RHEOLOGICAL PROPERTIES OF 3D PRINTING CONCRTE CONTAINING SISAL FIBRES

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Abstract

The aim of this study is to investigate the effect of different parameter of mix composition on fresh and rheological properties of printable mortar. Different binders were tested such as limestone filler (LS), and fly ash (FA). The water/binder ratio of 0.50 was used in this investigation. Superplasticiser (SP), viscosity modifying admixture (VMA) and sisal natural fibres (NF) were used in the investigation at different percentage. Furthermore, positive effect of fibres can be reversed when the dosage in fibres is too high (difficulties to extrude and drainage phenomenon which led to a stiffening of the mix). VMA caused an important loss of workability and time gap which influenced the passing ability of the mix through the extruder. Adding sisal fibres led to a greater yield stress, higher cohesiveness of the mix. Increase SP led to an increase in slump flow and penetration values and reduced the yield stress which can affect the stability of the shape of printed layers.

Keywords:

3D Printing; Chemical admixtures; Extrudability; Penetration; Sisal Fibres; Slump flow; Yield stress

1 INTRODUCTION

3D printing methods using polymeric materials was developed since the 90's, this additive manufacturing is on rise nowadays. As formworks represent 35 to 60 % of the global cost of a concrete construction, this innovation embodies an important financial benefit, in addition to improving the construction rate and architectural liberties. Different processes have been invented so far whose well-know are Contour Crafting, concrete Printing and D-shape, respectively invented in the USA, UK, and Italy [khoshenevis et al., 2006, Lim et al. 2012].

The D-shape method, also called the Binder Jetting, consists in squirting a binder over a powder placed in an adequate mould. Cement can act as the powder and water or other liquid as binder. Another alternative is to inject the cement paste as a binder to aggregates playing the powder role. The architect Enrico Dini has created different 3D printed objects using this method. Yet, the binder process restricts the layer height, the duration of the printing and the level of details of the structure. Exposure to humidity of the powder has to be taken in consideration as well [Wangler et al.; 2016].

Another well-known construction-scale 3D printing method has been developed by researchers at Loughborough University and is called "Concrete printing": 3D printing by layered extrusion. Indeed, once the adequate concrete mix design completed, layers of this cement paste are placed with a printing nozzle, on top of each other, without needing any formwork or vibration compacting [Le et al.; 2012].

Layers were extruded with a simple modified joint gun, rheological and fresh properties of different mix design were evaluated. Concerning binder's composition, it has been concluded that adding 24% of fly ash (FA), 8% of silica fume (SF) and using natural fibres from 0.2 to 0.6% (NF) results in great printability properties, improvement in rheological and fresh performances and in bleeding and segregation resistance. Plus, the addition of a Nanoclay-based viscosity modifying agent (VMA), Actigel, has proved to be efficient in developing 3D printing essential characteristics [Rubio et al.; 2017].

The aim of the present experimental work was to analyse the effect of fly ash (FA), limestone powder (LS), percentage of natural sisal fibres (NF), the dosage of superplasticiser (SP) and viscosity modifying admixture (VMA) on the rheological behaviour and fresh properties of a printable mortar. The rheological parameters were evaluated by the static yield stress using the cylindrical slump test.

2 MATERIAL AND MIX DESIGN

Fly ash and limestone powder (LS) were used as binder with cement. Portland Cement CEM I 52,5N corresponded to the BS EN 197-1:2000. Fly Ash, conform to BS EN 450-1:2005, came from Scot ash Ltd. This material had a surface gravity of 2.21 and a % passing 45 µm sieve of 85. Limestone powder is provided by Omya Company and had a specific gravity of 2.75, loss of ignition of 41.42 and % passing 45 µm.

Each mix contained 1.18 mm sieved sand and respected a sand-to-binder ratio of about 2 and a water-to-binder ratio of 0.50. The temperature of the water has been kept around 16°C for all the batches.

Superplasticiser (SP) and viscosity modifying agent (VMA) have been used at different dosages for a given binder. SP was a polycarboxylate polymer solution having a specific gravity of 1.07 g/ml. SP has been employed from 0.03 to 0.6 % of binder. Purified palygorskite nano-clay (PPNC) is used as a mineral VMA to control stability and flow of the fresh state. The Acti-Gel 208® used is a highly purified magnesium aluminium silicate that is self-dispersing and has been used at 0.8 % of binder.

Besides, sisal fibres (NF) were 15 mm long with a specific gravity of 1.52 g/cm^3. These fibres contained water at a dosage of 7.63 %. NF has been added to the mix design at different dosages: 3.6 (NF1) or 6.0 (NF2) (kg/m^3).

3 MIXING AND TEST METHODS

3.1 Mixing procedure

It has been demonstrated that cement-based materials behaviour is strongly time-dependant. This is why strict procedures must be developed in order to make the time parameter constant throughout all the different mortar compositions, to allow comparisons.

The mixing procedure required the use of a 2.5 L capacity mixer. To avoid materials to go over the mixing bowl while the mixer is on, different volumes of cement paste have been calculated up to 2.2 L. This blender allows two different speeds, which will be referred to later on by low (140 rpm) or high speed (285 rpm). First of all, dry materials are positioned in the mixer: binders as cement and fly ash, and sand. In order to obtain a good particles repartition, the blender is turned on for 30 seconds, on the first speed (low).

Secondly, water mixed with superplasticiser is inserted into the mixing bowl: this is when the time counting starts (t = 0). Binders, sand, water and SP are then mixed for 30 seconds at low speed.

Finally, natural fibres and VMA in some cases are added to the mix and blended for 30 seconds at low speed.

To ensure good repartition of fibres and a proper cohesion of materials, the mix is blended for 2 minutes at high speed. Finally, the mixture is blended a last time at low speed for 1 minute for the mixture to settle. At this stage, the mortar is tested for rheological and fresh characterisation, as described in the test methods section.

3.2 Testing methods

Two simple tests have been performed on every single mortar mixture to define the fresh properties of mortar: the penetrometer test and the flow table test.

In order to describe pastes rheological properties, a semi-empirical test, slump test, have been employed.

Indeed, right after mixing, the first test is the penetrometer test, followed by the flow table test and then by the slump test. The time is counting as well: each test must last for 3 minutes ± 1 minute. After that, the density of the mix is measured. It is important to note that the paste is mixed by hand during 15 seconds before each test and the density measurement.

The standard BS ISO 13765-1:2004 characterises the use of the cone penetrometer. In fact, this test aims to determine the workability and the consistency of the mortar under a certain stress caused by the dropping cone (only under gravity). Firstly, the cement paste is introduced in the conical mould in two layers. In order to have a homogeneous filling and to avoid voids in the moulds, each layer is compacted with ten short strokes. The excess of mortar is removed using a trowel or a spatula and the mould is placed under the dropping cone. Then, the testing machine is adjusted so the cone just touches the surface of the sample. Finally, the button is pressed for 5 seconds and the cone is released: it penetrates into the mortar sample under its own weight. The value read on the dial is noted: this penetrometer allows a precision of 0,01 mm. The cone is then dropped 3 more times in order to have more values and so the mean value will be more precise.

The flow table test was use to measure the stiff mix consistency by taking the spread of the mortar under a certain type of stress (jolting; BS EN 1015-3:1999). The first step is to place the conical mould centrally on the flow table. The mortar is then introduced in two layers; each layer is compacted by ten strokes. The mould must be maintained properly when filling it. The excess of mortar is then removed. After 15 seconds, the mould is slowly and vertically lifted: two values of both diameter of the slump (two perpendicular directions) and height are measured. Finally, the sample is spread out on the flow table by jolting: 15 jolting in 15 seconds are required. Afterwards, measurements are taken another time: two perpendicular diameters of the spread.

The third test is quiet similar in execution then the previous one: the slump test. Indeed, the procedure followed for this test is similar to the flow table test, previously exposed: the cylindrical mould (Fig. 1) is centrally placed onto the flow table; the mortar is introduced in two 10-strokes-compacted layers into the mould (firmly maintained); the excess of mortar is removed to have a plain surface and the mould is lifted after 15 seconds; finally, two values of both heights and diameters are written down in order to have more precise averages. However, this semi-empirical test allows estimating the static yield stress of the mix depending on the ratio height/spread. Three different flow regimes exist with an important stress tensor difference: when H >> R, H ≈ R and H << R – where H is the height and R is the radius after the mould's lift, as in Fig. 1 [Coussot et al., 2005].

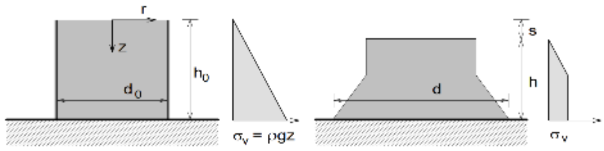

Fig. 1: Notations for estimating the yield stress with the slump test

After these previous tests, the density is taken in a cylindrical mould filled with two layers of mortar, each of them compacted by 10 strokes.

Moreover, in order to describe the workability of the different mix design with time, the opening time procedure has been led. This implies to realize, right after mixing, the penetration test, the flow table and the slump test through time, every 15 minutes for about 2 hours at least. All the compositions and the results are presented in Table 1.

4 RESULTS AND DISCUSSION

4.1 Effect of fly ash and limestone powder

Two compositions of mortars were analysed to evaluate FA effect on fresh and rheological properties: the first one corresponded to the reference (100% of binder was cement) and the second binder was made of 24% of FA of replacement of cement. Results are plotted in Figure 2.

Fig. 2 indicated a slight reduction of the flow table spread when FA was used (from 206 to 204 mm). Similarly, the penetration of mix containing FA was lower than reference mix.

Concerning rheological behaviour, the addition of FA also affected the estimated yield stress similarly as the slump flow. It could be observed that when the flow table spread increased, the yield stress decreased. Adding FA lead a slight increased of yield stress from 1718 to 1721 Pa. This phenomenon was coherent regarding cohesion improvement and segregation decrease when adding FA [Sonebi 2002].

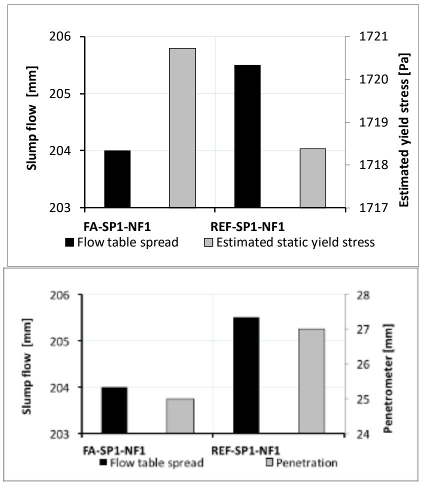

Fig. 2: Influence of FA on flow table spread and estimated yield stress

Moreover, from an extrudability point of view, it has been observed that buildability of layers after printing was better with FA compared to reference mix.

The fact that the bleeding was reduced when FA was included in the composition could be explained by the particles size and shape. Indeed, FA surface area equals 420 m²/kg whereas cement's was 370 m²/kg: consequently, more water was needed to cover the particles' surface. Also, cohesiveness and stability were respectively improved and reduced by replacing a portion of cement by FA, due to the ball bearing effect of FA particles. Indeed, FA was known as a flow-enhancing additive because its spherical particle shape acted to decrease rheological parameters (yield stress and plastic viscosity). However, increasing FA content may lead to reduction of workability because of the absorption of SP [Sonebi 2006].

It has been shown that replacing cement by LS influenced the fresh and rheological properties in a contrary way than when adding FA. In fact, with the substitution of cement by LS, for the same SP dosage, the flow table spread increased from 206 to 229 mm and the penetration from 25 to 39 mm. The estimated rheological parameter was also affected by reduction from 1718 to 1243 Pa.

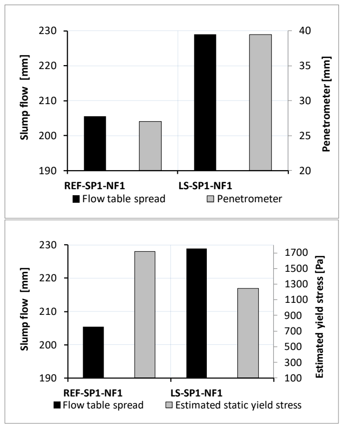

Fig. 3: Influence of LS on penetration, flow table spread and estimated yield stress

Concerning the extrusion, mix containing LS has been extruded but the buildability of the layers was not suitable, lower layer didn't sustain the upper ones. As Santos et al. (2017) reported that LS was often used when a fluid cement paste was required. In same case, LS could be the cause of segregation into the cement paste matrix [Santos et al.; 2017].

Consequently, it has been demonstrated that binder only made with cement and limestone powder was not suitable to be applied in 3D printing processes with mortar.

4.2 Effect of SP and VMA

The effect of dosage of SP on slump flow, penetration and estimated yield stress is illustrated in Fig. 4.

Results have demonstrated that concerning FA- mixes, flow table spread, penetration and estimated yield stress respectively changed from 204 to 205 mm, from 27 to 29 mm and from 1721 to 1603 Pa with the addition of SP. Slight changes were observed for slump flow and penetration. However, estimated yield stress was significantly reduced by adding more SP.

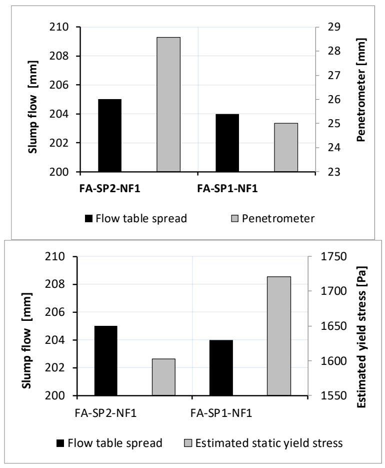

Fig.4: Influence of SP on penetration, slump flow and estimated yield stress

Workability, consistency and printability were highly influenced by increasing SP because of its impact on the inter-particles' matrix by steric repulsion of cement particles [Schmidt 2013].

Tab. 1: Compositions and fresh and rheological properties

COMPOSITION [kg/m³]									Penetrometer	Flow table spread	Estimated static yield stress
	Cement	**Water**	**Sand**	**FA**	**LS**	**SP**	**NF**	**VMA**	[mm]	[mm]	[Pa]
REF-SP1-NF1	618	311	1232	-	-	0.136	3.6	-	25	206	1718
FA-SP1-NF1	463	311	1195	144	-	0.136	3.6	-	27	204	1721
FA-SP2-NF1	463	311	1195	144	-	0.272	3.6	-	29	205	1603
FA-SP1-NF2	463	311	1195	144	-	0.136	6.0	-	24	195	1761
LS-SP1-NF1	463	311	1195	-	144	0.136	3.6	-	39	229	1243
FA-SP1-NF1-VMA	463	311	1195	144	-	0.136	3,6	5	25	199	1763
FA-SP1-NF2-VMA	463	311	1195	144	-	0.136	6.0	5	19	193	1767

The effect of VMA on the fresh and rheological properties has been studied at different fibres' amounts (NF1 and NF2) and results are presented in Figs. 5 and 6. Fig. 5 has shown that the addition of VMA for a given SP and fibres dosages led to a reduction of flow table spread and penetration and an increase in the estimated yield stress. For NF1 mixes, slump flow, penetration and yield stress varied respectively from 204 to 199 mm, 27 to 25 mm and 1721 to 1763 Pa with the addition of VMA. Concerning NF2 mixes, the same properties respectively changed from 195 to 193 mm, f 24 to 19 mm and 1761 to 1767 Pa. Visually, extrudability of VMA mixes was easier because of higher viscosity of the material and layers had good stability and buildability. It was slightly represented by Fig. 6.

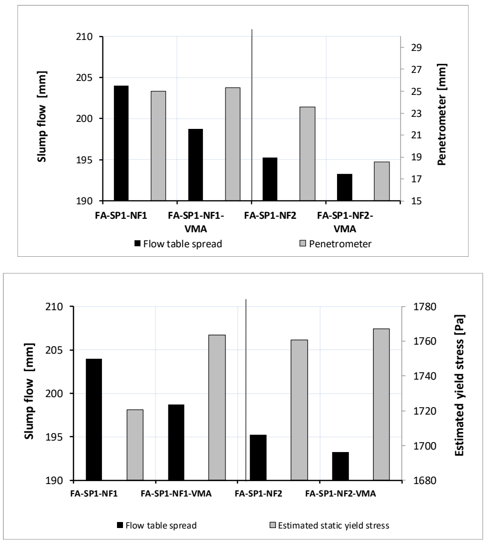

Fig. 5. Influence of VMA on rheological properties

Particle size of nano-clay based VMA was finer than FA and cement particles: binder and VMA particles were intermingled and more particles contact created a high shear resistance. The material matrix gets higher cohesiveness and water to binder ratio needed was higher. This variation of water demand explained the higher stability and buildability of mixes with VMA. Plus, Cirgel VMA used here corresponded to a Nano-clay based VMA pre-dispersed in water: this enhanced the effectiveness of the viscosity agent. Generally, VMA was often used to improve segregation and bleeding resistance and to influence flow properties and rheological behaviour of cement-based materials. This was confirmed by other authors [Leemann et al. 2007; Sonebi 2006, Sonebi 2010, Schmidt et al. 2013] therefore, using VMA was appropriate to 3D printing with cement-based materials.

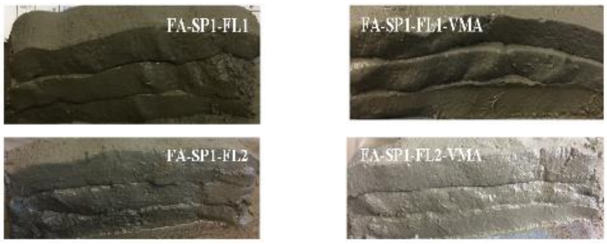

Fig.6: Influence of VMA on extrusion and visual observation

4.3 Effect of fibres

The effect of NF fibres content was evaluated for given FA, SP and VMA dosages. Results are plotted in Fig. 7. With the addition of NF fibres (from 0.6 to 1.0 % of binder), both flow table spread and penetration values were decreased and the estimated yield stress increased. For FA-SP1-NF1 or NF2, these 3 parameters respectively varied from 204 to 195 mm, from 27 to 24 mm and from 1721 to 1761 Pa with the increasing amount of fibres. The same trend was observed with mixes including VMA. The results are respectively named here after: from 199 to 193 mm, from 25 to 19 mm and from 1763 to 1767 Pa.

Concerning the extrudability and buildability, both mixes with NF1 or NF2 had great extrusion and sustainability properties. Thus, the previous results highlighted that increasing fibres content influenced the workability and reduced the flow of the material by improving its cohesiveness. Adding fibres could be assimilated to increasing the volume of fibres: the more fibres were added, the more connected the matrix was and a filtering process could begin. Improvement of fresh and rheological properties, as previously presented, proved that the network created by sisal fibres was more efficient for NF2 than NF1, thus with increasing amount of fibres [Peled et al. 2003].

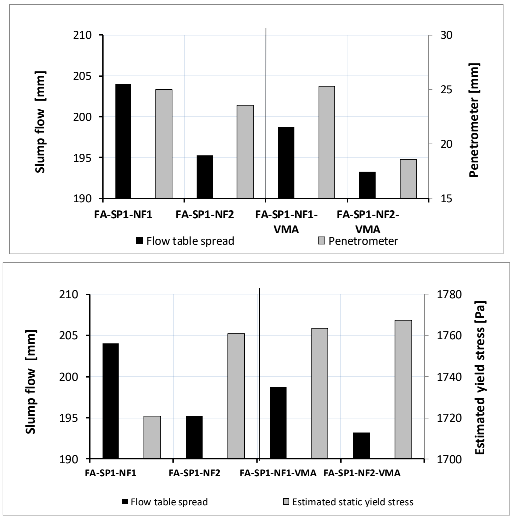

Fig. 7: Influence of NF dosage on fresh and rheological properties

4.4 Opening Time

As previously mentioned the opening time generally refers to the evolution of workability with time and corresponds to a certain period of time when the material gets acceptable extrudability and buildability properties. A proper average in time have to be found: long enough for the material to be maintain at a constant flow and short enough to avoid inter-layer bonding and segregation [Le et al.; 2012].

Also called Time Gap, this time period has been measured in the present experimental work using the same 3 fresh and rheological tests (penetrometer, flow table and cylindrical slump test) and the estimation of yield stress, every 15 minutes, for 2 hours. The open time of cement-based material was linked to its setting time. Usually, in 3D printing with cement-based materials, open time is defined by the period in which the material is still extrudable.

To examine the effects of FA with time, two mixes were compared: a reference mix – with 100% cement binder – and 24% replacement of cement binder by FA. Results for slump flow and estimated yield stress with time were plotted in Fig. 8.

Using FA has been proved to make flow table spread and penetration decrease and yield stress increased. These results have shown that FA impacts remained with time because in a 2 hours' time period, the estimation of yield stress for FA-SP1-NF1 remained higher than the estimation from REF-SP1-NF1. The flow table and penetrometer results followed the same trend as well: the reference mix values were always higher than the mix with 24% binder replacement by FA. Indeed, the pozzolanic behaviour of FA and its fine particles' size led to improvement of fresh and rheological properties of mixes.

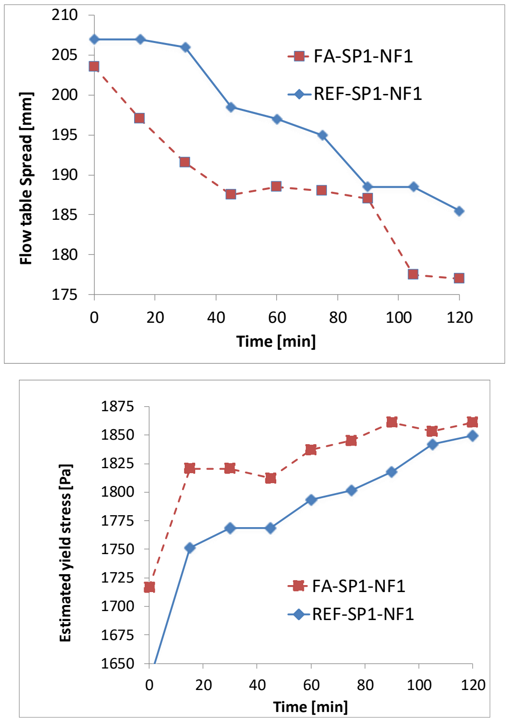

Fig.8: Influence of FA on flow table spread and estimated yield stress vs. time

Even after 2 hours, the extrusion of both mixes with the air-pressure gun remained possible with good extrudability and buildability. However, the reference mix was harder to extrude because it was becoming dry. Some layers extruded after 2 hours with the REF mix had a perfect visual buildability but it seemed they were reaching the limit of workability required to be extrudable, i.e. too dry whose first consequence was drainage.

Consequently, FA improved the extrusion of materials in time and on a long period of time and so helped maintaining mortars' consistency and workability [Rubio et al. 2017].
The effect of limestone powder on the opening time is illustrated in Fig. 9.

As expected, LS influenced slump flow, penetration and yield stress in time by increasing fresh properties and decreasing rheological properties. Indeed, for fresh properties (slump flow and penetration), higher values were obtained with FA- mixes and lower values were obtained for the rheological parameter, the yield stress.

However, about the estimation of yield stress could be noticed a slight diminution of the yield stress difference between FA- and LS- mixes, as they tend to get closer in values. Yield stress of LS was increased significantly from 0 to 120 min (from 1250 to 1750 Pa). Both FA and LS results were similar between 100 and 120 minutes. Even if limestone powder is considered as inert, it has significant effects on particles packing, water demand and hydration [Svermova et al. 2002].

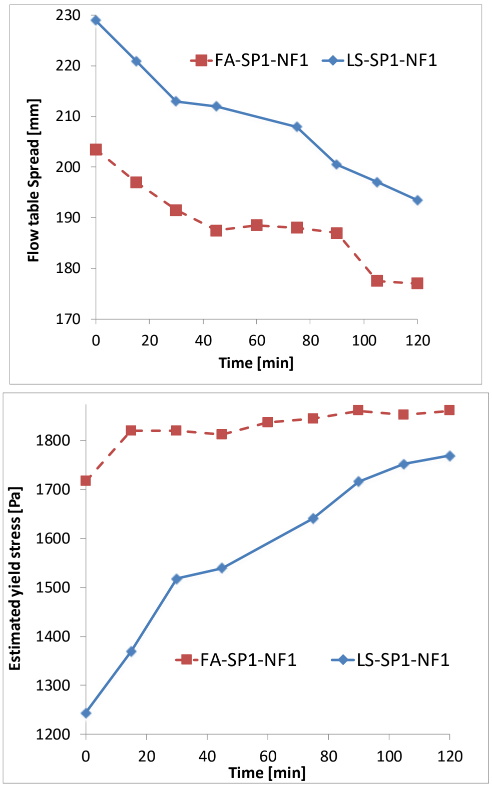

Fig. 9: Influence of LS on slump flow, estimated yield stress

Concerning the extrusion at the end of the opening time (t = 120 min) with both mixes, it has been visually observed that shapes and sustainability of layers were quiet similar. Indeed, it was in this period of time that both yield stress tends to approach the same value due to reduction of setting time when using LS as cement replacement.

Effects of VMA on opening time have been studied and results are plotted in Fig. 10. Results with the addition of nano-clay based VMA have shown improvements of flow table spreads and penetration. Indeed, with time this VMA solution increased the cohesiveness of mortar and thus binder and VMA particles were intermingled and more particles contact created a higher shear resistance. Plus, Cirgel VMA used here corresponded to a nano-clay based VMA pre-dispersed in water: this led to an enhancement of the effectiveness of the viscosity agent. Indeed, the estimated yield stress was expected to increase with time and with the addition of VMA. Plus, the flocculation strength of the suspension was improved and so was the structural stability of the mortar. Consequently, adding Cirgel into the mix design combined with the built up rate effect and thus reduced the opening time [Leemann et al. 2007].

Four mixes have been compared using NF fibres to evaluate the effect of fibres' amount on mortar's fresh and rheological properties with time. Both comparisons of FA-SPA-NF1/NF2 and FA-SP1-NF1/NF2-VMA followed the same trends in the evolution of their fresh and rheological parameters (Fig. 11).

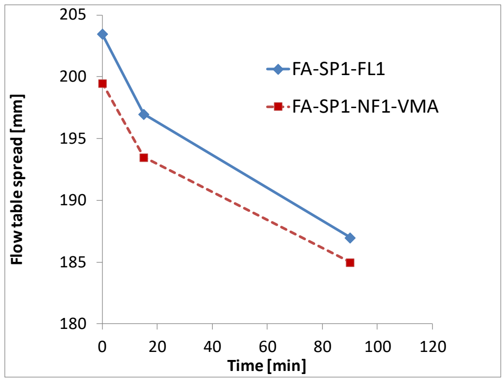

Fig.10: Influence of VMA on flow table spread with time

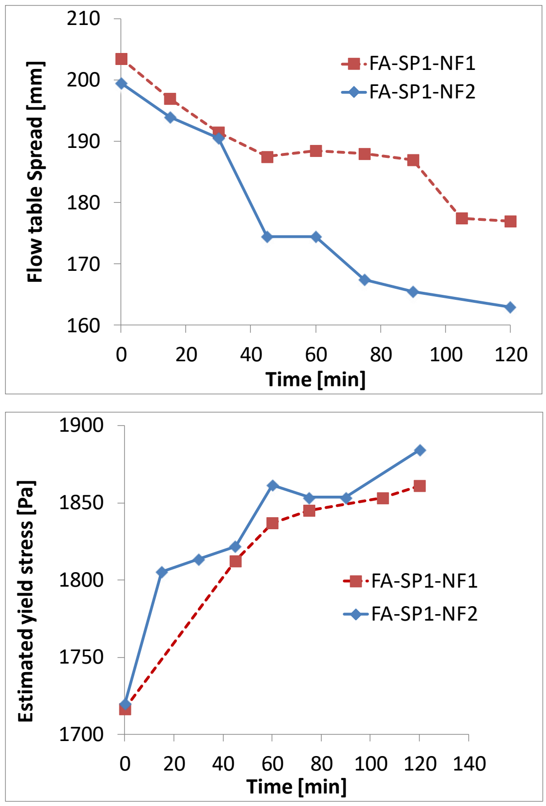

Fig. 11: Influence of NF fibres' amount on opening time flow table spread and estimated yield stress

Results have highlighted that with time, both slump flow and penetration values decreased with higher NF fibres content and yield stress increased with this addition. Higher NF fibres amounts led to the formation of a denser fibres network. Therefore, estimation of yield stress over 2 hours highlighted a slight decrease with fewer fibres and the portable vane results have confirmed this evolution. Moreover, the portable vane calculations of yield stress have also pointed out the acceleration of the setting time within the last hour of testing by a significant improvement of the rheological parameter for NF2 content. High packing density added to the build-up rate of cement-based mortars implied a consequent loss in workability and acceleration of the setting time. Thus, adding NF fibres in higher rate could lead to drainage and stiffening of the paste [Martinie et al.; 2010].

About the extrudability of these mixes, the addition of fibres has resulted in more stability of the layers under upper-layer's weight and regular shapes of extruded material.

5 CONCLUSIONS

The addition of 24% of fly ash (FA) increased slightly yield stress, cohesiveness, structure homogeneity and stability appeared to be an advantage to print layers. Indeed it reduced the fluidity of flow table, bleeding and the segregation of layers. Additionally, it improves the pass ability through the extruder and shows a greater resistance to penetration. A decrease of the time gap can be noticed.

Concerning binder's compositions, it can be concluded that replacement of cement by 24% of limestone powder was not appropriate for printable mortar because it increased flow table spread and penetration, and reduced the yield stress.

Increasing SP led to an increase in workability and a reduction in yield stress decreased. Although, SP improved workability and passing ability through the extruding nozzle, buildability of layers was significantly and badly affected by SP and high amount of SP allowed bleeding (risk of drainage), segregation and shrinkage cracking at early age.

The addition of VMA allowed the mix to gain in cohesiveness, stability and buildability. It has also improved segregation and bleeding resistance and improved fresh and rheological properties. Interested results were obtained with mixes using VMA and have been proved this admixture to be efficient in the 3D printing field.

The addition of sisal fibres (NF) from 0.6 to 1% of binder, reduced the flow of the mortar by improving its cohesiveness with a denser network of fibres. Indeed, with the addition of higher amounts of NF, the yield stress increased and the fresh properties decreased.

Appropriate workability, good segregation and bleeding resistance, easy extrudability and great printability and buildability were required in order to apply 3D printing processes to cement-based materials. Indeed, extruded layers must maintain it-self and might not deform under supplementary layers' load. It also means that layers must bond together to avoid voids, cold joints and other weakening issues.

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