and 0.33 w/c and containing PCE HRWR are investigated. The flow curves for mixtures made with ordinary and low-heat cements, and incorporating 8% silica fume used as partial cement replacement are presented in Figs. 5 and 6, respectively.

As can be seen in Figs. 5 and 6, the incorporation of 8% silica fume in mixtures proportioned with 0.30 and 0.33 w/c resulted in lower shear-thickening response compared to mixtures made without silica fume, regardless of the cement type. Similar observations were reported by Cyr et al. [28]. The incorporation of 8% silica fume in mixtures made with PC resulted in lowering the shear-thickening response (n value) from 1.77 to 1.40 and from 1.60 to 1.10 for w/c of 0.30 and 0.33, respectively. In the case of mixtures made with LHC, the use of 8% silica fume changed the response from shear-thickening to almost linear Bingham behavior and from Bingham behavior to shear-thinning response (n values of 1.00 vs. 0.96) for mixtures proportioned with 0.30 and 0.33 w/c, respectively.

Silica fume has a mean diameter (D_{50}) of 0.065 μm, which is approximately 200 times lower than that of Portland cement. The specific surface area of the silica fume is 50 times higher than that of both cements. The reduced shear-thickening response with silica fume mixtures can be due to the finer particles that can be inserted between cement particles, thus reducing inter-particle friction by bearing effect. Previous research has shown that when incorporated in well-dispersed systems, the use of proper silica fume content can lower yield stress and viscosity of cement-based materials [23,24], thus resulting in lower HRWR demand for a given fluidity level. It can be stated that shear-thickening response can be decreased or even eliminated as the mixture becomes more polydispersed by adding finer particles to enhance the powder skeleton. This bearing effect seems to be more important in the case of Portland cement.

As highlighted by test results, and as can be observed in Fig. 7, shear-thickening response can be affected by various mixture parameters, such as w/c, HRWR type, chemical composition of cement, and silica fume. An increase in w/c, i.e. reducing the solid concentration, decreases the shear–thickening behavior of grouts, especially in the case of shear-thickening response. The highest decrease (negative slope) is observed with grouts made with PC–PCE HRWR combinations. A similar trend is generally observed for the other HRWR–cement combinations. In the case of mixtures incorporating silica fume, the increase in w/c showed the greatest decrease in shear-thickening response.

4. Discussion

The balance between the attractive forces and the repulsive forces due to surface charge or adsorbed species governs the behavior of particulate suspensions [38]. For many years, several authors explained the pseudoplastic and shear–thickening behavior of solid particles by the order–disorder state and flocculated–deflocculated system caused by attractive and repulsive forces [28,35,36]. The Brownian forces acting on particles are dominated by hydrodynamic forces [25,39–41]. When the hydrodynamic forces are sufficiently

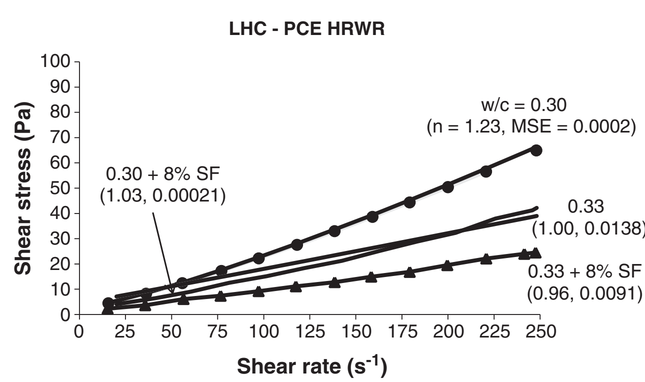

Fig. 6. Flow curves of mixtures made with low-heat cement, 8% silica fume, and PCE HRWR.

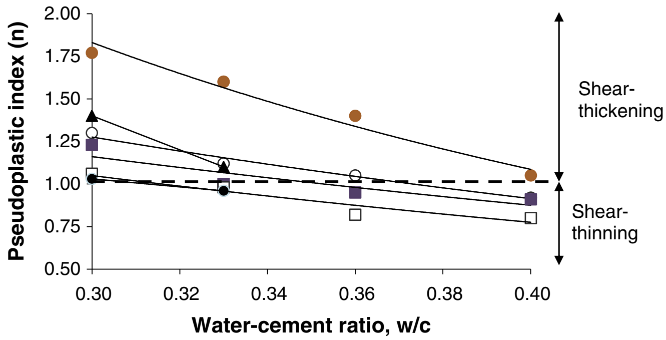

Fig. 7. Variation of pseudoplastic index (n) with w/c of various mixtures.

large to overcome the attractive forces and ensure a dispersed system, an ordered state prevails, and the system behaves as a shear-thinning material characterized by a decrease of apparent viscosity with shear rate. However, when the hydrodynamic forces overcome repulsive interparticle forces, hydroclusters form, resulting in particle collisions where a state of disorder may take place [25,29].

An increase in degree of shear-thinning behavior can be useful to increase the stability of cement-based systems after casting (static stability) without hindering transport and casting operations, where the material is subjected to higher shear rates. However, the existence of shear thickening can induce a dramatic change in suspension microstructure, such as particle aggregation, flow rate during pumping [39,40], and filling capacity of cement-based materials.

For both PCE and PNS HRWR types, mixtures exhibited shear-thickening behavior when used in combination with PC, except for 0.40 w/c mixtures. In the case of LHC, 0.30 w/c mixtures exhibited shear-thickening response while those made with higher w/c showed a shear-thinning behavior, regardless of HRWR type. The use of PC resulted in greater shear-thickening response compared to LHC, regardless of HRWR type. This may be due to a relatively higher HRWR adsorption with PC, in which the aluminate phases (C_3A and C_4AF) are higher compared to LHC [12]. The shear–thickening response observed with low w/c systems investigated in this study is likely to be a physical phenomena primarily due to the solid concentration of the system [39] and, secondly, to the type and dispersing mechanism of HRWR in use [26,27]. Indeed, lowering the solid concentration of the mixture reduced the shear-thickening response. The cement–HRWR combination seems to have also an impact on the shear-thickening response. The use of HRWR acting by steric effect exhibited a higher shear-thickening response than for HRWR acting by electrostatic effect, regardless of the type of cement. The use of fine silica fume particles reduced the shear-thickening response of dense grouts due to a bearing effect resulting from the polydispersed system.

The presence of polycarboxylate-ether chains in dense suspensions seems to cause disperse-phase aggregation instead of repulsion of particles, resulting in cluster formation. Indeed, as the solid concentration increases, the inter-particle distances decrease so that the polymers' molecules may not have sufficient space between cement particles, thus resulting in particle clusters enveloped by long polymers [42]. The formation of these clusters, composed of compact groups of particles, may be promoted when the shear forces are sufficient to drive particles into contact. The formation of clusters can also entrap more water otherwise used to lubricate the system, hence resulting in higher inter-particle friction causing greater viscosity of the system. This phenomenon may also occur in dense suspensions. At low velocities, the entrapped liquid is sufficient to fill the inter particle space and acts as a lubricant, and the flow is well ordered. At higher velocities, the liquid is, however, unable to fill the inter-particle space created between particles, and friction greatly increases, causing an increase in viscosity.

5. Conclusions

The effect of w/c on pseudoplastic responses of high-performance grout with different cement–HRWR combinations and silica fume was evaluated. Based on the above results, the following observations can be pointed out:

1. Low w/c grouts exhibited greater shear-thickening response compared to mixtures proportioned with higher w/c. On the other hand, grout mixtures proportioned with a w/c of 0.40 exhibited shear-thinning behavior, regardless of cement–HRWR combination.
2. The new generation of polycarboxylate HRWR acting by steric effect exhibited high shear-thickening response compared to those acting by electrostatic effect. This behavior is more pronounced for grouts made with portland cement and low w/c.
3. The use of portland cement resulted in greater shear-thickening response compared to low heat cement, regardless of the w/c and type of HRWR in use. This may be due to the higher degree of adsorption of HRWR that may occur with portland cement compared to low heat cement.
4. Grouts made with portland cement–PC HRWR combination led to substantial shear–thickening for w/c lower than 0.40 and shear-thickening for w/c equal 0.40. In the case of low heat cement, mixtures exhibited shear–thinning behavior, except for those made with 0.30 w/c.
5. For both cement types (PC and LHC), substitution of cement by 8% silica fume can decrease the degree of shear-thickening in grouts containing optimum dosages of PCE HRWR.

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