


Printability of materials for extrusion 3D printing technologies: a review of material requirements and testing

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ABSTRACT: One of the major challenges facing 3D printing for construction is the technological suitability, ‘printability’, of the materials used. These cement-based materials differ from those used in other sectors, which has a series of conditioning factors that are the object of the present analysis. This article first reviews the definition of the term ‘printability’ and its constituent stages. Those stages condition the requirements to be met by cement-based materials, whether designed for other uses or developed ad hoc, and therefore the tests applicable to determine their aptitude for use in additive manufacturing for construction. That is followed by a review of the standardised tests presently in place for mortars and concretes that can be used to verify a material’s compliance with such requirements. The paper concludes with a recommendation on the advisability of developing a standard test or suite of tests to ascertain printability.

KEYWORDS: Concrete; Cement; 3D printing; Testing; Additive manufacturing; Construction; Printability.

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RESUMEN: *Imprimibilidad de materiales para la impresión 3D por extrusión: Una revisión de requisitos y ensayos.* Uno de los retos más importantes de la impresión 3D en la construcción es la tecnología de los materiales utilizados, estos materiales en base cemento se diferencian de los utilizados en otros sectores, lo cual tiene una serie de condicionantes que se analizan y desarrollan en el siguiente trabajo. Este artículo revisa en primer lugar la definición del término imprimibilidad y de cuáles son las fases que debe incluir. Estas fases a su vez condicionarán los requisitos que estos materiales en base cemento deben tener, así como que ensayos o pruebas se pueden realizar a un determinado material, bien que haya sido formulado para otros usos o bien que haya sido desarrollado ad hoc, con el objetivo de ver si es apto para su uso en fabricación aditiva en construcción. Por último, se presenta una revisión de los ensayos que actualmente existen y permiten comprobar el cumplimiento de los mencionados requisitos, ensayos que ya se encuentran normalizados tanto en morteros como en hormigones. Planteando si debería existir bien un ensayo o grupo de ensayos normalizados que permitirán conocer previamente si un material es imprimible.

PALABRAS CLAVE: Hormigón; Cemento; Impresión 3D; Pruebas; Fabricación de aditivos; Construcción; Imprimibilidad.

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1. INTRODUCTION

Many materials have been used in additive manufacturing, better known as 3D printing, and more specifically in fused deposition modelling (FDM), origin of the technology in place in the construction industry. In most sectors, ranging widely from agriculture to healthcare and automobile and aeronautical manufacture, polymers are the materials of choice and prototype formulation a necessary procedure.

The best known of such polymers (1) include acrylonitrile butadiene styrene (ABS) and polylactic acid (PLA), along with others for more specific applications such as polyethylene terephthalate (PET) or thermoplastic polyurethane (TPU). All are thermoplastic polymers that become more fluid at a given temperature, rendering them readily extrudable with no significant degradation. When a substrate is required the 3D printer deposits material that is subsequently removed, a sacrificial material that serves as support until the polymer cools and therefore hardens (2).

Whilst the use of such polymers has been consolidated in other sectors, they are seldom deployed in construction due to high costs and structural limitations involved.

The need for higher temperatures to change from a solid to a liquid state for extrusion in additive manufacturing (AM) is a feature common to polymers. Analogously, the addition of water for reaction is a trait shared by hydraulic materials. The resulting mix is workable for a certain period of time, after which it is no longer extrudable for deposition and subsequent setting. That explains why reviews tend to focus on these materials' 'early-age' behaviour (3, 4). A distinction must be drawn, however, between pre- and post-extrusion early-age behaviour (5).

As in traditional construction, cement-based materials are indisputably the products most generally used today in industry AM. Such widespread application can be explained by their low cost, ready availability and capacities, not to mention their simplicity of use.

Technology and material design, whether referred to cement-based or other products (such as plaster, mixed substances, ceramics or geopolymers), are closely interrelated in 3D printing for construction, insofar as the former conditions and largely determines the specifications that must be met for the latter to be 'printable'.

Printability has been defined in a number of ways, depending on the author. Hou and Duan (6) deemed it the capacity of a fresh material to be continually extruded and deposited with a given deformation before setting. Others such as Nerella and Mechtcherine (7) defined it as a combination of pumpability, extrudability and buildability. Buswell *et al.* (8) and Panda *et al.* (9) contended that printability can be

assessed in terms of the deformation of the recently printed constituents in a certain number of layers, a property called buildability in other papers, such as one by Ma *et al.* on the printable properties of cement-based materials bearing copper waste (10).

Printing with cement-based materials comprises a number of stages (Figure 1).

- Feeding, normally pump-driven, the fresh material to the nozzle.
- Extruding and depositing the material, where requirements are conditioned by the method (ram or screw extrusion).
- Hardening, in which the material must exhibit sufficient early-age strength to accommodate successive layering.

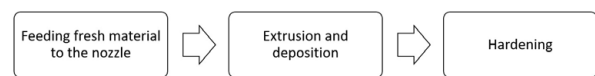


FIGURE 1. Stages in 3D printing.

Attendant upon each stage is a series of conditioning factors that determine the material's printability. Here printability is consequently defined to mean the capacity of a fresh material to be pumped/carried to the printer nozzle for extrusion and deposited layer-by-layer subject to a given minimum and predictable deformation.

This article reviews the requirements to be met by cement-based materials, along with the tests presently in place and in use in different projects to verify their compliance with such requisites.

Lastly, a range of cement-based materials presently available on the market, albeit for other uses, are analysed for a priori compliance and possible usability in 3D printing.

Key research question in the paper are:

What standardized tests are applicable to analyse printability of cement-based materials in additive manufacturing for construction to verify a material's compliance with such requirements, and what are the results from performed tests?

What are the recommendations on the advisability of developing a standard test or suite of tests to ascertain printability, based on the study?

2. 3D PRINTING STAGES IN CONSTRUCTION

2.1 Pumping fresh material

Concrete pumping, an on-site technology thanks to which the material can be poured where it would otherwise be unfeasible, hastens construction, thereby raising output and lowering costs. In additive manufacturing or 3D printing it is the method most usually deployed to carry the freshly mixed material to the printer nozzle. Alternative systems involving

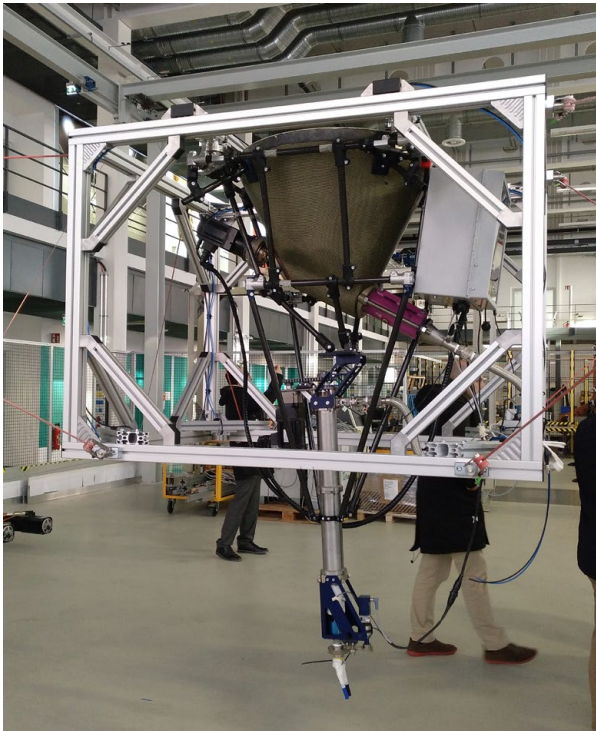


FIGURE 2. 3D printer with intermediate deposit (HINDCON project).



FIGURE 3. Pipe delivering printing material (3D Cons project).

intermediate deposition to ensure a continuous flow of material have also been tested, however. Although less common than pumping, these procedures (Figure 2), have proved able to suitably maintain a constant supply of material while avoiding possible pumpability issues.

In additive manufacturing, the material is generally pumped through a flexible pipe to ensure nozzle positioning and mobility (Figure 3). The pressure used must be sufficient for the material to deform and travel through the pipe. Depending on the size of the aggregates, this material can be considered a mortar or micro-concrete. Any change in the composition may affect fresh mix performance. Characteristics such as the water/cement ratio, aggregate particle size distribution or the presence of superplasticizers or other chemical admixtures modify

material rheology and must therefore be determined a priori.

That determines a need to estimate the pressure required to pump a given mix of fresh material a given distance or height at a given known and controlled speed. Such estimates lay the grounds for optimising the composition of the cement-based material and defining the pumping pressure required to feed the printer, for an uninterrupted and constant flow rate is indispensable in additive manufacturing. The extensive research conducted on that issue in recent decades has given rise to a number of prediction and test models as well as numerical simulations of in-pipe particle flow (11), using different types of rheometers and tribometers to explore concrete pumpability (12, 13).

Nonetheless, in actual on-site practice pumping is still estimated with simple and empirical methods such as the slump test to determine freshly mixed concrete or mortar consistency. Based on the results of that test, expressed in centimetres (14), the material is classified under one of four consistency categories: true slump, zero slump, shear slump or collapse. There are values for a concrete to be pumpable, for 3D printed materials it is necessary to test in each case and it is not possible to determine a common value for all materials and pumping systems.

For mortar, characterised by a smaller aggregate size, fresh state consistency can be determined with two tests. The first involves placement on a spread or flow table to establish consistency in terms of the mean diameter of the resulting spread (15). The second is based on the penetration of a plunger in the sample (16). Traditionally a mortar has been said to be pumpable if it has a flowable consistency. Neither of these tests has proven suitable for accurately estimating pumping pressure, however.

Estimation can be found with slump tests and numerical simulation. ACI standard 304.2R-96-2000 (17) on placing concrete with pumping methods, like many of the diagrams used by concrete pump manufacturers, accepts the simplifying assumption that head loss and flow rate are linearly correlated. The empirical formula used is Equation [1]. Simplified formula for calculating pumping pressure, defines the pressure (p)-flow (q) p/q, m³/h relationship as linear during pumping. The coefficient that relates the two is equal to a constant that depends on circuit geometry (determined as length, L (m) and diameter, D (m)) and mix properties. That constant, b (coefficient on the tables published in the standard), is expressed as the mean slump.

$$p = b \times \frac{16L}{\pi} \times \frac{1}{D^3} q \quad [1]$$

New procedures have been developed in recent years that deliver a greater quantity of more accurate

information than those traditionally used to find the pumping pressure required to ensure a suitable supply of material from the mixer to the printer nozzle. These procedures attempt to be more sensitive to water/cement ratio, aggregate shape and admixtures than the aforementioned viscometers and concrete dispersion tables. Some of these methods are the sliper tube rheometer (12) or the study of pumping pressures with a tribometer (13), which measures the friction between the fresh material and the pipe through which it flows.

2.2 Extrusion

In extrusion, the second stage of AM consisting in controlled layer-by-layer deposition of the material pumped through the nozzle to attain the desired geometry (Figure 4), rheology is the determinant parameter.

Nonetheless concrete rheology, understood as its fluid and fresh state resistance to deformation, i.e., its workability, is not the sole factor involved in extrusion. Other properties such as homogeneity, suitable setting time and high thixotropy also affect the outcome.

As the material must remain sufficiently workable to be extruded without clogging, workability in concrete refers to how readily it can be laid on site after mixing. The American Society for Testing and Materials (ASTM) defines it as the property that determines the ease with which a freshly mixed concrete can be mixed, placed, consolidated and finished to a homogeneous condition (18).

At this time a wide variety of tests is applicable to determine the 'open' time (19), or the time during which a product is workable before hardening. Specific tests are in even place for different types of concrete. In self-compacting concrete, for instance, the most suitable procedure depends on the self-compacting method (20-23). European standard



FIGURE 4. Printer nozzle in operation (3DCons Project).

EN 196-3 (24) specifies the methods for determining ordinary cement setting times, although many other cement-based materials also apply the tests described, in light of their benchmark status. For bespoke materials expressly designed for additive manufacturing tests must be adapted and studied for calibration.

Another parameter that must be established is mix temperature prior to extrusion. Like any other chemical reaction, cement setting is affected by temperature. Early-age properties of 3D-printable mortars consequently depend heavily on the initial stages of (heat-generating) hydration kinetics. Hence the importance of assessing the extent to which the properties measured under standard laboratory conditions (20 °C) may be affected by variations in temperature in real out-of-laboratory printing.

Lastly, the material to be used must be thixotropic or exhibit accelerated early-age setting to maintain its shape after extrusion. Further to the definition of thixotropy, that entails studying the variation in material consistency when exposed to external actions such as extrusion and deposition.

Based on Mewis (25), Sonebi & Yahia (26) define thixotropy as a reversible, isothermal, time-dependent decrease in the apparent viscosity when a material is subjected to increased shear rate. There is no specific test to measure mortar or concrete thixotropy. Tests for self-compacting concretes are in place, as well as a test described in European standard EN 13062:2004 (27), to determine thixotropy in products for protecting reinforcing steel, which could be likened to the type of material at issue here.

That test consists in applying the freshly mixed material on two steel plates, one set horizontally and the other vertically. Thixotropy is the quotient of the mean thickness at the top of the vertical plate divided by the mean of the thicknesses on the horizontal plate.

2.3 Layer-by-layer deposition

In this final stage of 3D printing the conditioning factor is the buildability of the fresh mix used. Here the most important factors are the mechanical properties of the fresh mix, which must be highly thixotropic (or contain accelerating admixtures) to accommodate the deposition of one layer over another with acceptable and controlled deformation.

In this final stage of 3D printing, the conditioning factor is the buildability of the fresh mix used. Compared to traditional concrete, there are two mechanical problems that condition the buildability.

The first one is the evolution of the mechanical properties of the concrete in the early ages, from the plastic and deforming state to the hardened state, the layer-by-layer construction method requires a rapid development of the compressive strength to withstand the tension of the previous layers. Here the

most important factors are the mechanical properties of the fresh mix, which must be highly thixotropic (or contain accelerating additives) to accommodate the deposition of one layer on top of another with acceptable and controlled deformation.

The second problem is the strength of the bond between layers, this also occurs in other types of concretes such as self-compacting concretes with so-called “cold joints” or “distinct layer castings”, which are the interfaces where there is a reduction in mechanical properties.

The strength of this interlayer bond can be affected by a number of factors. Firstly, the parameters of the printing process itself, including the interval time, printhead speed and the height of the printing nozzle Panda et al. (28, 29) found that low values of the printhead speed and the separation distance of the printing nozzle from the printing nozzle led to an increase in the interface bond strength. On the other hand, Wolfs et al (30) reported that there was no clear relationship between nozzle height nozzle height and interface bond strength.

Other properties have effects on this adhesion, Roussel and Cussign (31) observed that the thixotropic property of cementitious materials had a negative effect on the interface bond strength, which is in contradiction with other requirements.

Two other properties of particular significance for characterising the mix and assessing its fresh state variations in volume are fresh mix compactability (32) and entrained air content (33, 34).

3. MATERIALS

A distinction may be drawn in materials apt for additive manufacturing between those designed for that purpose and the many others that are market-available but not designed for 3D printing per se. This paper reviews some of both types and their characteristics that determine how they should be assessed for the use at hand.

3.1 Materials designed for 3D printing

- Materials with not late-stage admixture

The mixing and pumping system that feeds material to the printer is common to most 3D printing setups used in international projects.

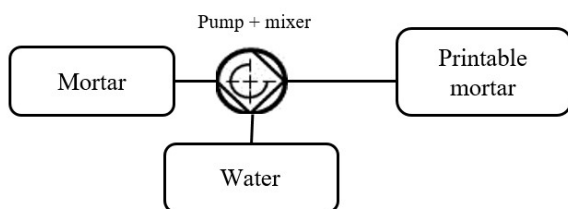


FIGURE 5. Flow chart of one-pump 3D printing facility.

In all those cases the pre-prepared materials, delivered in sacks, were mixed with water on site for subsequent uninterrupted pumping from the mixer to the extrusion nozzle (Figure 5).

The pre-prepared material contained the admixtures needed to ensure the necessary workability, pumpability, resistance to premature ageing and compliance with all other requisites.

- Late-stage admixed materials

Another approach, consists in preparing several litres of very fluid material apt for a given printing time, designed to flow under its own weight via a screw-based system to a mixing chamber placed immediately prior to the nozzle (Figure 6).

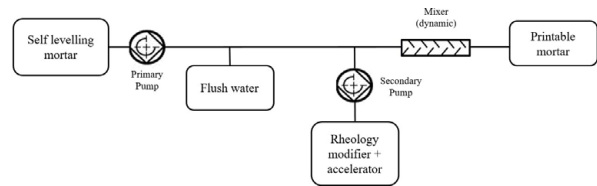


FIGURE 6. Flow chart of two-pump 3D printing facility.

There viscosity modifier agents (VMAs) or accelerating admixtures are added to the mix for extrusion to the geometry defined. Due to such late-stage inclusion of the admixtures, the material acquires a rheology that ensures workability as well as early strength development.

When tested, this process met the aims established, with the material exhibiting ready pumpability, ample open times and high early-age mechanical strength.

Fibres are sometimes added to cementitious materials designed for 3D printing to improve mechanical properties, mainly at flexural strengths where these materials have their limitations. When fibres are included in the cementitious material it is necessary to study the effect of different content percentages on workability and mechanical properties (35, 36). That is, the tensile strength should improve while maintaining an acceptable workability for 3D printing (37). There are also studies aimed at investigating the effect of fibre orientation in 3D printed concrete, determining its possible effects on the mechanical properties of 3D printed specimens and comparing them with those of conventionally moulded specimens. Some results revealed that smaller nozzle size and higher fibre volume fraction significantly improved fibre alignment parallel to the printing direction (38).

In order to correctly characterise a ultra-high performance fiber-reinforced concrete, it is necessary to determine the influence of its dosage, fibre content and fibre type. And finally the choice of test methods (39).

3.2 Materials with similar characteristics to those used in 3D printing

- Shotcrete (gunned or sprayed mortar)

In this procedure mortars (material containing no coarse aggregate) or concretes (material containing coarse aggregate) are air-blown onto a substrate on site at high pressure from a hose. The purpose is to cover the soil itself where otherwise impossible due to sloping terrain or other factors, ultimately to enhance strength and durability and optimise weatherproofing thanks to the scant porosity of the cover.

This technique is commonly used to stabilise embankments, clad tunnels and build swimming pools.

Gunmed concretes and mortars must meet a number of requirements that concur with the characteristics called for in 3D printing. As readily pumpable materials bearing setting stabilisers, they have flexible open times, high early-age strength and good substrate bondability, all features shared with additive manufacturing.

As those features of the materials used in sprayed concrete are shared with 3D printing materials, subject to the imperative prior study, these materials are candidates for adaptation to use in the additive manufacturing.

- Rendering or float coats

The mortars used as protective indoor and outdoor surface coverings and finishes are called rendering or float coats. Depending on the characteristics of the material they can be prepared for single or multilayer application.

The properties of these mortars that have prompted their use in a few pioneering 3D printing projects



FIGURE 7. Float coast testing.

include: early-age strength (although the values are not comparable to those of other mortars), impermeability, substrate bonding, flexibility and quality of finish.

As in the case of gunmed mortars, some of the characteristics of the rendering materials have induced their use in early tests of 3D printing projects (Figure 7). Those first tests gave very suitable values for some characteristics such as interlayer or

substrate bonding, however others such as early-age strength, while acceptable were not very high. The result was that their use was discarded later in the project and ad-hoc materials were developed.

- Repair mortars

Repair mortars, polymer-modified cement-based mortars and epoxy-based materials are developed to specific requirements to remedy damaged or deteriorated concrete and mortar.

Repair mortars are characterised primarily by excellent workability and finish, good substrate and interlayer bonding and excellent bonding to materials such as steel.

Their setting times can be controlled and they exhibit high early-age strength, essentially no drying shrinkage and high thixotropy.

With such features these mortars are worth exploring for their 3D printability.

4. TESTS

Bevies of projects implemented in recent years have entailed experimentation with many kinds of tests to assess the materials used. A fair share of those tests are not mutually comparable, inasmuch as they are designed to determine different characteristics in keeping with specific project needs (40).

Others, however, such as mechanical strength or setting times measured to standard, are comparable. Those tests reveal the differences between the materials used in different projects as well as between them and the commercial materials tested to determine their printability.

Development of appropriate fresh state property testing methods, especially for terms such as “printability” and “extrudability”, is essential for the proper process development. Additionally, development of standard test methods with reproducibility is necessary: As these test methods are generally custom-designed for now (41).

4.1 Fresh state tests

- Setting time

In some projects, tests have been run to determine

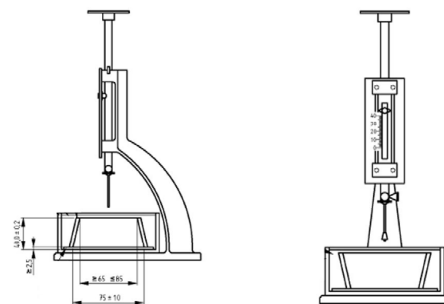


FIGURE 8. Vicat apparatus.

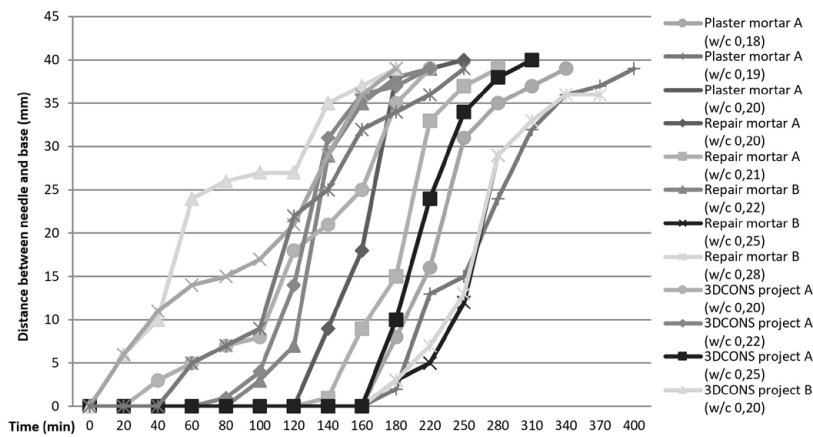


FIGURE 9. Setting times for several types of mortar.

setting times as specified in standards for ordinary cement (24). Such tests are not applicable to the materials used in other projects, however, because the standard method is not apt for measuring very short setting times, as when the mortar or concrete bears accelerating admixtures.

Another standard method deploys a manual device call the Vicat apparatus (Figure 8), comprising a probe or needle that penetrates a standardised mould containing the test material. The probe is lowered

at pre-set intervals and the depth of penetration recorded.

Figure 9 shows the setting times measured with a Vicat apparatus, and in table 1 we can see the beginning of setting of the mortar. It can be seen the comparison between mortars formulated for other applications and mortars developed ad-hoc for a project. The figure shows that the onset of setting is earlier in the mortars developed for 3D printing, while commercial premixed mortars show much longer setting times.

TABLE 1. Data for the trial runs shown in FIGURE 9.

MATERIAL	Plaster mortar A			Repair mortar A			Repair mortar B			3DCONS project A			3DCONS project B		
water-cement ratio (w/c)	0.18	0.19	0.20	0.20	0.21	0.22	0.25	0.28	0.20	0.22	0.25	0.20	0.22	0.25	
time (min)	Distance between needle and base (mm)														
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
20	0	0	0	0	0	0	0	0	0	0	0	6	6	0	
40	0	0	0	0	0	0	0	0	3	0	0	8	9	0	
60	0	0	0	0	0	0	0	0	4	0	0	16	12	5	
80	0	0	0	0	0	0	0	0	6	1	0	24	14	7	
100	0	0	0	0	0	3	0	0	7	3	0	25	15	9	
120	0	0	0	0	0	7	0	0	8	8	0	26	16	15	
140	0	0	9	9	1	29	0	0	12	14	0	27	17	21	
160	0	0	16	17	9	32	0	0	18	26	0	27	20	24	
180	8	2	22	21	15	35	3	3	21	33	10	28	24	25	
200	14	8	34	36	22	36	5	7	23	36	24	35	29	32	
220	20	13	38	38	33	38	12	13	25	37	34	36	35	33	
240	31	14	39	39	35	39	29	29	32	38	36	37	37	34	
260	33	18	40	40	37			33	36	39	38	39	39	36	
280	35	24			39			36	39		39			37	
300	37	32						36			40			39	
320	39	36													
340		37													
360		39													

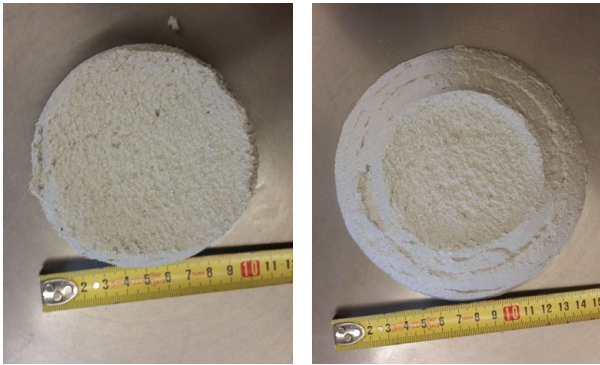


FIGURE 10. Two mortar patties generated by slump tests for consistency.

- Consistency

The slump test, used to determine the consistency of fresh masonry mortars (15) may also be applied to potential printing *materials*.

This test delivers good results for mortars where consistency depends on the water/cement ratio or other parameters, but is unsuitable when a thixotro-

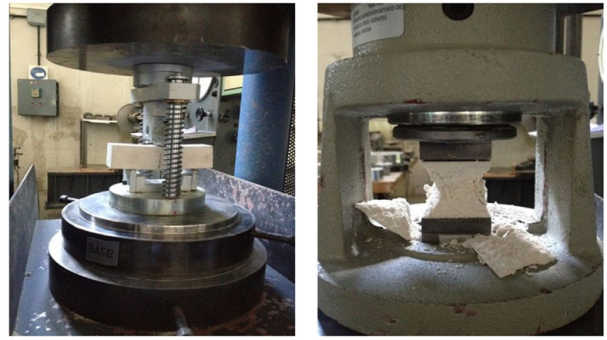


FIGURE 11. Mechanical strength tests on prismatic specimens.

py-high, freshly mixed material holds its shape (see photographs reproduced in Figure 10).

4.2 Mechanical strength

Both compressive and flexural strength (Figure 11) are among the parameters most routinely tested

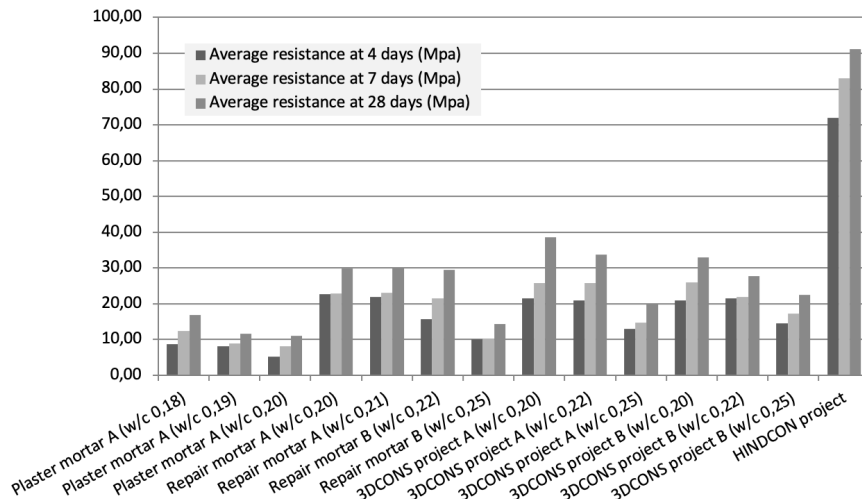


FIGURE 12. Compressive strength test results for several materials.

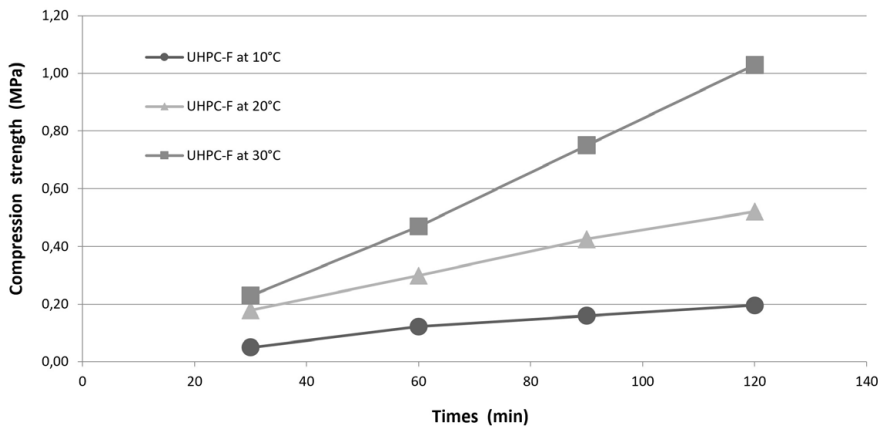


FIGURE 13. Early-age compressive strength in materials mixed at 10 °C, 20 °C or 30 °C (HINDCON Project).

in most projects. The usual procedure is to run the test on a 4x4x16 cm prismatic specimen, further to the standard for mortars (42).

Figure 12 gives the results for a number of materials tested, according to which strength varied substantially from 15 MPa for 28 d rendering mortars to over 90 MPa for an ultra-high performance concretes (UHPC). Further to those findings, whilst both materials are printable, their performance in other respects is not comparable.

These tests can be conducted after several days or at very early ages, from 30 min to 120 min, and also accommodate comparison among materials such as the ultra-high performance concretes (UHPC) shown in Figure 13.



FIGURE 14. Data collection on material settling.

4.3 Shape and stability

After extrusion-mediated layered deposition, data must be collected (Figure 14) to ensure that any settling of the material under its own weight lies within the pre-established tolerance interval.

Printing stability must also be verified on both horizontal and vertical substrates as the layers are applied. As shown in Figure 15 on horizontal substrates the aim is to ensure constant verticality and



FIGURE 15. Shape and stability measurement.

stability or at least to determine the number of layers that can be deposited before the operation must be momentarily suspended. Similarly, on vertical surfaces the aim is to establish the number of layers ensuring good substrate and interlayer bonding. Those three parameters - shape, stability and tolerances - define a material's buildability.

5. CONCLUSIONS

Determination of construction material printability is contingent upon standardising the definition of that term. Whereas for some authors printability is confined to material pumpability and extrudability, for others, such as the present authors, the definition should also cover a material's early-age buildability.

Caution must be paid to what definition of printability that is applied for the type of tests that are conducted. There is a substantial difference between assessing a material's rheology only and also assessing its mechanical strength, particularly at early ages.

One research question in this study was related to what standardized tests are applicable to analyse printability of cement-based materials in additive manufacturing for construction to verify a material's compliance with such requirements, and what are the results from performed tests. One conclusion drawn is that material printability might be established in keeping with its behaviour in three stages of AM: pumping, extrusion and deposition. Different tests in place for each stage have been used in projects implemented to date, applying standardised trials routinely applied to concretes and mortars.

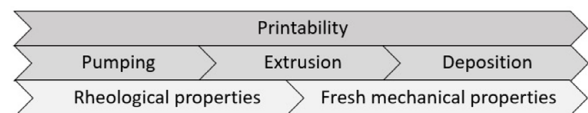


FIGURE 16. 3D printing stages and properties.

Another research question aimed for recommendations on the advisability of developing a standard test or suite of tests to ascertain printability, based on the study. As illustrated in Figure 16, the first criterion for establishing printability is conditioned by material rheology, whereas the second is defined in terms of fresh-state behaviour. The tests to be run should consequently be geared to assessing those properties.

The second criterion involves determining the values and characteristics demanded of a printable material, bearing in mind that the respective values are not conditioned solely by the definition of printability, but also by the printing system itself: requirements differ depending on the circumstances. For instance, the requirements for a material to be layered onto a vertical surface, calling for enormous interlayer and substrate bonding strength, cannot

be the same as for a material kept very fluid during pumping and subsequently admixed with accelerators immediately prior to extrusion.

Any number of products are currently available on the marketplace that are potentially applicable to additive manufacturing. Both they and the materials explicitly developed for 3D printing have been tested in a number of projects. The results are comparable in some cases although in most the findings on material printability are not reliable. However, the study illustrates several important experiences from printability testing.

In light of those considerations, this study identifies a need for a clear definition of construction material printability as well as for a test or suite of tests customised to each type of printing system that would deliver data with which to determine whether a material is printable.

AUTHOR CONTRIBUTIONS:

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