FRESH AND RHEOLOGICAL PROPERTIES OF 3D PRINTING BIO-CEMENT-BASED MATERIALS

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Abstract

The aim of this study is to investigate the effect of different mix composition on fresh and rheological properties of a bio-printable mortar. Different binders were tested such as fly ash (FA), silica fume (SF), and used with CEMI and sand for a water/binder ratio of 0.50. Then, superplasticizer (SP) and natural flax fibres 15 mm (FL) were used in the investigation at varying percentage for a given binder. Two viscosity modifying agents were also tested. Adding flax fibres (0.2% to 0.6%) led to a greater yield stress, higher cohesiveness of the mix. It also demonstrated more stable structure of layers which allowed to not collapse under layer own weight and other successive layers. With the incorporation of fibres the time gap is reduced. 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. Increase SP led to an increase in slump flow and penetration values which can affect the stability of the shape of printed layers. Good linear correlation between slump flow and penetrometer was established.

Keywords:

3D printing, flax fibres, opening time, penetration, slump flow, yield stress

1 INTRODUCTION

Contour Crafting is currently the most effective method researchers have found so far. The concrete layers are poured out by robotic machines and can be used for small-scale industrial parts and building structures [Malaeb 2015].

Inspired by the 3D printing with polymer materials, 3D printing opens new horizons to cement-based materials of the construction industry. As formworks represent 35-60% of the overall costs of concrete structures, it would be an important financial advantage to use this new technology. Besides, 3D printing in building structures without formworks permits to improve the construction rate and open to new architectural liberties. Replacing humans work by additive technology automated allows building in polluted zones or on planets as reported in the project of contour crafting with the construction of bases on the moon for the NASA [P.3D 2015]. Several applications were reported on applications and prototypes of 3D printing [Khoshenevis 2006] [Le 2012] [Alec 2016] [Scott 2016] [Alec 2016] [Lloret 2015].

The two previous pioneers of 3D printing concrete as companies were the Shanghai Company, WinSun [Wangler 2016]. It was reported that to 10 full scale houses have been printed in less than 24 hours with 3D printing concrete. The method is appealing by the low cost of each house, around 5000$ [Malaeb 2015]. The largest 3D printer has been created by the World's Advanced Saving Project launched in 2010 by the Italian Centro Sviluppo Progetti Company. It was 12 meters tall in a hexagonal shape [Wangler 2016].

Finally, HuaShang Tengda Company (China) has recently managed to incorporate reinforcements to its 3D printed concrete structure. In fact, they have printed a 400 m² villa around iron frameworks by using an original nozzle design. They first raised the complete steel bars and pipes complex of the house. Then, concrete has been printed over it with big scale 3D printer [Scott 2016].

The printability of fresh cement based materials is the ability of the layer to maintain itself and the weight of the layers subsequently deposited. The time gap between two deposited layers must be sufficiently long to provide adequate mechanical strength capable of sustaining the weight of the subsequently deposited layers and also short enough to provide an optimized bond strength and building rate. It appears that it's the shortest time gap which allows the stability of the structure during construction. The ability of the first layer to support itself and others layers is link to its rheology and more precisely to its yield stress [Perrot 2015].

The aim of this study is to evaluate the effect of different mix composition parameters included dosage of superplasticizer (SP), addition of fly ash (FA) and silica fume (SF), the dosage of flax fibres (FL) and type of viscosity modifying agent (VMA) on fresh and rheological properties of a printable mortar.

2 EXPERIMENTAL PROGRAMME

Fresh properties of printed cement-based mortar compose of fly ash and silica fume, incorporating fibers (FL), VMAs (diutan gums, nano-clay) and superplasticizer were investigated. The following properties of the mortars were examined: the slump flow, the penetration test, the rheological properties (yield value), and the extruded property. Moreover, the relationships between the fresh properties were evaluated.

2.1 Materials

The mortars were prepared with a fixed water-to-binder ratio (w/b) of 0.50. Portland cement CEMI 42.5N specified by BS EN 197-1: 2000 (specific gravity of 3.13) was used in all mixes. Fly ash from Scot ash ltd was added and conformed to BS EN 450-1:2005. Undensified silica fume (SF) were used with a percentage of 8% relative to the mass of cement. The percentage of SiO_2 was more than 90% and the specific surface area of SF was 17500 m^2/kg with a specific gravity of 2.2.

A synthetic polycarboxylate polymer-based superplasticiser (SP) with solid content of 30% and specific gravity of 1.05 was used.

Diutan gum was supplied by Kelco-crete which is an anionic polysaccharide gum as used as viscosity modifying agent (VMA1). It was used as powder at 0.05% (by mass of binder). A second VMA Acti-gel208 nano-clay based composed of attapulgite-clay small needles with negative charges (VMA2) is used at 0.10% (by mass of binder).

Sand with maximum particle size of 1.18 mm was obtained by sieving oven-dried (at 105 ±5 °C) sand with particle size of 0–5 mm.

Flax fibres supplied by Azichem Ltd (Italy) were used for this investigation have a length of 15 mm. Monofilament flax fibres are used to improve cohesion, holding, mould-ability, and to limit the cracking of cementitious composites.

2.2 Mixing and testing procedure

The mortars were prepared with high-shear Hobart mixer in 2-L batches. Premixed solid components, i.e. cement, fly ash (if any), silica fume (if any) and sand, were mixed for 30 seconds at low speed (140 rpm). Next, water at temperature of 16 ±0.5 °C and SP were added together to the mixer and mixed for 30 seconds at a low speed (140 rpm). Then the mixer was stopped, any lumps of solids were crushed, fibres (if any) and VMA1 or VMA2 (if any) were added (within 1 min), and the mortar was mixed again for 2 min (4 min when VMA2 is added) at a high speed (285 rpm). Then VMA1 (if any) is added and the mortar is mixed 1 min at a low speed (140 rpm). The temperature of the mortar after mixing was 20 ± 1 °C. For all tests the timing is given from zero time – that is, the time when the cement particles first touch the mixing water.

The penetration test (cone plunger) was started at 6 min (immediately after the end of mixing – Fig. 1). After filling the cone mould with mortar in two layers, the cone plunger is adjusted in order to allowing the cone to just touch the surface of the mortar sample. Then the cone plunger is released allowing the plunger to penetrate into mortar paste under its own weight for 5 seconds. After 5 seconds, the penetration value is noted to the nearest millimetre.

Then the slump flow test was started at 10 min (Fig. 2). The dimensions of cone were 100 x 70x 60 mm. The cone-shaped mould was placed in the centre of a jolting table. After filling with mortar in two layers, the cone was gently lifted 15 times (approximately 30 s after finishing of placing of the grout). When the flow stopped, the spread of the mortar was measured with a ruler in two perpendicular directions. Finally, extrudability of mortar mixes is tested with the simple modified joint gun described in Fig. 3.

Rheological measurement was carried out with a computer-controlled vane viscometer (Haake VT550). At 6 ± 1 min, approx. 800 ml of the sample was introduced into a plastic container where the vane was plunged. After 30 s rest, the test was started and the mortar was sheared at different shear rates.

The two rheological parameters, yield stress (τ_0) and plastic viscosity (μ_p), were obtained from the modified Bingham model fitted to the experimental data (down-curve). The yield stress at a zero shear rate was extrapolated from the Bingham model. Unfortunately, as mortar mixes were too stiff to be characterized with the viscometer, a semi-empirical alternative is used to determine the yield stress using cylinder measurement (slump – Fig. 4, [Pashias 1996]) : $s' = 1 - 2\tau'_y(1 - 2\ln(2\tau'_0))$ where:

- Cylinder dimensions : h_0 = 138 mm and d_0 =64 mm
- s = h_0 - h with h_0 the initial height and h the final height
- s' = s/h deformation quantities are dimensionless
- $\tau'_0 = \tau_0/\rho m.g.h_0$ stress quantities are dimensionless

The test procedure is detailed in the following steps. The mould is placed centrally on the flow table and the mortar is introduced in two layers. Each layer is compacted with ten short strokes in order to have a uniform filling of the mould. The mould is maintained in place firmly during the filling. The excess of mortar is removing with the help of a palette knife. After 15 seconds the cone is gently and vertically lifted and the height of the spread mortar is measured (Fig. 4).

The weight of the mortar was measured in constant volume and the fresh density of the mortar was calculated.

The workability life is a crucial parameter which is influenced by the build-up rate of layers. It was measured by the time at which it reached a defined limit of stiffness or workability during the gun extrusion test.

The workability life test measuring the evolution of the consistency, yield stress and extrudability during time was performed beginning with the first measurement of the flow table test and penetrometer test, and then cylindrical slump test and extrudability test. After, these tests were repeated every fifteen minutes until the mortar was too stiff. These tests are interesting because it can indicate the printable length of the layer which influences the overlaying time gap between two layers.

Fig. 1: Penetration test

Fig. 2: Slump flow test

Fig. 3: .Extrudability test

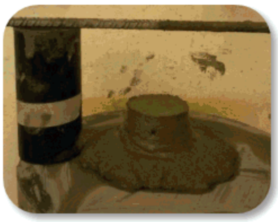

Fig. 4: .Slump of cylinder test

3 MIX PROPRTIONS

Table 1 summaries the mix proportions of 3D mortars tested in this investigation. The w/b ratio used was 0.50 and binder-to-sand ratio was 0.50. Four parameters were investigated: effects of fly ash as replacement of 24% (by mass of cement) and silica fume (8% replacement of cement), dosage of SP (0.275(1/2 SP) and 0.55% (SP) of binder), dosage of PP fibres (0.2% to 0.6%), and effect of type of VMA (% of binder by mass, VMA1, VMA2).

The diutan gum based VMA was added at 0.05% and the nano-clay based VMA was added at 0.1%. Then flax fibres were added with a varying percentage from 0.2 to 0.6% relative to the total batch volume in order to reduce the shrinkage and the deformation in plastic state.

4 RESULTS AND DISCUSSION

As previously stated, the purpose of the experimental program is to analyse the effect of different mix compositions on the rheological behaviour and fresh properties of a printable mortar in using basic tests (penetrometer test, flow table test, cylindrical slump test). The rheological parameters were evaluated by yield stress.

In this investigation, stiff mixes are used and it causes some difficulties in applying a rheometer test for mortar mixtures. Therefore, the slump test as a simplified rheological test is adopted in order to estimate the static yield stress.

Tab. 1: Compositions of all mixes made with flax fibres (kg/m^3)

	Cement	Water	Sand	FA	SF	VMA1 (%)	VMA2 (%)	SP (%)	FL
SP FL	463	307	1204	144	-	-	-	0.55	1.2
SP 3 FL	463	310	1195	144	-	-	-	0.55	3.6
1/2 SP FL	463	308	1204	144	-	-	-	0.275	1.2
1/2 SP 3 FL	463	311	1195	144	-	-	-	0.275	3.6
SF 1/2 SP FL	432	274	1224	146	75	-	-	0.275	1.2
SF 1/2 SP 3FL	432	278	1215	146	75	-	-	0.275	3.6
VMA1 SP 3FL	463	310	1195	144	-	0.05	-	0.55	3.6
VMA2 SP 3FL	463	310	1195	144	-	-	0.10	0.55	3.6
VMA2 SF SP 3FL	463	305	1215	144	75	-	0.10	0.55	3.6

4.1 Effect of binder on fresh and rheological properties of printed layers

Results of penetration and slump flow, obtained by flow table, composed of 100% of cement, 76% of cement and 24% of fly ash are plotted in Fig. 5.

Figure 5 indicates a decrease in the slump flow diameter and in parallel an increase in the static yield stress (from 789 to 913 Pa and 656 to 887 Pa) with the addition of silica fume. Silica fume improved the cohesiveness, reduces bleeding and the setting delay. The surface area of SF was 17500 m²/kg which is really higher than those of cement and fly ash (370 m²/kg and 370m²/kg, respectively). Consequently, bleeding and segregation is reduced as more water is needed to cover the surface of these particles [Sonebi 2010] [Sonebi 2015]]. Silica fume counterbalances the addition of SP and fly ash (retarder effect) which decreases the spread diameter and increases the yield stress.

The results of the penetrometer have the same trend compared to the results of slump flow as it can be noticed in Fig. 5. The penetrometer results indicate a decrease in the penetration for the mix made with FL and the addition of fly ash. Similarly, penetrometer values also decreased when a high dosage of flax used (3FL) normal quantity of flax.

SF permitted to obtain a stable structure which is very interesting for the 3D printings which need to keep after the extrusion the shape of the layer under its own weight and under successive layers without degrading the extrudability (Fig. 6).

It can be noticed a greater slump flow for the mix 1/2 SP 3FL compared to 1/2SPFL mix. It can be explained by a technical imprecision during the measurement or the compaction of these very stiff mortar mixes. Indeed as the mix is stiff, some air might be entrapped during the compaction in the cone.

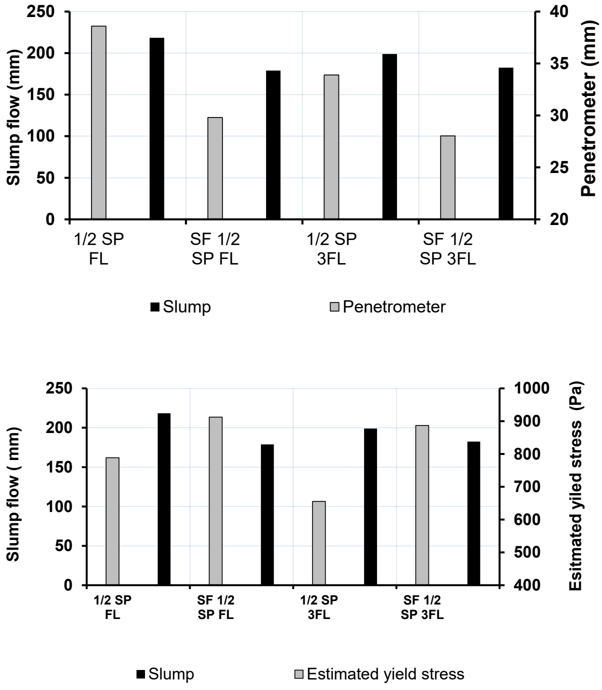

Fig.5: Effect of silica fume on slump and penetration, and estimated yield stress

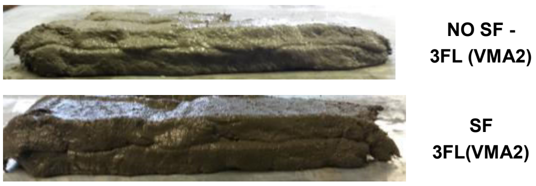

Fig. 6: Effect of SF on extrudability of layers

4.2 Effect of fibres on fresh and rheological properties of printed layers

Fig. 7 indicates a slight decrease in the slump flow with the increase of the percentage of flax fibres from 0.2% to 0.6% (FL to 3 FL). It can be noticed that increasing the percentage of fibres enhanced the cohesiveness which limited significantly the flow of the mix and has an influence on its workability [Martinie 2009] [Kaci 2011]. Adding fibres resulted in a better cohesiveness and more stable structure of mortar to not collapse under layer own weight and sustained successive layers weight. The flax fibres network might be noticeably less dense than other polypropylene mesh due to the length [Marine 2017]. As mix with a dosage of 0.55%SP was too fluid, the semi-empirical formula used wasn't able to calculate the static yield stress. But referring to the results of the slump flow, the increase of the flax fibres to 3FL might not affect significantly the yield stress and the workability of the mix.

The results of the penetrometer (Fig. 7) indicate the same penetration with the addition of 3FL fibres. The results of the penetrometer might confirm the results of the slump flow and the hypothesis that 0.2 or 0.6%FL did not have an influence on penetration of plunger. A higher dosage in flax might be tested in further research.

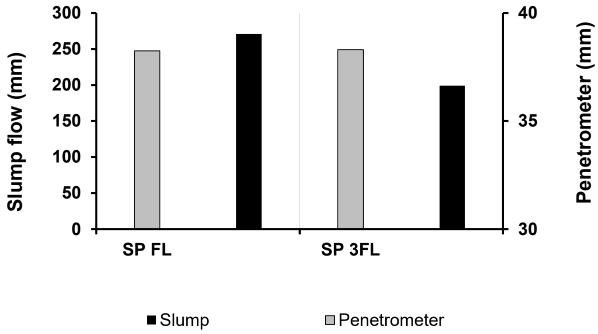

Fig. 7: Effect of dosage of flax fibres on slump flow and penetration

4.3 Effect of chemical admixtures on fresh and rheological properties of printed layers

Superplasticiser

As expected the reduction of SP (0.55 to 0.275%) led to decrease in a slump flow (Fig. 8). Addition of 0.55% SP led to a higher of slump flow (around 268mm). Unfortunately, as mixes with 0.55% of SP were too fluid (Fig.8), the semi-empirical formula used wasn't able to calculate the static yield stress of these flax mixes. Thus a conclusion about the influence of the superplasticiser dosage on the workability of the mortar mix can't be done. But referring to the results of the slump flow, the decrease of the dosage of SP might not affect significantly the yield stress and the workability of the mix.

The results of the penetrometer have the same trend compared to the results of slump flow as it can be noticed in Fig. 8. The results of the penetrometer indicate a reduction in the penetration values with the decrease of SP from 0.55 to 0.275% (SP to 1/2SP). Penetrometer results confirmed the influence of the effect of superplasticiser on consistency and flowability. Fig. 9 presents the shape of 2 printed layers made with SP (0.55%) and 1/2SP (0.275%).

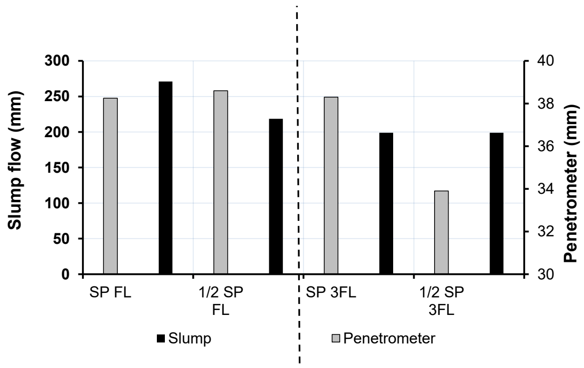

Fig. 8:.Effect of dosage of SP and slump and penetration

Fig. 9: Shape of 2 printed layers with increasing of SP

Effect of VMAs

Fig. 10 shows that the addition of diutan gum for a given dosage of SP decrease significantly the slump diameter and in parallel increase the static yield stress (from almost zero to 623 Pa). With diutan gum the flowability and deformability are reduced and the cohesion is enhanced due to the entanglement and intertwining of VMA polymer chains at low shear rate. At a dosage of 0.05%, VMA1 influences radically the consistency, the yield stress, the cohesiveness and increase the viscous behaviour of the mortar with flax fibres [Sonebi 2010] [Sonebi ; 2006] [Sonebi 2003].

The results of the penetrometer have the same trend compared to the results of slump flow as it can be noticed in Fig.10. The results of the penetrometer indicate a decrease of the penetration with the addition of diutan gum (VMA1). Fig. 11 shows the effect of VMA1 on the printed layers. It can be seen the shape is much better with VMA1.

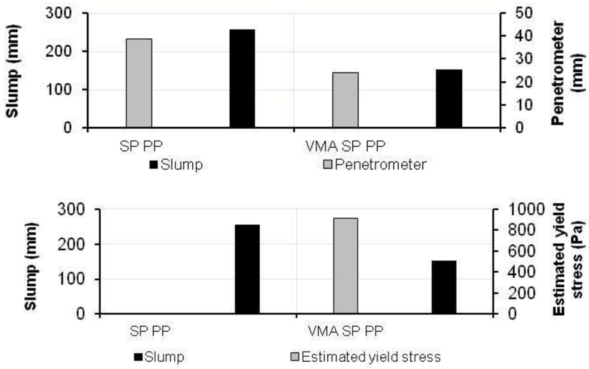

Fig.10: Effect of VMA1 on slump flow and penetration and estimated yield stress

NO VMA1

VMA1

Fig.11: Effect of VMA1 on shape of 2 printed layers

Fig. 12 shows that the addition of nano-clay gel VMA2 for a given dosage of SP decrease significantly the slump flow. However, the impact of VMA2 on the static yield stress can't be noticed (Fig. 12) because of the semi-empirical formula which wasn't able to determine the static yield stress of this fluid mortar mix. As the slump flow decreased with VMA2, it might be expected an increase of the static yield stress. Indeed, the particle size of nano-clay is significantly finer than those of cement and fly ash. Consequently, particles are inter mingle with the binder which increase the number of physical contact points and lead to enhance shear resistance. In addition, nano-clay increased the flocculation strength of the suspension which improves the structural stability. Additionally, cohesiveness of the mix is improved with the addition of nano-clay. [Nathan 2010] [Kim 2010]

The addition of silica fume characterized by a small particle size distribution seems to combine its effects with VMA2 ones and led to decrease further in slump flow (221 with VMA2 to 205mm with VMA2 and SF).

It can be noticed that in the reduction of the slump flow diameter, the effect of VMA1 with a reduction of about 49% is more efficient than the effect of VMA2 with a reduction about 18%. This difference could be explained by the type of mechanism and dosage. VMA1 cause an important loss of workability which influences the passing ability of the mix through the extruder. Therefore, VMA2 might be a better solution which improves the stability of the structure without decreasing dramatically the workability.

The results of the penetrometer haven't the same trend compared to the results of slump flows as it can be noticed in Fig. 12. The results of the penetrometer indicate slightly the same penetration with the addition of VMA2. These results might indicate that VMA2 isn't able to resist to this type of load of plunger. Nevertheless, it can be noticed that the addition of silica fume led to a decrease in the penetration values as stated before in the part concerning the influence of silica fume on consistence of mortar. Fig. 13 shows the effect of VMA 2 on printable layers.

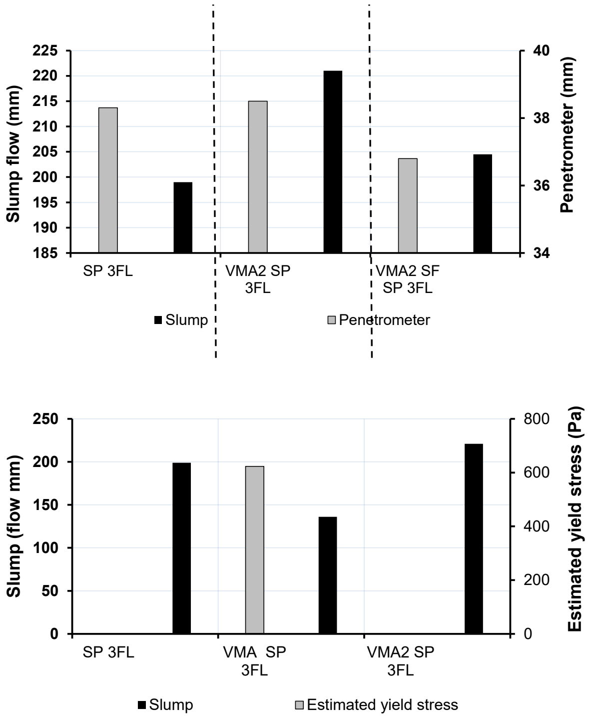

Fig. 12: Effect of VMA2 on slump flow, penetrometer and estimated yield stress

NO VMA2

VMA2

Fig. 13: Effect of VMA2 printed layers

4.4 Correlation

Both flow table and penetrometer can measure the consistency of mortar. Consistency is the deformability of mortar when subjected to a certain type of stress. A correlation between slump flow and the penetrometer for the same type of mortar was established for mixes without Sf and VMA (Fig. 14).

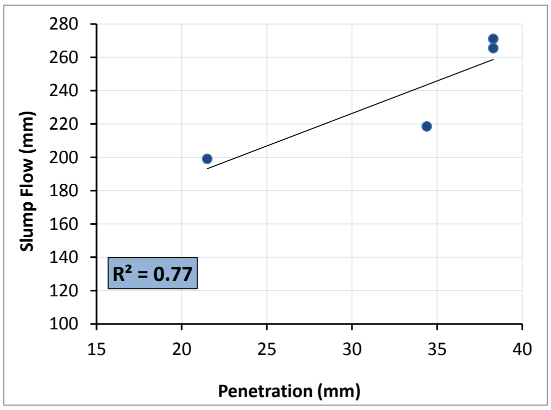

Fig. 14: Correlation between slump flow and penetrometer (No VMA and No SF)

4.5 Opening time

Time gap represents the time period in which the mortar is still reasonably workable and extrudable in layers. The change of workability with time related to the setting time of the cementitious mixes was measured using the flow table test and in some the estimated yield stress. The extrudability was tested in extruding layers with a simple modified joint gun.

Some example of the evolution of rheology of a fluid mix (SP 3FL), an intermediate mix (VMA2 SP PP) and a stiff mix (1/2 SP 3FL) during the time gap experimentation are presented in Fig. 15. This test is interesting because it can indicate the printable length of the layer and influences the overlaying time gap between two layers.

	Fluid mix : SP 3FL	Stiff mix: ½ SP 3FL
10 min		
40 min		 Plug
End		

Fig. 15: Extrudability of layers in function of time

Effect of silica fume

It can be observed on that adding SF in mixes containing flax fibres reduced the consistency and workability due to the increase of the cohesiveness and high reactivity of silica fume (Fig. 16). The mix without silica fume was already not extrudable after 10 min, and adding silica fume accentuate the difficulty to extrudability.

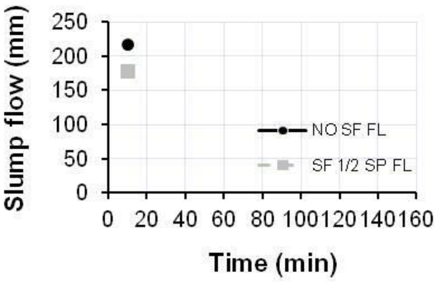

Fig. 16: Effect of SF on extrudability

Effect of superplasticiser

Open time was investigated with varying SP from (1/2SP to SP) by weight of binder (Fig. 17). As previously reported, the decrease of SP influences significantly the spread diameter, the yield stress and the time gap of the mix. Indeed, it can be noticed a decrease of 60 min with the addition 1/2SP. Indeed high dosage of SP can cause segregation; bleeding and retardation of cement hydration might be the cause of this increase of time gap.

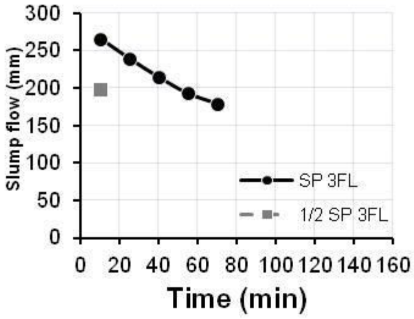

Fig. 17: Effect of SP on slump flow in function of opening time

Effect of VMAs

The results show that the addition of diutan gum for a given dosage of SP (0.55%) decreased significantly the open time of the mortar (Fig. 18). It can be noticed a decrease of 30 min with the addition of 0.05% by weight of cement of VMA1. Action of VMA1 combined with built up rate of the cementitious mix reduced the time gap.

In addition, the slope of the VMA1 curve is lower than the slope of the mix without VMA which means that the mix is very stiff and can't spread anymore (19% drop without VMA1 compare to 9% with VMA1)- Fig. 17. Flax fibres have a fibres network less dense than the one of polypropylene fibres due to its length; it allows a greater time gap.

The addition of 0.1% of nano-clay shows a decrease in the open time of 15 min which is half lower compared to the results with the addition of VMA1. As previously reported with the addition of VMA1, VMA2 curve is lower than the slope of the mix without VMA which means that the mix is very stiff and can't spread anymore (27% drop without VMA compare to 16% with VMA2, Fig. 18). VMA2 increased the cohesiveness, viscosity, and flocculation of the suspension. Action of VMA2 combined with built up rate of the cementitious materials reduced the time gap.

VMA2 allowed a better flowability, cohesion and passing ability through the orifice of the gun than VMA2 and its steric repulsion and permit a better extrusion and greater open time.

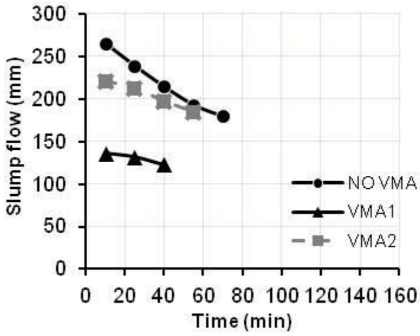

Fig. 17: Effect of VMA1 and VMA2 on slump flow in function of opening time of mix with flax fibres

5 CONCLUSION

This study was focused to investigate the effect of different mix compositions on the rheological behaviour and fresh properties of a printable mortar in using basic tests like the penetrometer test, the flow table test and the cylindrical slump test. Layers are extruded with a simple modified joint gun. This tool permitted in association with the slump test to determine the time gap and the extrudability of the different mixes. Conclusions can be drawn:

Concerning the binder composition, regarding the analyse of results it can be observed that adding 24% of fly ash (FA) and 8% of silica fume (SF) increasing yield stress, cohesiveness, structure homogeneity and stability appeared to be an advantage to print layers. Indeed it reduced the too important fluidity, bleeding and the segregation of layers as it was previously noticed with a reduction of the flow table diameter. 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.

The addition of flax fibres (FL) fibres (0.2% to 0.6%) permitted to the mortar to have a greater yield stress, cohesiveness and have a more stable structure to not flow under layer own weight and successive layers weight. With the incorporation of fibres the time gap is reduced. Additionally results might highlight the influence of the type, length and diameter of fibres. 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).

The incorporation of VMAs permitted to gain in cohesiveness, viscosity avoiding bleeding and segregation which is an advantage for 3D printing. However, the type and dosage of VMA is very important. As it was previously stated, VMA1 cause an important loss of workability and time gap which influences the pass ability of the mix through the extruder. Therefore, VMA2 might be a better solution which improves the stability of the structure without decreasing dramatically the workability and time gap. Moreover increasing the dosage of SP improved the workability, the passing ability trough the extruder and led to a greater slump flow diameter and penetration. However a too high dosage of SP increased also risk of bleeding and segregation of the mix.

A good correlation between penetrometer and flow table results was obtained.

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