



Use of calcium sulfoaluminate cements for setting control of 3D-printing mortars



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HIGHLIGHTS

- A printable OPC-CSA mix has been developed.
- Manual device has been used to simulate 3D printing.
- Mechanical properties of the printable mix has been studied.

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ABSTRACT

Printing cementitious structures in 3D (3D Printing) requires the use of a mortar which setting is rigorously controlled. In this paper, a mix made out of two types of cement, ordinary Portland cement (OPC) and Calcium Sulfo-aluminate cement (CSA), was adopted to control the printability of a mortar. The latter was formulated in order to be extrudable, buildable and to reach a compressive strength comparable to that of a traditional mortar. Finally, a printable mix made out of 7% CSA and 93% OPC was developed. The cement paste of the developed mix was tested using isothermal calorimetry to study the impact of CSA on the heat of hydration of the mix. The mortar is first checked for the two characteristics of printability (extrudability and buildability) by a manual home-made device. Then, an estimation of the rheological properties of the mortar using a penetration test was done. Afterwards, the strength of the mortar was tested and a compressive strength of 88 MPa was found after 28 days for non-printed specimens and 79 MPa for printed specimens.

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1. Introduction

3D printing (3DP) is a process of additive manufacturing (AM) defined by the ASTM as being “the process of joining materials to make objects from 3D model data, usually layer upon layer” [1]. It is an automated process based on CAD software to realize objects from bottom to top. Many methods of 3DP have been developed for several types of materials [2]. This technology has covered so far the use of many materials such as metals [3], ceramics [4] and plastic polymers [5]. For example, 3DP noticed a remarkable development in the aircraft, medical, and food industries [6–8]. In construction, 3DP could decrease the need for assembly work to a one-step process [9,10]. Moreover, it would provide a flexible and low cost production by reducing the use of tools and molds [11]. Manpower and risks could be decreased and the building procedure

facilitated [6]. A new industrial revolution is being launched through this technology. Recently, it is developing fast and expectations are growing to their peak. However, this method is still immature to answer all expectations [11,12].

Whereas 3D printing of cementitious material is used in different countries [13,14], very few scientific publications are noted [15–18]. Few of these publications discuss the material properties and their characterization methods. Indeed, there are no defined standard tests for the material efficiency. One of the obstacles facing the implementation of 3DP in the construction field concerns lack of specifications for the printed materials as discussed by Wu et al. [19]. These authors define two basic specifications that have to be fulfilled by a mortar to be considered as printable: extrudability and buildability. Extrudability is defined by the workability enabling the material to get out of the nozzle of the printer. Buildability is related to the need of the material to bond together so that layers lay down in a right manner, stay still and stiff enough to handle the superposition of layers without collapsing.

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Wangler et al. [20] related the ratio of the initial and final yield stresses of the first printed layer to the number of layers printed. They explained that in order to print a structure of 80–150 cm made out of layers of 1–3 cm thickness, the yield stress ratio had to increase by a factor comprised between 25 and 150. Thus, the yield stress of the material is required to change rapidly. At this stage comes the importance of chemistry for controlling the behavior of the mortar over time.

Few studies mention the mixture composition of 3D printing mortars. Malaeb et al. [16] printed a wall consisting of two 77 cm parallel lines of 10 cm height and separated by 10 cm. Their mix was accelerated by a combination of retarders and accelerators added at different stages to extrude the mortar from a 2 cm diameter nozzle. A 28 days compressive strength of 42 MPa was obtained [16]. Le et al. [17,18] studied the hardened properties of high-performance printing concrete by adding admixtures such as fly ash, silica fume and fibers to the mix. An amino-tris (methylenephosphonic acid), citric acid and formaldehyde retarder and polycarboxylate based super-plasticizers were used as well to develop the printable mortar, with constant flow, that would be extruded through a 9 mm diameter nozzle. The compressive strength of the material was tested for printed specimens sawn and drilled from larger printed components, whereas mold specimens were cast in 100 mm cube steel molds complying with the BS EN 1239-3 code. A compressive strength of 91–102 MPa was found for printed specimens that was 5–10% lower than mold cast specimens depending on compression orientation with respect to printing direction.

The printing process used in the studies of Le et al. [17,18], Malaeb et al. [16] and Gosselin et al. [14] was automated. Le et al. used digitally-controlled printing process with a 9 mm nozzle ($D_{\max}=2$ mm) [17,18]. Malaeb et al. used a machine designed for 3DP to move on a tri-axial plane with a 2 cm diameter nozzle ($D_{\max}=2$ mm) with two attached trowels lagging behind it [16]. As well, Gosselin et al. [14] used a printing machine that consists of a print head mounted on a 6-axis robotic arm.

The studies mentioned above present different mixes of printable mortars using Portland cement together with various admixtures. Another way of changing the reactivity of Portland cement is by the use of sulfo-aluminate cement. In fact, sulfo-aluminate cement is showing improvements regarding the evolution of the cement hydration [21]. Its main hydration product is ettringite that is one of the basic constituents of rapid hardening cements [22,23]. Mixes of calcium sulfo-aluminate cement (CSA) and ordinary Portland cement (OPC) are applied in special applications. These blends of OPC and CSA were used for example in US and Japan in the sixties in order to produce expansive cements where OPC is blended in a mainly CSA cement mixture to control its expansion. However, expansion may also lead to voids formation and result in a decrease in compressive strength [24,25]. CSA is applied in other fields like stabilization of wastes and it is considered as green cement since its fabrication process is done at lower temperature than OPC and releases less CO_2 than OPC [26,27]. CSA can be used to improve the properties of high grade slag cement. Michel et al. [28] ended up with increasing the strength at 24 h due to the hydration of the sulfo-aluminate cement and later a high strength due to the interaction between the Portland and slag.

The main objective of this paper is to develop a printable mortar that maintains its mechanical properties at 28 days. Printability refers here to a mortar that is both extrudable and buildable. In order to fulfill these requirements, CSA is used in a mix principally made of OPC. CSA, acting as a setting accelerator, is expected to bring the characteristic of buildability to a workable mortar and therefore promote it to a printable mortar. A procedure divided into 4 points was adopted. First, an appropriate device visualizing the printing process was developed based on a manual printing

process allowing testing the mortar for its extrudability and buildability. Second, the isothermal calorimetry was studied to understand the effect of the composition of the binder on the heat of hydration of the cement paste. Third, rheological characteristics of the material using penetration test allowed us estimating the yield stress of the mortar. Fourth, after finding the printable material, its mechanical behavior was verified.

2. Methodology

The 3DP process is simulated in the laboratory by a manual process that represents the automated one but on a smaller scale. The manual device used for printing is made of a silicon gun shown in Fig. 1. At one end, a manual pressure can be applied for extrusion and at the other end a nozzle is positioned with 5 cm diameter at its start and 1 cm at its end, allowing for the extrusion of a mortar containing sand particles lower than or equal to 2 mm. In fact, it has been shown in our previous publication, that a diameter ratio of $D_{\text{nozzle}}/D_{\text{max}}$ larger than 5 ends up in no blocking to occur during the extrusion [29].

In order to be extrudable, the mortar should have a certain workability allowing it to pass through the nozzle without blocking under the pressure applied manually on the device. However, it is not expected to flow under its self-weight. In addition, no segregation, bleeding or filtration (sand particles clogged in gun and water and cement filtrating through the granular skeleton) are allowed to appear. These issues can be detected as signs of blocking.

Once the mortar is extruded, it needs to comply with the second characteristic: buildability. The weight of the superposed layers is supposed to be held by the bottom layers. The rheological properties of the fresh mortar therefore have to change throughout time in order to become stiffer as the height of printed material increases. At a certain height, the bottom layers even have to start hardening to maintain their width and prevent the printed structure from falling during printing more layers.

Once the mix developed complies with these specifications, it is considered to be applicable in the 3DP process and then given the name printable.

3. Material and mixes

Two cements have been used: an OPC (CEM I 52.5) EXTREMAT® CEM I 52.5N-SR3 CE PM-CP2 NF I (99% Clinker) and a Calcium Sulfo Aluminate Alpenat cement, both from Vicat company. A new generation polyvalent non chlorated acrylic copolymer superplasticizer (SP)/high water reducer, SIKA VISCOCRETE TEMPO 11 was used. A calcareous 0/2 mm crushed sand with of 19% particles smaller than $63 \mu\text{m}$ was used provided by Carrieres du Boulonnais. The following Tables 1–4, present the chemical and mineralogical characteristics of the OPC and the CSA provided by the manufacturer.

For isothermal calorimetry measurements, only cement pastes were studied. Table 5 presents the composition of all cement pastes tested. Mixes are labeled according to the percentage of OPC and CSA used: the first number refers to the percentage of OPC and the second to the percentage of CSA. For example,



Fig. 1. Device used for mortar printing.

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