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The rheological properties of modified self-compacting cementitious paste

Farih Messaoudi [a], Hafida Marouf [b,*], Mohammed Sonebi [c], Abdelaziz Semcha [d]

[a] *Department Of Civil Engineering, Saad Dahlab University, Blida, 09000, Algeria.*

[b] *Department Of Civil Engineering, Belhadj Bouchaib Center University, Ain Temouchent, 46000, Algeria.*

[c] *School of Natural and Built Environment, Queens University of Belfast, Belfast, UK.*

[d] *Department Of Civil Engineering, Ahmed Draya University, Adrar, 01000, Algeria.*

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ABSTRACT

Self-compacting concrete (SCC) development marks an important step towards efficiency and conditions working in construction sites and in the precast industry. SCC easily flows into more complex shapes and through reinforcing bars, reduces the labor required for placement; no vibration is required to ensure proper concrete compaction. This concrete contains a high binder volume, which is controlled by their rheological behavior. The paste binders consists Portland cement (with or without additional cementations' materials), water, chemical additives and fillers. In this study, two tests series were carried out on self-compacting cement pastes made with marble waste additions as addition minerals. The first series of this survey was to determine the flow dough time of using a Marsh cone, the second series was to determine the same dough rheological parameters, namely the elastic limit and the plastic viscosity at using a rheometer. The results of this survey allowed us to study of the elastic limit evolution, the viscosity and the marsh flow time cone paste as a paste composition function.

1 Introduction

Self-compacting concrete (SCC) improves the efficiency and working conditions on construction sites and in the precast industry. It flows easily into complex shapes and through reinforcement bars, reduces the manpower required for the placement; no vibration is required to ensure correct compaction of concrete, also due to the self-leveling characteristics of SCC the need for screeding operations is reduced and costs can be minimize the casting time, machinery and fuel required to operate them [1-3]. The development of self-compacting concrete (SCC) and high performance concretes is specifically

* Corresponding author. Tel.: +213 549696297.
E-mail address: hafidamaarouf@yahoo.fr

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linked to progress in the field of adjuvant and the use of mineral additives [3] Several predictive models of the rheological behaviour of cement-based materials are developed [4-6]. The viscosity of cement grout was determined using a coaxial rotating cylinder viscometer Fann (smooth cylinders, no serration) that determined apparent viscosity at different shear rates [7, 8], these models require control of the constituents of the concrete properties, including the rheology of paste [9-11]. The rheological performance of fresh concrete mainly depends on the cement paste matrix when similar amounts of coarse and fine aggregates are used [12, 13]. Indeed, today's concrete are often composed of mineral additives, their presence affects the behaviour of cement pastes and therefore the rheological behaviour of concrete and all related properties, so it is necessary to study in their presence. The use of cement-based grouts with mineral admixtures can contribute to a technical feasible low cost solution having double benefit for the environment: reducing wastes and consumption of natural resources. Cement grouts were fluid mixtures which contain water, cement, and in some cases chemical admixtures, fine sand, and/or supplementary cementing materials. The complex rheological behaviour of cement based grout is non-Newtonian and thixotropic [3].

The rheology of cement paste is a factor of prime importance regarding the transporting, pumping, pouring and flowability of the material. Many studies on the rheology of cement grouts have shown that these materials are viscoplastic fluids presenting a yield stress which must be reached by the shear stress so that the flow took place [14, 15]. The yield stress is regarded as the material property that denotes the transition between solid like and fluid like behaviour. Consequently, it is the minimum stress that makes the fluid flow like a viscous material. Inter-particle forces between the solids in a suspension result in a yield stress that must be overcome to start the flow. In addition, an applied stress that is lower than the yield stress will be resulted in a deformation like a solid instead of flowing of paste [16].

Viscosity is considered to be the most important grouts property. Viscosity is the relationship between shear rate and the stress applied on the material. In the case of non-Newtonian behaviour, it is referred to as 'apparent viscosity' or 'shear rate dependent viscosity'. Cement grouts and cement based materials are subject to hydration process and the viscosity change accordingly the viscosity of the cement based materials. Thus, the rheological behaviour of cement based grouts changes with time.

The most use model is the Bingham model which is widely used owing to its simplicity and the linear relationship between shear stress and shear rate. However, the cement based materials rheological behaviour could be better explained by the Herschel-Bulkley model since it can explain the shear-thinning [17-19] or shear-thickening behaviour [20, 21], depending on many parameters such as solid concentration (water/cement ratio), interaction between particles (attractive due to the effect of viscosity agents or repulsive due to the effect of superplasticisers), size and shape of grains [22].

In all the cases, it can be described satisfactorily by the Herschel–Bulkley model [23] characterized by three parameters: yield stress $\tau 0$,consistency K and exponent b which relate, in the case of simple shear, the shear stress τ to the shear rate γ.

The use of mineral admixtures such as limestone powder or filler (LSP), fly ash, silica fume or ground granulated blast furnace slag (GGBS), etc affects the interaction between superplasticiser (SP) and cement. The experimental program presented in this paper was carried out with the purpose of studying and clarifying the effect of the waste mineral admixtures in the presence of polynaphthalene sulfonate superplasticizer (SP) type and viscosity agent (VA) on the rheological behaviour of SCC paste.

The main objective of this study was to evaluate the effect of the percentage of replacement of cement by waste of marble powder (mp), the water-to-binder ratio (w/(c+mp)), the dosages of superplasticiser (SP) and viscosity agent (VA) on the rheological properties and flow time. The rheological properties were measured with the Haake rheometer and flow time by Marsh cone. The correlations between the flow test results and the rheological parameters were developed in this study.

2 Materials and Experimental Procedures

2.1 Materials used

The cement used was a CEM II/A 32.5R manufactured by the Lafarge cement factory located in Algeria conforming to the NF EN 197-1 standards [24], having a specific gravity of 3.1 and a Blaine specific surface area of 4519 cm2/g. The mineralogical composition of cement for C_3S, C_2S, C_3A, and C_4AF was 58.8%, 18.1%, 9.1%, and 8.2%., respectively. Marble powder (mp) used mainly composed of calcite $CaCO_3$ with a higher content of 90% of economic interest because it is a powder after recovery of waste from a marble with a specific gravity of 2.65, and a Blaine specific surface area of

7312 cm2/g. The superplasticiser (SP) used was Medaplast SP40 based on polynaphthalene sulfonate (PNS) with a specific density of 1.2 and a solid content of 40%. The viscosity agent (VA) was Cimcil L25 in liquid solution and having density of 1.20.

2.2 Experimental Procedures

2.2.1 Rheometer test

The test used to measure the rheological parameters of the paste was Haake RheoStress 1 (Fig. 1). The principle of the test is to shear a sample of paste between two plates in horizontal surfaces, one was at rest and another one was mobile (rheometer plate-plate geometry). This rheometer was equipped with a valve rotor speed imposed. After several adjustments, the gap between the two plates was validated to 1.5 mm according to [25]. Tests were performed at 20°C (± 1°C). The flow curves were analysed and modelled by the software, Kaleida Graph (Version -4.03). The protocol followed in this investigation was as follows: presheared at 10 s-1 followed by a rest period of 2 min, then a linear ramp increasing rate from 0 to 70 s-1 was applied through the rheometer for 5 min .

Fig. 1 – Rheometer Haake Rheostress 1 test

2.2.2 Marsh cone test

The Marsh cone test measures the flow time of a given volume of a grout through a cone of a standard size (Fig. 2), is described in NF P18-452 standard [26].

Fig. 2 – Marsh cone test

The funnel Marsh cone used in this study has a capacity of 1000 ml and an internal orifice diameter of 4 mm. The time needed for a grout sample to flow through the cone is proportional to the viscosity of the grout. The flow time increases with an increase in viscosity. The flow time measurement is established by taking a representative sample of 1000 ml of grout, plugging the lower orifice of the cone. Once the grout passed through the orifice of the funnel, the time for flowing of grout was recorded.

3 Experimental Program

The various compositions tested are formulated constant mass of 500 g. Superplasticiser and viscosity agent are expressed in percentage of the mass of cement. The compositions of paste tested are summarized in the form of groups with in each case one or two components, which varied. The experimental program of six groups of pastes is summarised in Table 1.

Table 1 - Experimental Program

Group	w/(c+mp)	c/mp	SP (%)	VA (%)
1	0.40	1, 1.2, 1.4, 1.6, 1.8, 2	0.2, 0.4, 0.6, 0.8, 1, 1.2, 1.4, 1.6	0
2	0.45	1, 1.2, 1.4, 1.6, 1.8, 2	0.2, 0.4, 0.6, 0.8, 1, 1.2, 1.4	0
3	0.50	1, 1.2, 1.4, 1.6, 1.8, 2	0.2, 0.4, 0.6, 0.8, 1	0
4	0.40	2	0.2, 0.4, 0.6, 0.8, 1, 1.2, 1.4, 1.6	0.5, 1, 1.5, 2
5	0.45	2	0.2, 0.4, 0.6, 0.8, 1	0.5, 1, 1.5, 2
6	0.50	2	0.2, 0.4, 0.6, 0.8, 1	0.5, 1, 1.5, 2

Notations: **SP:** Superplasticiser; **VA:** Viscosity agent; **w:** Water; **c:** Cement; **mp:** Marble powder.

4 Results and Discussions

4.1 Marsh cone

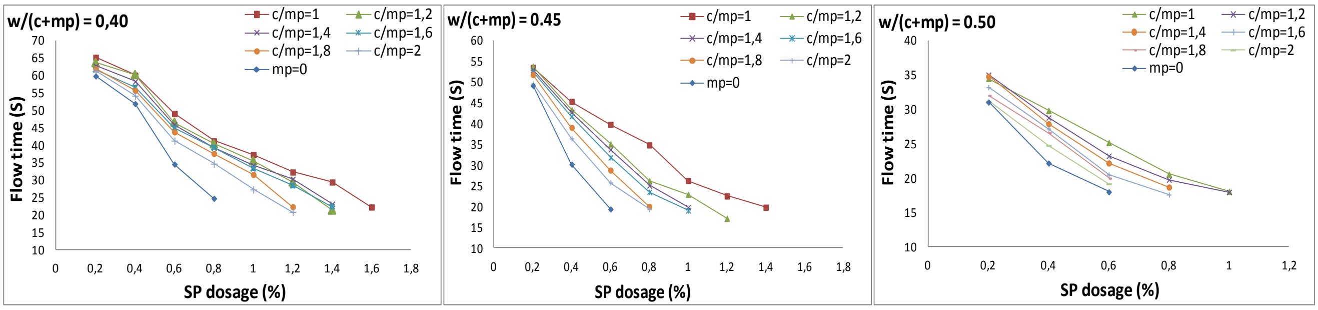
In this part, the analysis of the results led to investigate the effect of combined variables of the dosages of marble powder, SP and VA on the flowability of self-compacting pastes.

Figures (3a - 3b - 3c) present the results of the flow time values of Marsh cone of groups 1, 2 and 3 mixes made without viscosity agent in function of the variation of SP and c/mp ratio. As expected, the increase of dosage of SP resulted in a reduction of flow time. This was due to thicker adsorbed polymer layer of SP on the particles and consequently weaker van der Waals attraction between the particles. Therefore lower forces are needed to disperse the particles and lower cohesivity [27]. This can be attributed to better steric and electrostatic repulsions among cement particles that react with SP, leading to a better deflocculation of the particles in the paste and thus reducing the plastic viscosity.

As expected, the reduction of w/(c+mp) ratio led to ancrease in the flow time values. This can be attributed to less free water for lubrication of particles leading to a more friction between particles and therefore increasing the viscosity and reducing the flowability.

The flow time increased with the increase in the quantity of marble powder in the paste. Figure 4 shows the results of the variation of the flow time obtained for pastes made at the same ratio (w/(c+mp) = 0.40) and a dosage of SP = 1%. The increase of c/pm from 1 to 2 resulted in a reduction of the flow time. This can be attributed to high specific surface area of marble powder compared to the cement. An increase in the flow time can be explained by the significant need for water and the large specific surface of the marble powder [28, 7, 29].

For fixed dosage of SP at 1%, the flow time with a ratio of c/pm = 1 was decreased from 36.1s to 30.1 s for ratio of c/mp = 2 (cement equal to twice of marble powder). Thus, the substitution of the cement by the marble powder has an inverse effect on the fluidity of the paste.



(a) (b) (c)

Fig. 3 – Variation of the flow time versus SP and c/mp for pastes of groups 1, 2 and 3

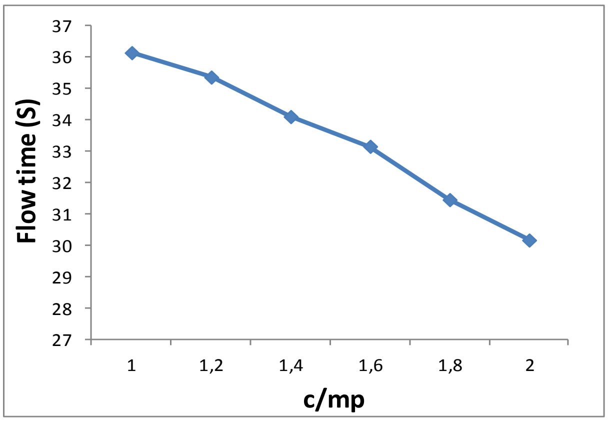
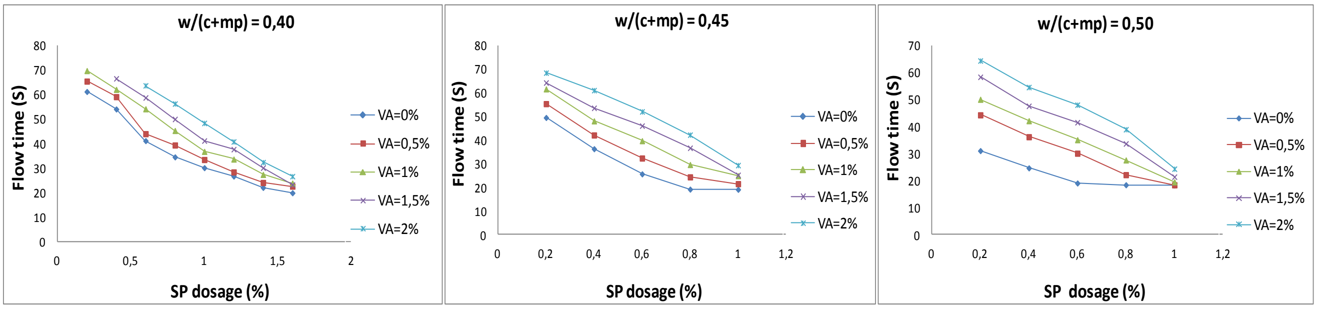


Fig. 4 – Variation of the flow time as a function of c/mp for a fixed SP 1%.

Figures (5a - 5b - 5c) show the results of flow times of groups 4, 5 and 6 (made with VA) with the variation of SP and VA, where the c/mp ratio was fixed at 2. Similarly in this case of group of mixes, the increase of SP led to a reduction in flow times for mixes containing VA. It can be observed that an increase in the flow time values as VA increased from 0 to 2%, regardless of the dosage of superplasticiser and w/(c+mp) ratio.



(a) (b) (c)

Fig. 5 – Variation of flow time versus SP and VA for groups 4, 5 and 6.

The influence of the dosage of viscosity agent on the flow time is presented in Figure 6 for the same ratio (w/(c+mp) = 0.45) and a fixed dosage of SP at 1% and c/mp = 2. There is significant variation in the flow time as a function of the dosage of the viscosity agent. It increases in the flow time of control mix (0% VA) by 12.5% and 48%, respectively when the dosage of VA increased to 0.5% and 2%.

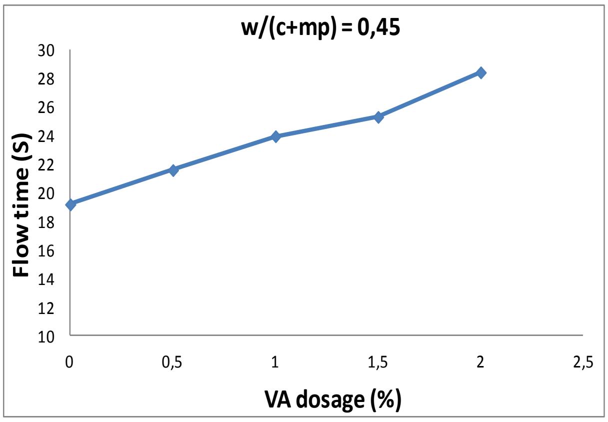


Fig. 6 – Variation of flow time versus VA and SP = 1%.

4.2 Rheological parameters

In this section we have studied the behaviour of the various self-compacting pastes by rheometer tested without and with the viscosity agent. For these cases, we studied the combined influence of the content of marble powder and SP on the yield stress and plastic viscosity.

The rheograms recorded by us tests Haake RheoStress 1 of the different paste followed a law of Herschel-Bulkley type that is expressed by equation (1). These results were consistent with what was reported in the literature [12, 30, 31].

$$\tau = \tau_0 + b(\gamma)^c \qquad (1)$$

Where: τ is the shear stress, τ_o the yield stress, γ represents the shear rate, b the consistency and c the fluidity index. Figure 7 shows an example of rheogram.

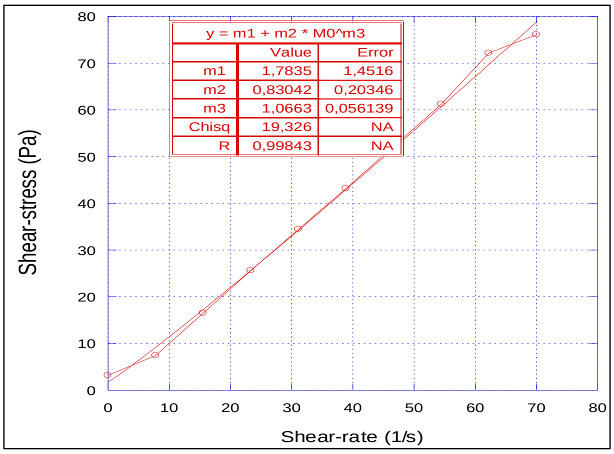
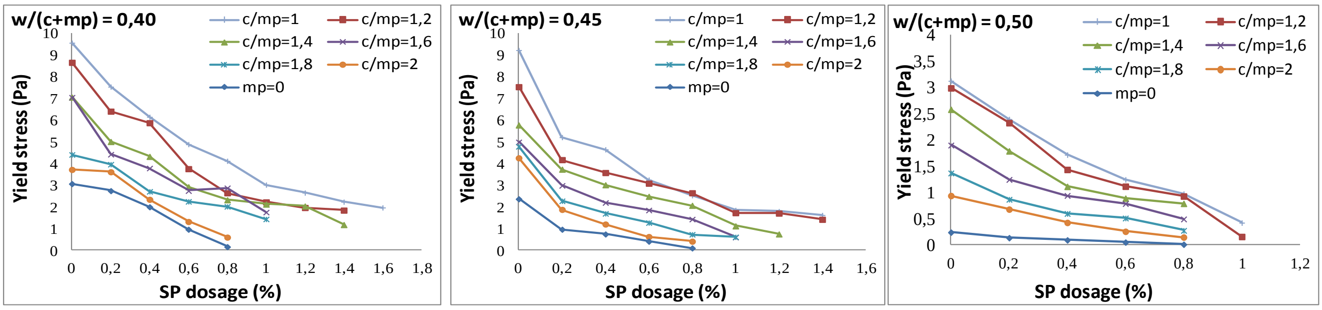


Fig. 7 – Example of rheogram obtain with rheometer

Figure 8 shows the variation of the yield stress in function of SP for the case of pastes of groups 1 and 2 and 3 made with w/(c+pm) = 0.40, 0.45 and 0.50, and the ratio of c/mp was varied from 0 to 2.

In these figures, it can be observed that the increase in SP dosage and w/(c+mp) led to a reduction in the yield stress values, while c/mp kept constant.

Depending on the mode of action of SP and SP-particle interaction, i.e., compatibility and affinity between the cement and SP, it coats the grains, especially fine particles (cement and fine addition) by adsorbing SP on the surface and make grains flocculate [32]. Meanwhile, the substitution of cement by marble powder (c/mp varied from 0 to 2) resulted in an increase of yield stress (Fig. 8).



(a) (b) (c)

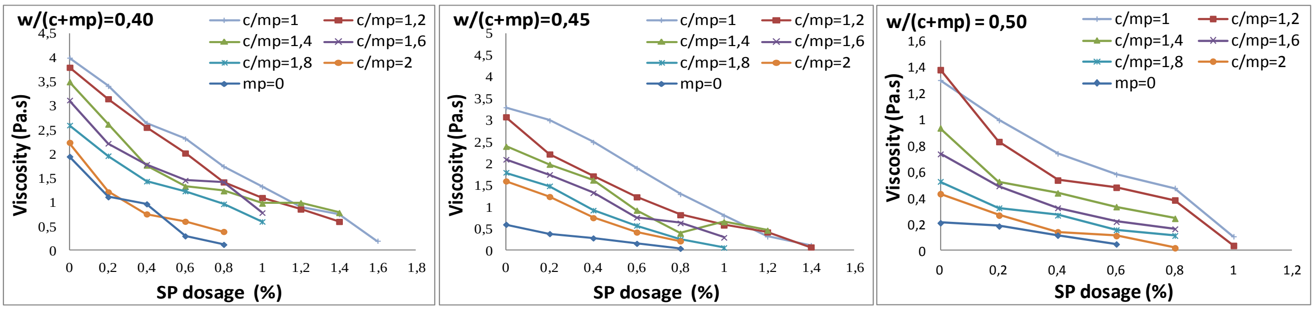
Fig. 8 – Variation of the yield stress versus SP for pastes of groups 1, 2 and 3.

In Figure 9, the combined influence of the content of marble powder and SP on the viscosity of the various self-compacting paste are plotted for groups 1, 2 and 3. Similarly to the results of yield stress, the increase of w/(c+mp) and SP led to a reduction in the plastic viscosity. In contrary, the substitution of cement by marble powder increased significantly the plastic viscosity.

Figure 10 shows the results of the yield stress depending on the variation of VA and SP, c/mp ratio is set at 2, the paste is formulated in a ratio w/(c+mp) equal to (0.40 for the group 4; 0.45 for group 5 and 0.50 for the group 6).

In Figure 10, it can observed that for these self-compacting pastes, the variation of yield stress values increased a slightly with the increase with increasing VA, while keeping all other parameters constant (w/(c+mp), c/mp and SP.

These results were consistent with the use of the viscosity agent which is often used to make the viscous materials without affecting too much their yield stress. The addition of SP at 0.4% lowered the yield stress for any dosage of VA, while w/(c+mp) kept fixed. Moreover, it also notes that the yield stress values decreased with increasing of (w/(c+mp).



(a) (b) (c)

Fig. 9 – Variation of plastic viscosity versus SP for cementitious pastes in groups 1, 2 and 3.

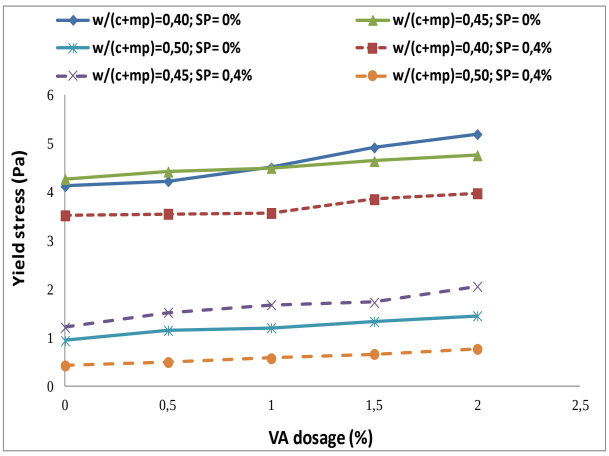


Fig. 10 – Variation of the yield stress versus VA and SP for cementitious pastes of groups 4, 5 and 6.

Figure 11 shows the results of the viscosity as a function of the variation of VA and SP, c/mp ratio was fixed at 2, the cementitious pastes was made with w/(c+mp) equal to (0.40 for the group 4 ; 0.45 for group 5 and 0.50 for group 6).

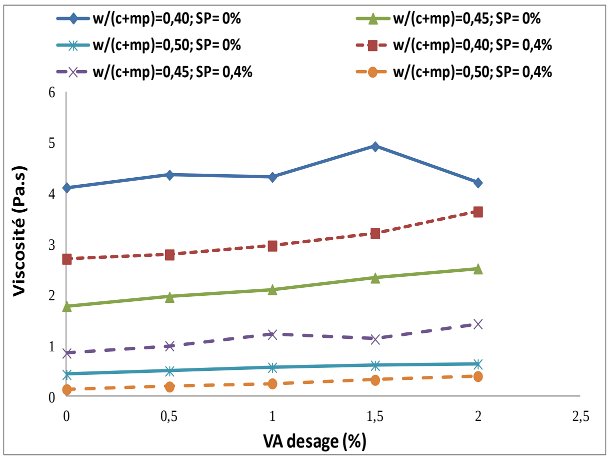


Fig. 11 – Variation in viscosity versus VA and SP for cementitious pastes of groups 4, 5 and 6.

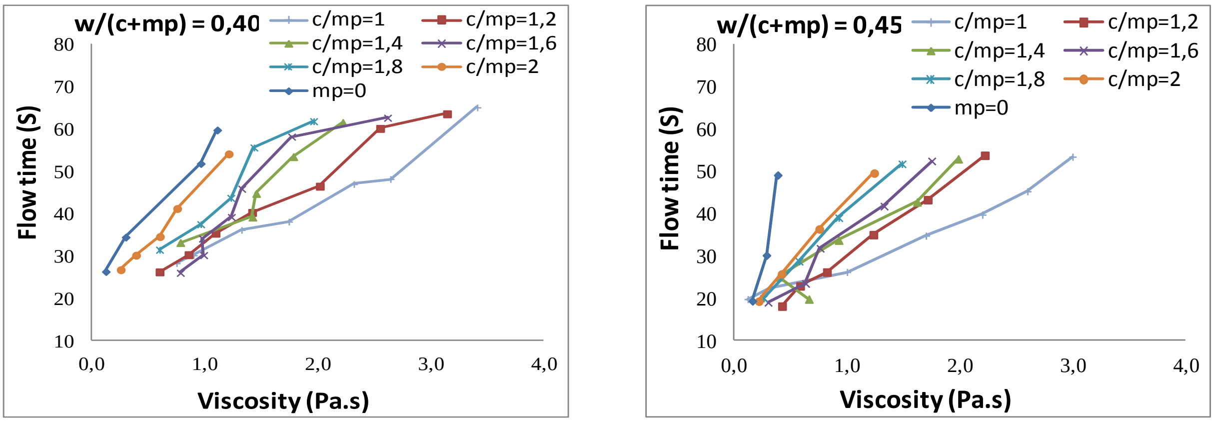
From these figures it can be seen that the values of plastic viscosity increased slightly with increasing of VA, when all other parameters (w/(c+mp), c/mp and SP) were kept constant. Adding 0.4% of SP resulted in a reduction of plastic viscosity for any w/(c+mp) and dosage of VA.

As with the yield stress, the values of plastic viscosity decreased with the increase in the ratio (w/(c+mp)). It can be noted that for paste made with w/(c+mp) = 0.5, the plastic viscosity values were very low.

For the effect of viscosity agent on the plastic viscosity, in case of self-compacting paste, it is slightly small compared to what is presupposed that, because the main role of viscosity agents is to bring the robustness to paste.

4.3 Correlation between flow time and viscosity

For groups 1 and 2 (without VA), all relationships between the results of flow times and the plastic viscosities are shown in Figure (12). These tests characterising grout rheology at high shear rate. For pastes made with the same ratio (w/(c+mp)), it can be noted that the flow time increased with the increase in the plastic viscosity. Additionally, the decrease ratio (c/mp) or with the increase of the quantity of marble powder led to an increase of both flow time and plastic viscosity. In general, the flow time correlated very well with the plastic viscosity [33].



(a) (b)

Fig. 12 – Correlation between the flow time and plastic viscosity to the pastes of the group 1 and 2.

The results shown in Figure 13 correspond to those of the correlations between the flow times and the plastic viscosities for pastes made with the same ratio (w/(c+mp) and c/mp = 2 in the presence of the viscosity agent.

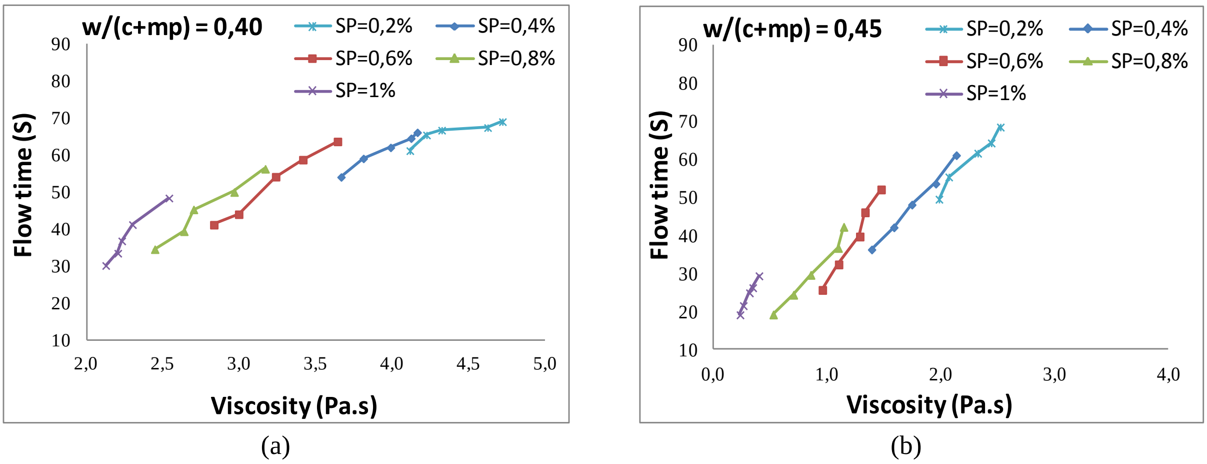


Fig. 13 – Correlation between the flow time and plastic viscosity to the pastes of the group 4 and 5.

For any w/(c+pm), similarly, the results in Figure 13 shows that the flow times increased with the increase in plastic viscosities. In this case of mixes, the dosage of VA was increased. The flow time and the plastic viscosity were reduced when the dosage of SP increased from 0.2 to 1%. Similarly to these groups of mixes, the flow time correlated very well with the plastic viscosity.

The increase of VA led to an increase in flow time, yield stress and plastic viscosity. This can be attributed to the entanglement and intertwining of VA polymer chains and the association of water between adjacent chains. At low shear, and especially at high concentrations, the intertwining of polymer chains can exhibit an increase in the apparent viscosity [8]. The factors that influence flow are described by Darcy's Law where the flow rate is inversely proportional to fluid viscosity. Therefore, VA increased the viscosity of the matrix and will improve the water retention.

An increase in SP content resulted in a reduction of the flow time, yield stress and plastic viscosity while the fluidity of grout is improved. This was due to thicker adsorbed polymer layer of SP on the particles and consequently weaker van der Waals attraction between the particles. Therefore lower forces are needed to disperse the particles and lower cohesivity [27]. This can be attributed to better steric and electrostatic repulsions among cement particles that react with SP, leading to a better deflocculation of the particles in the paste and thus reducing the plastic viscosity. The effects of VA and SP on paste correspond with the findings of other researchers [8, 33].

In case of an increase of marble powder proportion, while VA and SP were kept constant, it led to an increased flow time, yield stress, and plastic viscosity. This was due to the high surface area of marble powder (7312 vs. 4519 cm2/g for cement) resulting in a reduction of water needed for lubrification of paste.

5 Conclusion

The effects of the addition of waste of marble powder (mp), superplasticizer (SP), viscosity agent (VA), and water-to-binder ratio (w/(c+mp)) on the flowability, and rheological parameters were investigated.

Based on the results of this investigation, the following conclusions can be drawn:

The dosage of SP has a significant effect on the values of flow time, yield stress, and plastic viscosity. An increased dosage of SP led to a reduction in flowability, and the rheological parameters. This can be attributed of better steric and electrostatic repulsions between cement particles that react with SP in their surfaces.

The variation in the VA dosage showed moderate effect on the flow time, yield stress and plastic viscosity. This can be attributed to the entanglement and intertwining of VA polymer chains and the association of water between adjacent chains. VA also binds to the water phase of the paste matrix, which resulted in increased water retention. In fact, an increase in VA led to reduced flow time, yield stress, and plastic viscosity.

For given dosages of SP and VA, an increased percentage of marble powder (reduction of cement to marble powder ratio c/mp) led to an increase in flow time, yield stress, and plastic viscosity. It is due to the associated increase in the water demand induced by the addition of high surface area of marble powder.

As expected, the reduction of water-to-binder ratio (w/(c+mp) significantly increased the flow time, yield stress, and plastic viscosity.

Good relationships between flow time and plastic viscosity were found.

In the same water/powder ratio, doubling the amount of marble powder causes a considerable increase in the flow time.

Waste marble powder increased viscosity. It can be seen as an effect of increase cohesion.

In the perspective and to better control the behaviour of self-compacting paste treated with marble powder, it is hoped that other characteristics such as thermal conductivity, shrinkage and permeability can be studied in future work.

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