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“Printable profile of sustainable modified cementitious materials for additive manufacturing applications in digital construction”

by

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Preface

This report is made to fulfil the requirement of the M.Sc. by Research of the University of Hertfordshire. The purpose of this report is to present the preliminary research work done, which took place at the Civil Engineering & Built Environment Group - Innovative Construction Materials Lab for the research project with subject “Printable profile of sustainable modified cementitious materials for additive manufacturing applications in digital construction”.

This preliminary research work is focused on

- Literature review on the application of additive manufacturing methods in construction and the materials used, with emphasis on 3D Printing Concrete-3DPC.
- The development and evaluation of preliminary experimental procedures for the efficient investigation of the key 3DPC properties (extrudability, buildability and interlayer bonding strength).

This report is divided in four chapters.

- The first chapter is an introduction presenting all the technological innovations of the third industrial revolution which promises to transform the construction industry to a more sustainable sector. Special mention is made of the additive manufacturing methods in construction, the materials they use, their applications, the advantages and the existing limitations.
- The second chapter refers extensively to the 3DPC. It presents all the information collected from the literature review on mix design, the fresh, hardened properties of 3DPC and how these are affected by printing parameters.
- In the third chapter, all the preliminary experimental procedures that were applied in the laboratory and their results are presented.
- In the last part of this report, there is an extensive discussion and remarks about the initial part of the research project. Conclusions are drawing and guidelines are presented for the successful development of the research project.

Abstract

The construction industry is a conservative sector with limited innovation improvements compared to other industrial sectors. The construction industry has a considerably bad reputation in regard to its carbon footprint. Based on these facts, the construction industry is pressed to change and to be more sustainable by using technological innovations which, are provided by the so-called *third industrial revolution*. These innovations are related to the development of the material science, robotics, environmental and computer sciences. The application of these innovations promises to transform the traditional construction to a digital construction. A main aspect of digital construction is the adoption of digital fabrication methods. The most well-known digital fabrication method is the additive manufacturing method. The research interest about the additive manufacturing methods in construction industry is rising in the recent years. This project studies the use of cementitious materials in additive manufacturing methods with emphasis on printable behaviour of 3D Printing Concrete (3DPC). This study presents all the necessary knowledge gained from the relevant literature review about 3DPC and its applications. Also, conclusions are drawn about the printable properties of 3DPC through the analysis of preliminary experimental outcomes.

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- Chapter 1 -

1.1. Introduction

A significant transformation of various industrial sectors (automobile, aerospace etc.) has taken place in recent years. The industry digitizes the manufacturing of its products by introducing technological innovations related to the development of novel materials, robots, software packages, high network capabilities and new manufacturing processes (notably the additive manufacturing process). As a result, the industry optimizes the production, it reduces the environmental impact and it is generally compliant with the principles of sustainable development. This remarkable transformation of the industry is known as the “third industrial revolution” – the digital industrial revolution (*Wangler et al., 2016*).

The industrial sector that has been less affected by these technological innovations is the construction industry. The construction industry is a conservative sector, with its fundamental principles being almost unchanged for decades. As a result, the lack of adoption of the innovations, contributes to the low overall sustainability index. In particular, the construction has a high energy consumption ratio (up to 40% of the global energy consumption), produces high amount of greenhouse emissions (38% of the global production and a high level of injuries in construction sites (*Agustí-juan and Habert, 2017*)). Based on these facts, the construction industry needs to shift and change some previously thought “unchallenged” principles.

In this chapter, the literature is reviewed in the area of technological innovations that aim to transform the traditional construction. The chapter focuses on the digital fabrication methods in construction, with emphasis on the additive fabrication methods. Also, the types of materials and the printing techniques are presented, used by additive fabrication methods. Finally, a reference about the limitations and the advantages of additive fabrication methods in construction is discussed.

1.2. Digital Construction

It is estimated that the construction industry is responsible for 40% of global energy consumption, 38% of global greenhouse gas emissions, 12% of the global water consumption and contributes to 40% of the total waste generated by the newly developing countries (*European Construction Technology Platform, 2005; De Schutter et al., 2018*). According to these figures, the construction industry has the largest environmental impact compared to other industrial sectors.

Based on the above figures, efforts are being made for the construction industry to become more sustainable. In more detail, the United Nations Conference held in Istanbul in 1992 has adopted a framework of guidelines, called Agenda 21 to develop and improve the global construction industry by 2021. These rules are related to different sectors of the construction industry, such as, construction management, construction materials, consumption of resources, etc. (*Nations, 1992*). Also, in the European Union, the European Construction Technology Platform was established a strategic research agenda (SRA) on 2005 for a sustainable and competitive european construction sector by 2030. The SRA recommends the construction to be the be increasingly client-driven, sustainable and knowledge-based. These goals are supported by research domains dealing with materials and technology, industry information and service in construction. (*European Construction Technology Platform, 2005*). (*European Construction Technology Platform, 2005*). In the UK, a joint strategy called ‘Construction 2025’ was established in 2013, where the British construction sector and Government are working in a partnership aiming to achieve 50% reduction in greenhouse emissions in the built environment, 33% reduction in the cost of construction (initial and whole life cost) and 50% reduction in the overall time from inception to completion by 2025. In order these goals to be achieved, three priorities have been established: (i) smart construction and digital design, (ii) low carbon emissions and sustainable construction, and (iii) improved trade performance through the effective research and innovation. (*Department for Business Energy and Industrial Strategy, 2013*).

Unfortunately, the construction industry is a conservative-traditional sector with high levels of turnover, which has difficulties to adopt innovations. The construction sector invests relatively little in research and development with low industrialization of construction processes, poor collaboration and data interoperability (*De Schutter et al., 2018*).

Although, there is a difficulty for stakeholders in the construction industry to make immediate progressive decisions for significant changes in the sector, academics and certain companies have highlighted the need for radical changes. These changes are based on the principles of the third industrial revolution - the digital one, transforming the traditional construction to digital construction concept.

The term digital construction is aimed at all the digital technological tools (Figure 1) used to improve the process of delivering and operating the built environment, making it more efficient, safe and more collaborative. Some of these tools are:

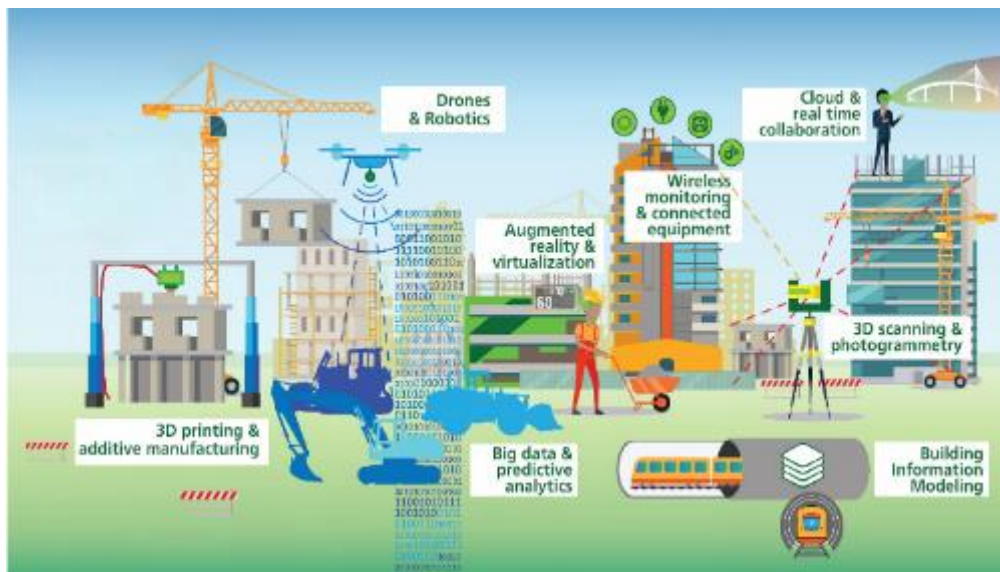


Figure 1 Digital Construction Tools (Digital Constructions, 2019) .

- The **optimal design** (Architectural and Structural) is based on the application of advanced parametric-design software by using advanced mathematical or physical models (genetic algorithms, heuristic algorithms, etc.). These algorithms approach the best solution in terms of optimal architectural/structural design and sustainability criteria (cost-efficient, embodied greenhouse emissions, energy consumption etc.). In most cases, solutions proposed for this kind of design are characterized by the shape complexity (see Figure 2) and composite / multimaterial matrices, giving hybrid behaviour to the various structural elements (Figure 3) (De Schutter et al., 2018).

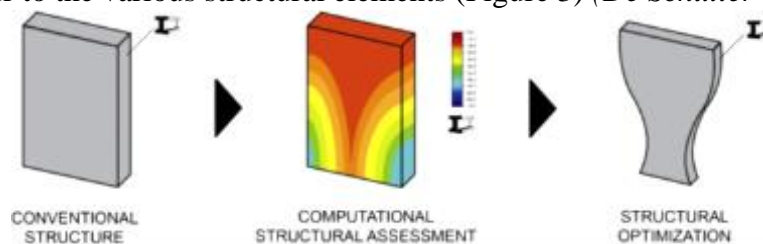


Figure 2 Shape optimization using advanced computational structural analysis. Aims the minimization of the environmental impact (embodied greenhouse emissions) through the material reduction (De Schutter et al., 2018).

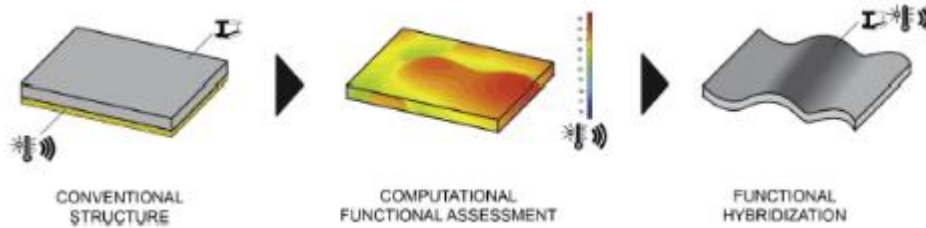


Figure 3 Shape and material optimization-functional hybridization (De Schutter et al., 2018).

Conventional manufacturing methods are unable to efficiently create these structures and they are gradually replaced by more efficient manufacturing methods (Figure 4), such as **the digital fabrication methods.**

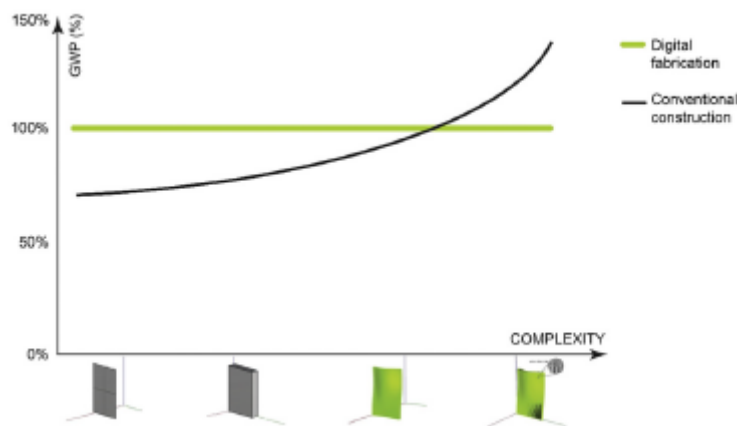


Figure 4 Complexity-related environmental advantage of digital fabrication vs. conventional construction in terms of % Global Warming Potential (De Schutter et al., 2018).

- The **digital fabrication methods** create objects directly from the design stage (digital file) to the manufacturing stage by using automated processes (without human involvement). Digital manufacturing methods have revolutionized various industrial sectors. Gradually, they are adopted by the construction industry, with the well-known additive manufacturing process or 3D-printing process. A detailed reference is made to the next paragraph 1.3.
- The design and development of new, innovative sustainable materials, capable of being used by digital fabrication applications. Important examples are the use of concrete as a printable material (3D printing concrete-3DPC). A detailed reference is made in Chapter 2.
- The use of **drones and UAV's** to collect the necessary information during the planning and on the site.
- **Effective collection and management of real-time information data** Effective collection and management of real-time information data using advanced communication networks (wi-fi, 5G etc.) and B.I.M. (Building Information Modeling) platform. As a result,

remarkable changes in planning and construction management, from design to construction and even further to maintenance, rehabilitation and recycling (Figure 5) (Sakin and Kiroglu, 2017).

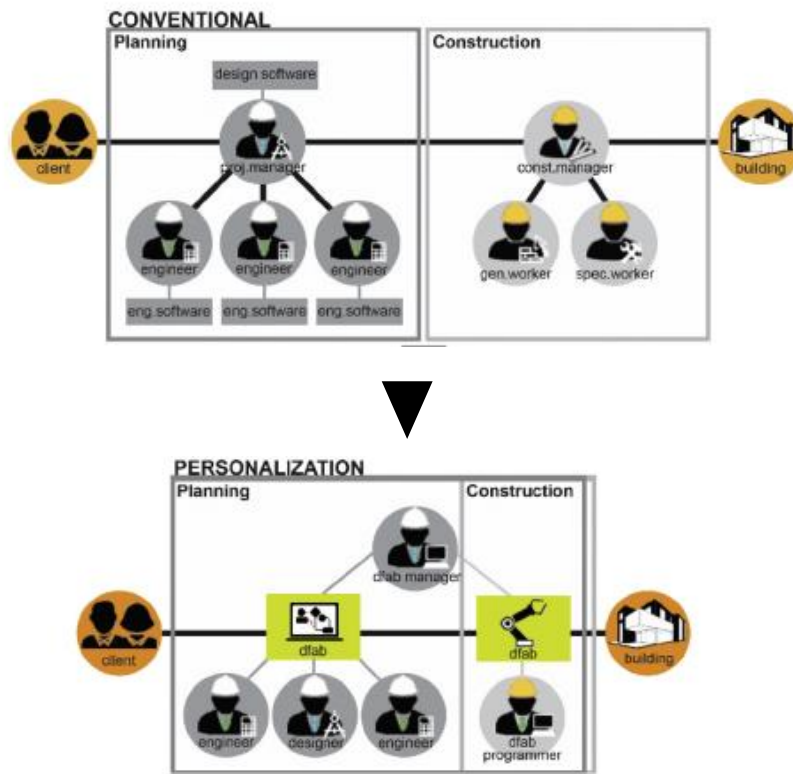


Figure 5 Evolution of traditional manager to a digital fabrication (dfab) manager (De Schutter et al., 2018).

1.3. Digital Fabrication Methods

The term digital fabrication methods refers to automated manufacturing methods, which use digital tools extensively, in the manufacturing process. These methods are widely applied in many industries (aerospace, automotive, and healthcare industries) with great success, thus upgrading the production process with the following main benefits (Ranjha, Kulkarni and Sanjayan, 2018; Romain de Laubier, Marius Wunder, 2018):

- Reduction of production time and cost (i.e. eliminating the formwork etc.).
- Creation of complex shape, multifunctional and multi-material elements.
- Reduction of accidents-injury rate (due to autonomous construction process with little human involvement).
- Reduction of environmental impact (reduction of waste materials, reduction of energy consumption etc.).

Initially, implementation of digital fabrication methods in the construction industry focused on the process of shaping and bonding sheets of material to form an object by using CNC technologies and create multiform facade buildings. Important examples are the Guggenheim Museum at Bilbao (Spain-1997), the Ray and Maria Stata Center (M.I.T. campus-2004) (Figure 6), etc.(*Buswell et al., 2007*).



Figure 6 Examples of digital fabrication process in construction based on CNC technologies. Guggenheim Museum at Bilbao (left) and Ray and Maria Stata Centre (right) (*Buswell et al., 2007*).

Today, there is a growing interest in the additive manufacturing methods, commonly referred to as 3D printing. These methods are based on the creation of components in a layerwise fashion directly from a digital file. The additive fabrication methods that have been researched and applied in the construction industry are differentiated by the following:

1. Printable materials
2. Printing system
3. Printing technique

1.3.1. Printable materials

Materials that have been studied and used for additive fabrication method applications in the construction industry have printable properties and cover a wide range of types and forms. These materials have been observed to have less environmental impact than the traditional (LCA- Life Cycle Assessment), mainly because the quantity of used materials and the quantity of waste materials are reduced during the construction process(*Agustí-juan and Habert, 2017*).

The predominant type of materials used, are the cementitious materials that have been modified for printable applications, followed by steel, stainless steel and aluminium. Also, polymers

natural or artificial, geopolymers and other natural materials (mainly printable clay) have been used (Clare Scott, 2017).

It has been reported in literature that the most prevalent forms of application of these materials are the paste and gel form. Particularly, for cementitious materials is mainly the paste version is used, while, for polymer the gel version is used. In addition, there are applications that use materials, as powders and as foams (3D printing polyurethane foam Batiprint 3D)(Figure 7)(Furet, Poullain and Garnier, 2019).



Figure 7 Application of 3D polyurethane printing foam (left) for construction of a dwelling house by using the Batiprint3D technique. The 95 sq. meters printed house is located in Nantes (France) and took only 54 hours to build (right) (Furet et al., 2019).

1.3.2. Printing Systems

The printing systems that have been applied up to date are:

- **Gantry:** The gantry is the frame structure that supports the printer head along the X / Y and Z axis. A gigantic gantry printer is used to create structural elements, as well as entire houses (Figure 8). Significant projects using this system are Contour Crafting (University of South California), 3D printing concrete (Loughborough University) and D-Shape (Labonnote et al., 2016; Sanjayan, Nazari and Nematollahi, 2019) .

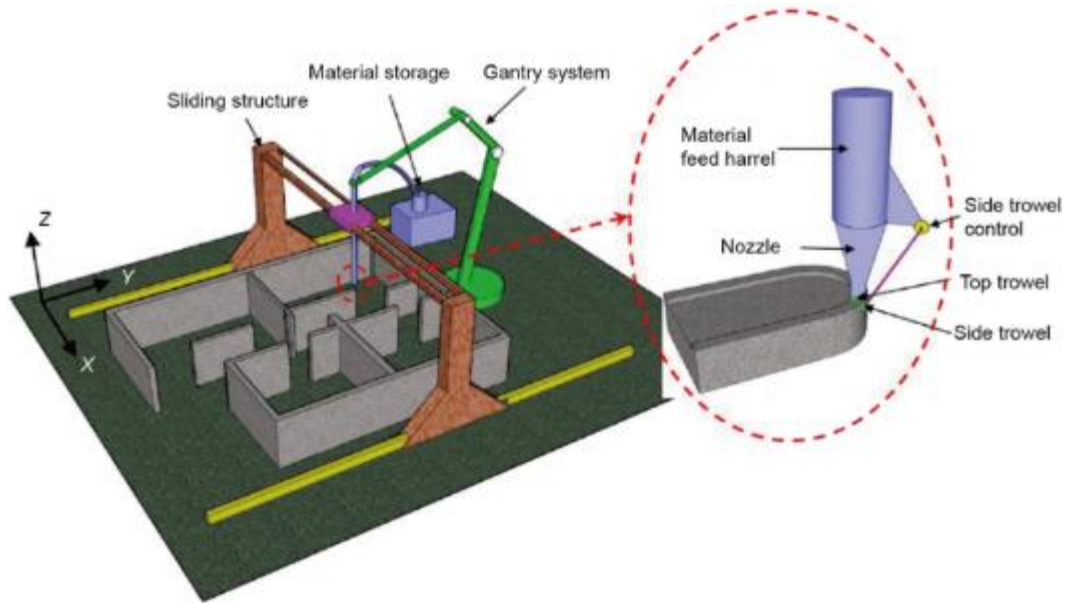


Figure 8 A Gantry 3D printing system for construction of a house (Sanjayan, Nazari and Nematollahi, 2019).

- **Cable-suspended platforms:** A cable suspended platform consisting of a printer head attached to an external frame using multiple cables. The printer head is controlled by motors that can extend or retract the cables in a fully automated manner. This system offers many benefits in terms of size, workspace area and transfer. Significant applications of the platform are the World Advances Saving Project (WASP in Italy) by using cementitious and natural materials. Also, a great application is the MIT-Media Lab project 'Spider Bot', which it extrudes expanding foam (WASP-BigDelta, 2012; Labonnote *et al.*, 2016).



Figure 9 WASP-Big Delta project. Full size 12m building out of mud and clay (WASP-BigDelta, 2012).

- **Swarm Approach:** This idea was first reported by Pegna in 1997, who suggested the creation of small robots able to build entire constructions (Labonnote *et al.*, 2016). Today, this method is directly applicable to the additive manufacturing process by creating small 3D printers running on wheels (Figure 10). An important project, based on this concept, is the

IAAC 'minibuilders' project, where three small robots were created: the foundation robots, vacuum robots and grip robots - small printers with four rollers, which are clamped on the structures and build these structures from foundation to upward by printing layers of material, while they are holding themselves on the layers they just printed. The printing material used for this project was clay (*Iaac-Institute for advanced architecture of Catalonia, 2016*).



Figure 10 Minibuilders by IAAC (*Iaac-Institute for advanced architecture of Catalonia, 2016*).

-Multi-purpose robots: Multi-purpose robots: A large number of applications of digital manufacturing methods in construction use robots or robotic arms. Robots are used either as printers (by extruding materials by themselves) or as ancillary constructors (such as moving and putting structural elements over the treated area and spreading several adhesives during the printing process, etc.). The main advantage, compared to the other methods, is that it performs several degrees of freedom. Interesting examples of robots in manufacturing are the creation of columns with the technology of slipforming (Smart Dynamic Casting - ETH Digital Fabrication) by extrusion self-compacting concrete (Figure 11c)(*Lloret et al., 2017*), the construction on- site houses by using 3D printing robots (*Cazza Construction*) (Figure 11b) and the NASA project of long-term human habitats for deep space and permanent outposts for the Moon and Mars, by using a robotic mobility platform (Figure 11a)(*Howe et al., 2014*).

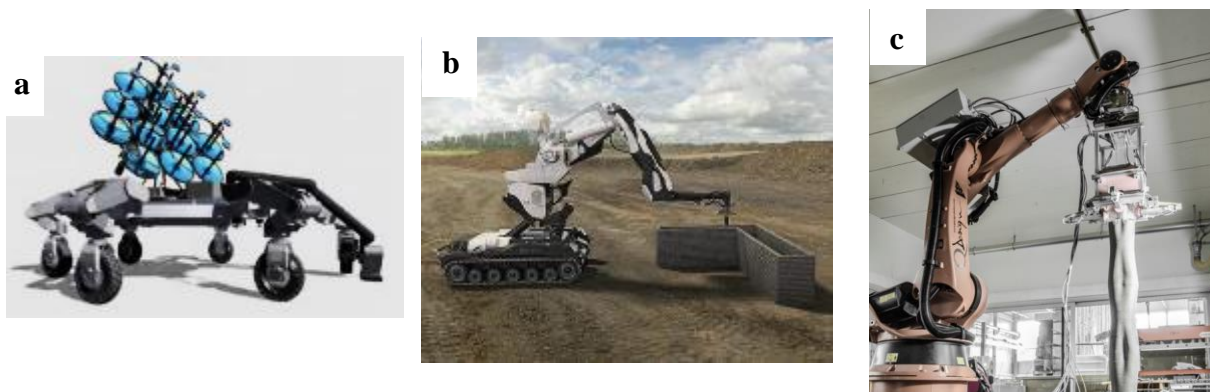


Figure 11 a) Robots application in construction ATHLETE robot with FACS Solar Concentrator for lunar habitation. (*Howe et al., 2014*) b) Cazza Construction- X1 3D printing robot and c) Smart Dynamic Casting- Slipforming (*Column Lloret et al., 2017*) (right).

1.3.3. Printing Technique

The main printing techniques which have been applied in the construction are:

- **Binder Jetting:** This technique of additive manufacturing, injects a binder, layer-by-layer, over a powder bed. The binder glues each powder layer with the other. This process is repeated until a 3D object is built. Based on this idea Enrico Dini - a pioneer and founder of D-Shape (today Monolete UK), created a gantry 3D printer, that injects a solution of magnesium oxide over a sand layer. As results, the binder reacts chemically with sand and creates a sandstone complex structure with notable strength properties. An important application of this method is the construction of the first printable bridge (total length of 12 m and a width of 1.75 m) in 2016 (Figure 12). For the bridge micro reinforced concrete was used. This bridge was built off-site by the Institute of Advanced Architecture of Catalonia-IAAC and Mr. Enrico Dini (*Markopoulou,Rodrigo Aguirre et al. 2016; Delgado et al., 2018*).



Figure 12 The first printed concrete pedestrian bridge by using binder jetting technique. Located in the urban park of Castilla-La Mancha in Alcobendas, Madrid (Markopoulou,Rodrigo Aguirre et al. 2016). .

- **Powder Bed Fusion (PBF):** The powder bed fusion technique is based on the selective fuse of the powder bed using thermal treatment. This is achieved by using a laser or an electron beam. This technique is applied to polymer and metallic materials. In construction, the most notable applications are the creation of optimized metal nodes for connection of struts and cables by ARUP (Figure 13a) and the construction of a complex cladding for the Bevis Mark Building in London by Skanska using metal and polymer materials (Figure 13b). A major drawback of this technique is that is suitable for small parts only(*Delgado et al., 2018; Buchanan and Gardner, 2019*).

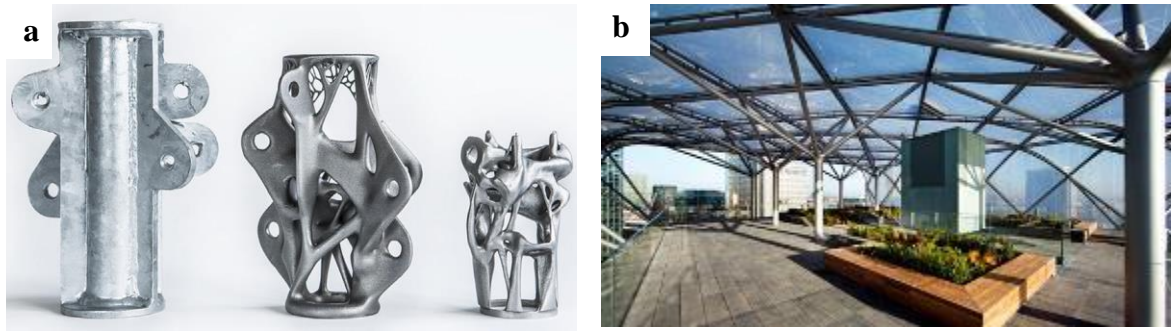


Figure 13 Powder bed fusion applications in construction: **a)** optimized metallic nodes by Arup and **b)** complex cladding by Skanska (Buchanan & Gardner, 2019; Delgado et al., 2018).

- **Direct Energy Deposition:** This technique focuses only on applications using metallic materials. DED is an additive manufacturing process, where focused thermal energy is used to fuse materials by melting them as they are deposited (as opposed to PBF where there exists powder bed). The form of the materials used may be powder or wires (WAAM). Materials are fused using arc welding tools, such as gas metal arc welding (GMAW), gas tungsten arc welding (TGA) and plasma arc welding (PAW). Significant application of this technique in construction is the creation of the first 3D printed metal bridge - MX3D (Tim Geurtjens, 2015) (Figure 14) . The bridge has a width of 2.5m and span of 10m, it was designed and built in collaboration with ARUP, Imperial College London, Alan Turing Institute and the MX3D manufacturing company in 2018. The technique applied is wire and arc additive manufacturing (WAAM) using a 6-axis robotic welding arm (Delgado et al., 2018; Buchanan and Gardner, 2019) .



Figure 14 The MX3D bridge (Tim Geurtjens, 2015).

- Material Extrusion and Deposition: With this technique the material is extruded by a nozzle and is deposited in a layerwise fashion onto a substrate. Each material layer supports its own weight and the weight of each subsequent layer. This technique is the most applied in construction. In recent years, the research interest in this technique is constantly growing. In particular, the number of journal articles published and conference proceedings over the period 2013-2016 have almost doubled, compared to the first 16 years of research (1997-2013), a major degree of development, which continuously grows (Figure 15) (Tay *et al.*, 2017). Based on this technique, the most notable automated systems are Contour Crafting (University of Southern California) and 3D Concrete Printing (University of Loughborough).

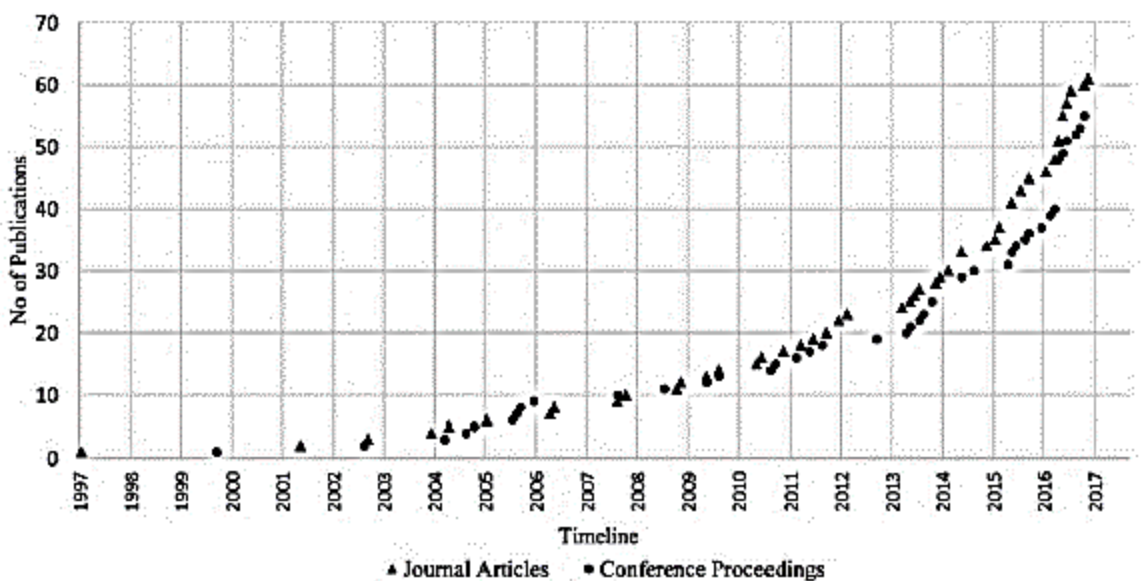


Figure 15 Trend of publication in the research field of material extrusion technique in construction from 1997 to 2017. (Tay *et al.*, 2017).

The **contour crafting system** is one of the first 3D printing systems invented by Dr. Behrokh Khoshnevis, University of Southern California in 2004. Contour crafting is a gantry-based system that extrudes material in a layer-by-layer way, which is shaped by trowels. The system uses trowels to create smooth and accurate surfaces (Figure 16). The trowels are moving at different angles in order to create various complex shapes. The system uses clay, concrete and ceramic materials to build large scale objectives. An important parameter of this system is the printing pattern which it is used. The printing pattern consists of an outer shell-wall that is attached and supported by an inner shell through a sinusoidal printing path (Figure 16). Currently, this system focuses on the use of ultra-high performance concrete UHPC (a mix of ordinary portland cement, supplementary cementitious materials, admixtures, fine aggregates and fibers) for the construction of entire houses.



Figure 16 Contour Crafting: Trowel system (Tay et al., 2017) (left) and printing pattern (right).

The **3D concrete printing** system is similar to contour crafting, but the main difference is that it does not use trowels and the printing patterns vary according to each application. The system was invented at the University of Loughborough UK in 2007 and uses mainly cementitious materials (Figure 17). Today, a lot of research groups are working on this concept, such as the Eindhoven University of Technology (3DCP project), Dresden University of Technology (CONPrint3D project), Nanyang Technological University (SC3DP project) etc. are focusing their interest on research and development of appropriate printable cementitious materials for 3D printing applications using gantry or robotic printing systems(Le et al., 2012).



Figure 17 A 3DCP element (University of Loughborough, UK) (Le et al., 2012).

1.4. Applications of additive manufacturing method in construction

The application of the additive manufacturing method in the construction tends to change the traditional way of designing and constructing a structural element or entire structure. In recent years, it has been recorded a significant increase of additive manufacturing applications (Figure 18) in construction since the method inception in 1997 (Buswell *et al.*, 2018).

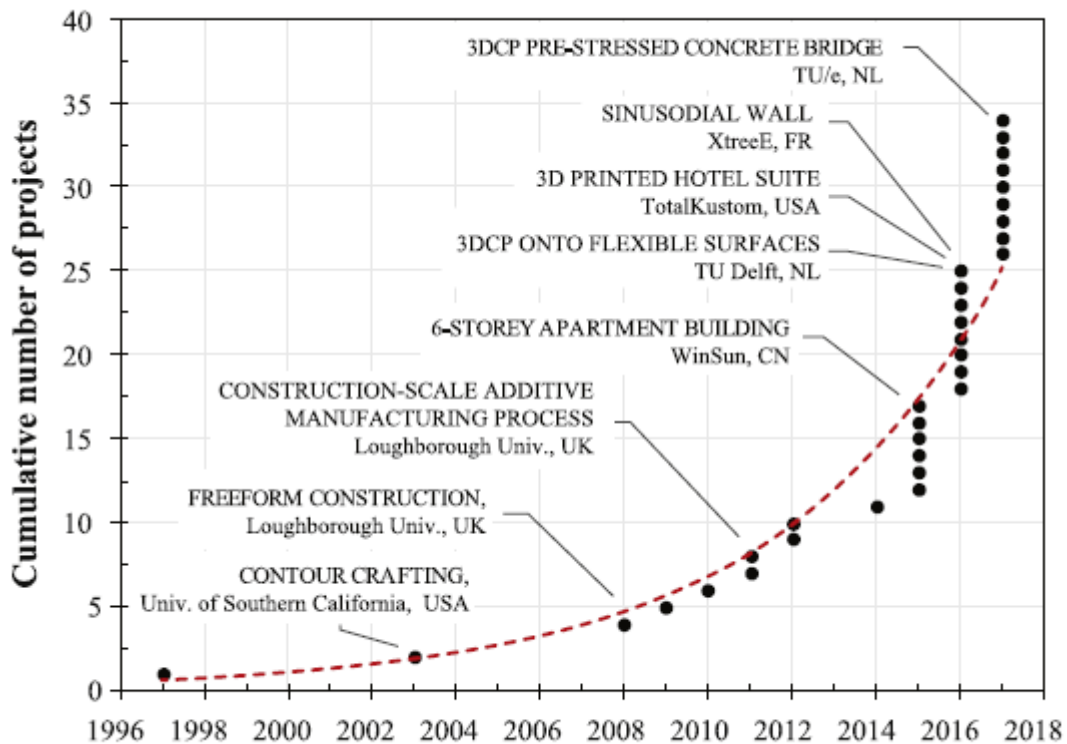


Figure 18 The rise of additive manufacturing method in construction applications since 1997 (Buswell *et al.*, 2018).

The most important innovation that has been introduced by the application of additive manufacturing methods in construction is the creation of sustainable complex shape-multimaterial printed structures.

1.4.1. Structural Complexibility

The creation of complex shape objects was difficult (complex moulds) and prohibitive (high production costs) a few years ago. Today with the aid of manufacturing methods in various industries, the creation of 3D complex structures is feasible and widely used. Complicated printing patterns range in size from a few μm to cm (mesostructures) have the potential to give new mechanical (stress-based topology optimization) and physical properties (multi-physics topology optimization) to objects (i.e. mechanical meta-materials). These printing patterns reflect topologies based on natural structures (i.e. material lattices), biological structures-

biomimicry (i.e. bone structures, shell structures) and generally structures that can give novel properties to the object.

In the construction industry, several examples of building structures based on structural complexity are noted. An important example is the construction of columns and beams, essentially mimicing the anatomy of a bone (Figure 19) using a cable-suspended printing platform which extrudes 3D printing concrete (cementitious materials and fibers). These structural elements consist of an outside dense shell (dense printing pattern) and internally display a hollow and spongy topology (based on collagen bone patterns) (Figure 19). As a result, these structural elements have a self-reinforced structure, more resilient than the traditional approach (SCG, 2016; Duballet, Baverel and Dirrenberger, 2017).

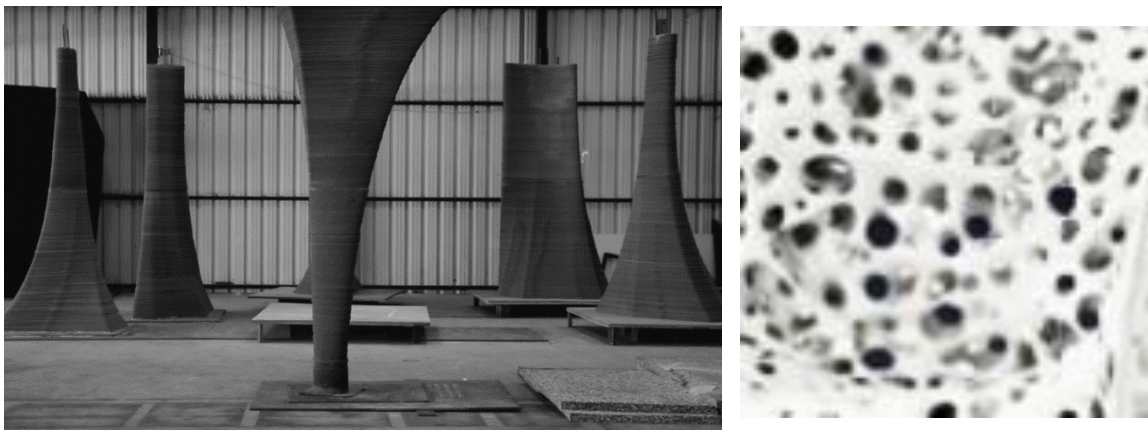


Figure 19 Siam Cement Group (SCG) columns mimic a bone anatomy (SCG, 2016) (left) and the inner bone structure (right).

1.4.2. Multimaterial Design

An important advantage conferred by the use of additive manufacturing methods in manufacturing is the creation of multimaterial-multifunctional structures. Printers that carry more than one printing head can print more than one kind of materials at the same time at different places (material customization by location). The French company XtreeE, based on this capability, built a truss-shaped pillar (Figure 20) by using optimal structural and topological design. The pillar was made of two types of concrete, 3D concrete for the shell and UHPC concrete for the core. UHPC concrete gives structural stability by replacing steel while the 3D concrete printing shell achieves the optimal topological and aesthetic result (Gosselin et al., 2016; Gaudillière et al., 2019).



Figure 20 XtreeE pillar in Aix-en-Provence, built in France 2016 (left) and section of the pillar (right) (Gaudillière et al., 2019).

In the case of the XtreeE pillar, UHPC concrete (mix of concrete and steel / polymers fibers) acts as a reinforcement of the structural element. In the context of 3D printing concrete reinforcement, numerous research groups and companies present different approaches. The most important approaches are:

- Mesh mould by ETH Digital fabrication Lab: In this approach, a complex reinforcement mesh (made of polymers or metallic materials) is created initially (Figure 21), and a self-compacting concrete is placed afterwards (Gramazio Kohler Research, 2014).

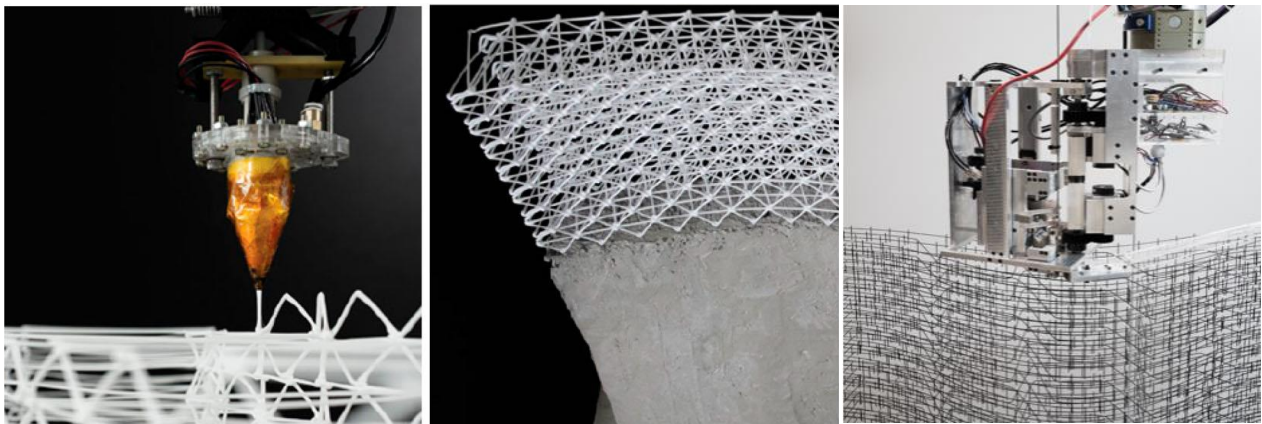


Figure 21 Mesh-mould reinforcement approach (Gramazio Kohler Research, 2014).

- Another approach is the placement of an extrusion gun to the back of the print head-nozzle. This gun extrudes fibers when the printing nozzle is printing aiming to apply tensile strength in the vertical direction. A typical example is the hybrid reinforcement printing system at Nanyang Technological University in Singapore. This system extrudes continuous steel cable and short PVA fibers as reinforcement for hybrid 3D-printed geopolymers (Figure 22)(Tay et al., 2017; De Schutter et al., 2018; Lim, Panda and Pham, 2018).

Unfortunately, the approaches for vertical and horizontal reinforcement in 3D printed concrete elements are not satisfactory and more research is needed on this direction.

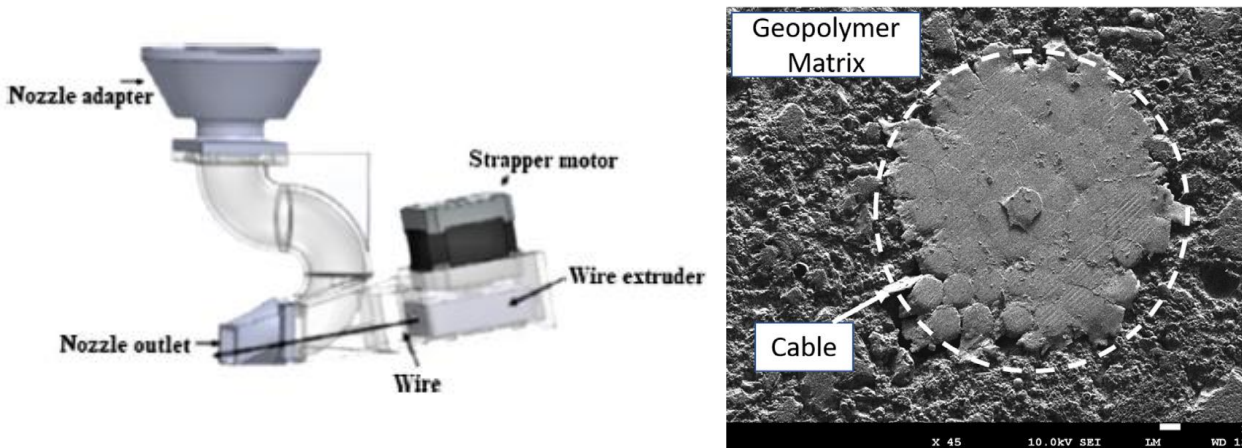


Figure 22 NTU- Hybrid reinforcement printing system (left) and SEM image of the hybrid reinforced geopolymer (right) (Lim, Panda, & Pham, 2018).

1.4.3. Sustainability

In the literature it is argued that the application of additive manufacturing methods in construction improves significantly the sustainability index of structures in terms of waste material reduction, construction cost and construction time. Specifically, the XtreeE pillar construction mentioned above, it was compared to the traditional method of construction and it was found that with the traditional method it requires 16.2% more construction time and 22.2% more workforce. Also, for the construction of walls, research has been carried out and it has been noted that the cost and the construction time are significantly reduced by applying additive manufacturing methods (Figure 23) (Buswell et al., 2007; Gaudillière et al., 2019)

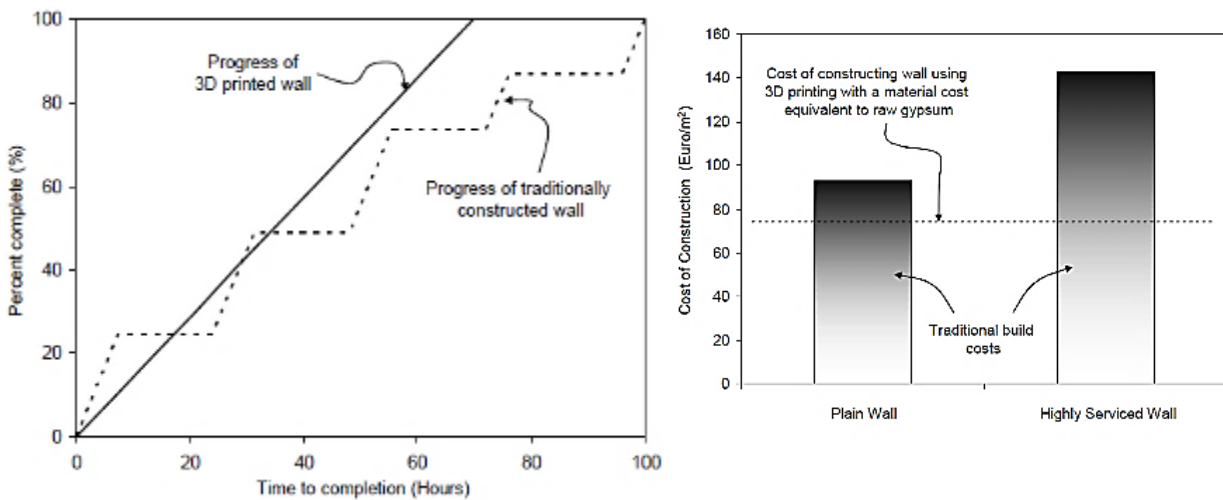


Figure 23 Time to completion (left) and cost of construction (right) of a 3D printed wall (Buswell et al., 2007)

- Chapter 2 -

2.1. Introduction

Cement-based composites are the main materials used for extrusion additive manufacturing applications in construction. These composites are a mix of cement, fine aggregates, admixtures, secondary cementitious materials, etc. capable of extruding through a nozzle (*Soltan and Li, 2018*). The 3D printing concrete must have properties capable of being flowable prior to deposition, to be both extrudable and buildable at the deposition, and rapidly harden after deposition. The properties of these new type of cementitious composites not only depend on the traditional factors (mix design and curing process/time) but also on the printing process parameters (printing speed, nozzle size etc.). For this reason, many researchers give the name "digital concrete" of this new type of concrete to emphasize the contribution of printing system and printing technique on the 3DPC properties (*Wangler et al., 2016*).

The 3DPC is an anisotropic material with a layered structure. This can create a considerable amount of challenges at multiple levels: material characterisation; structural design and structural performance. The current testing and design standards consider concrete as a monolithic material (*Putten and Schutter, 2019*). Provided that care has been taken during manufacture, monolithic concrete is assumed as a homogeneous composite with well defined (to a large extent) mechanical and durability properties. Nonetheless, that is not the case for 3DPC where there are neither characterisation testing standards nor structural design rules. Consequently, researchers are focusing on decoding and understanding the properties of this new composite. Looking at the literature, one can find a number of suggested innovative experimental procedures and methodologies aiming to understand the printable behaviour and the properties of 3DPC.

This chapter presents the literature review on 3DPC. Initially, reference is made to the mix design in the printable properties that must exist in fresh state, the printability criteria and the methods used to investigate the printable properties. Also, it presents the influence of printing system features and printing patterns on hardened 3DPC concrete. Finally, reference is made to the hardened properties of 3DPC (flexural strength, interlayer bonding, etc.), to testing methods, and to ways of improving these properties.

2.2. Mix Design

The essential components of the mix design of 3DPC (Appendix) are:

1. Ordinary Portland Cement. The most used OPC type in the 3DPC is CEM I 52.5. Other types of cement used are ASTM C150 Type II (*Kazemian et al., 2019*), CEM II 52.5 (*Mechtcherine et al., 2019*), CSA (Calcium Sulfoaluminate Cement)(*Khalil et al., 2019*), and Class H oil well cement (*Ma and Kawashima, 2019*).
2. Fine Aggregates. For 3DPC silica, fine aggregates with a particle size of 0.5-2mm are mainly used. There is a report (*Mechtcherine et al., 2019*) that a fine aggregate with a particle size up to 0.06mm has been used, but there is no reference which it has used aggregate with particle size larger than 2mm. Also, there are research projects which quartz fine aggregates, river sand (*Panda et al., 2019*) and recycled glass beads as fine synthetic aggregates (*Carlo, Carlson and Khoshnevis, 2012*) for more sustainable applications are used..
3. Clay. The main type that has been applied is attapulgite nanoclay , but there are reports of using palygorskite nanoclay (*Ma and Kawashima, 2019*) and bentonite (*Rushing et al., 2017*).
4. Fly Ash. In most reports fly ash is used. Some reports specify the Fly Ash to be of the type Class F (*Mechtcherine et al., 2019; Yu and Leung, 2019*).
5. Silica Fume. It is used almost in all cases without reference to be made to the size distribution, purity etc.
6. Admixtures. Numerous admixtures are used in 3DPC research. The most commonly used are Superplasticizers, High-Range Water-Reducing Agents (HRWRA) and Viscosity Modifying Admixtures (VMA)(*Bao et al., 2019*).
7. Fibers. There is an increasing number of works focusing on the application of fibers in the 3DPC concrete. The types of fibers used are HDPE (*Mechtcherine et al., 2019*) , PVA (*Bao et al., 2019; Yu and Leung, 2019*) and Polypropylene (*Doomen, 2016; Kazemian et al., 2019*) The fiber size in most cases is 6mm.

In the literature (Appendix) a wide range of different combinations of quantities are reported. To this date there is no evidence of a consensus in the research community for a standardised mix design methodology for 3DPCs.

2.3. Fresh properties

The lack of standardised tests for assessing the fresh properties of 3DPCs has resulted in a number of developed techniques reported in the relevant literature. Each approach proposes a different way to assess the properties relating to fresh stage. A common element of all techniques is that the 3DPC must be printable. Printable means that the materials is able:

- to extrude through a nozzle,
- to hold its shape during and after deposition of each layer and
- to gain the appropriate strength over the time supporting the subsequent layers.

These printable features of 3DPC in most applications are expressed as the three fresh properties of 3DPC: *Extrudability, Buildability and Open Time*.

2.3.1. Extrudability

Extrudability is the easiness and reliability for 3DPC to be deposited through a nozzle. In the work ‘Research Development in 3DPC: Cured-on-Demand with Adhesion Enhancement Delivery System’ (Verian, International and States, 2018) the extrudability is expressed as the ability of 3DPC to create a continuous filament. This property is approached qualitatively or quantitatively. In most works, the main focus is on the qualitative approach. Specifically, if the filament extruded through the nozzle retains its shape has no imperfections and discontinuities, then it is considered extrudable. Quantitative approach uses rheometers and flow tables to determine extrudability (Rushing et al., 2017; Rahul et al., 2019).

2.3.2. Buildability

This property refers to the ability of a printed 3DPC filament to sustain the weight of the upper filaments (in vertical orientation deposition), without shape deformation. The buildability determines the structural stability of the entire printed structure. As with extrudability, buildability is also qualitatively and quantitatively assessed. There are many methods and experiments which define buildability. The simplest method is to print layers (Tay et al., 2017) (Figure 24b), which are observed if they retain their shape under the weight of the upper filaments and the height of collapse of each filament is measured by using a ruler (Rahul et al., 2019). Because this method is expensive, and unsustainable due to waste materials, researchers adopted other methods, with the stacking plate to be the most predominant. *The stacking plate* (Figure 24a) is an experimental apparatus that simulates the layer-by-layer printed structure of

3DPC, uses different weights (these weights have a fixed value or simulate the weight of each filament) and measures the height reduction-collapse of each layer. There are also other apparatuses, such as the one used in the work ‘Cementitious materials for construction-scale 3D printing: Laboratory testing of fresh printing mixture’ (Kazemian *et al.*, 2017), the *cylinder stability test* (Figure 24c) is a semi-cylindrical device and measures the height collapse of the printed filament-cylindrical 3DPC sample under a constant pressure .

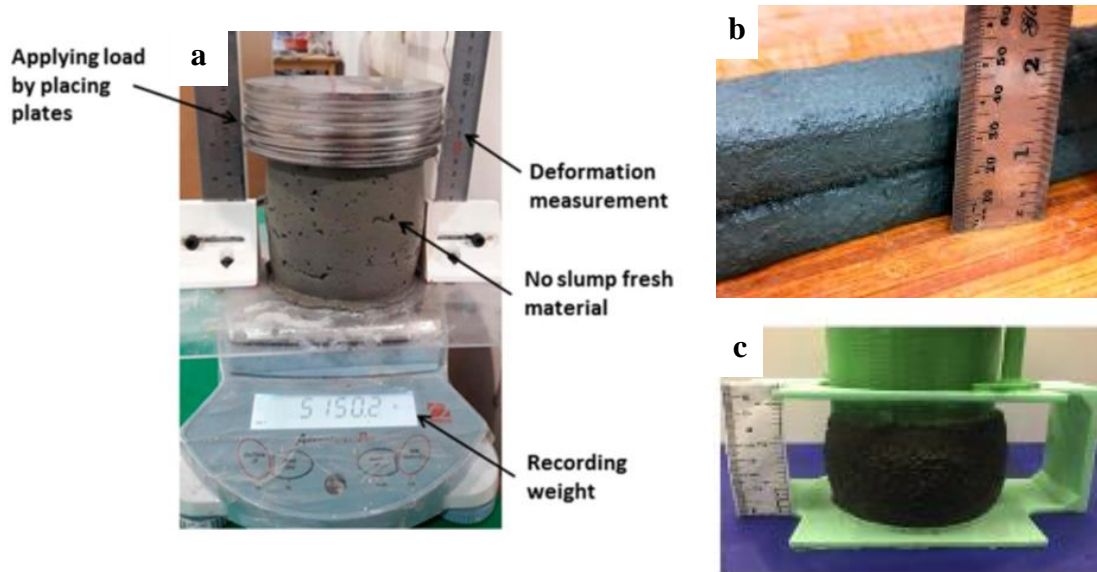


Figure 24 Buildability Tests: a) Stacking plate, b) Printing method (Rahul *et al.*, 2019), and c) Cylinder stability test (Kazemian *et al.*, 2017).

2.3.3. Open Time

Time is a variable that affects the properties of 3DPC. As it is known, concrete is a material with thixotropic behavior. That is, over time and with non-application of a shear stress the material will set. It is apparent that this is in direct correlation with the workability of the mix. The more the material sets, the higher the viscosity becomes. This behaviour can have a significant effect on the printability of 3DPC (extrudability and buildability). The time interval in which the 3DPC has stable printable properties (with small tolerance) is called open time.

In the literature, open time can also be found under the term ‘printable window’ (Kazemian *et al.*, 2017). Open time has two distinct limits: 1. Printability limit: the time when the 3DPC starts to be printable after mixing and 2. Blockage Limit: The time when the 3DPC cannot be guided out of printing nozzle. The printable window is determined using the stacking plate apparatus (the stability of 3DPC for specific time-intervals is checked to determine the

printability limit) and a Vicat setting time testing machine (the setting time diagram indicates the time when the deposited layer starts gaining strength to support subsequent layers deposited upon it).

Important for the understanding of the printable profile of 3DPC and its fresh properties, is the evolution of flowability of 3DPC over time, which is called flowability evolution (*Soltan and Li, 2018*). Figure 25 shows the idealized printable behaviour for 3DPC in terms of flowability evolution. The 3DPC must be extrudable before deposition for time period $0 \leq t < T_p$ (time of deposition), at the time $t = T_p$ must be extrudable and buildable and for $t > T_p$ only buildable.

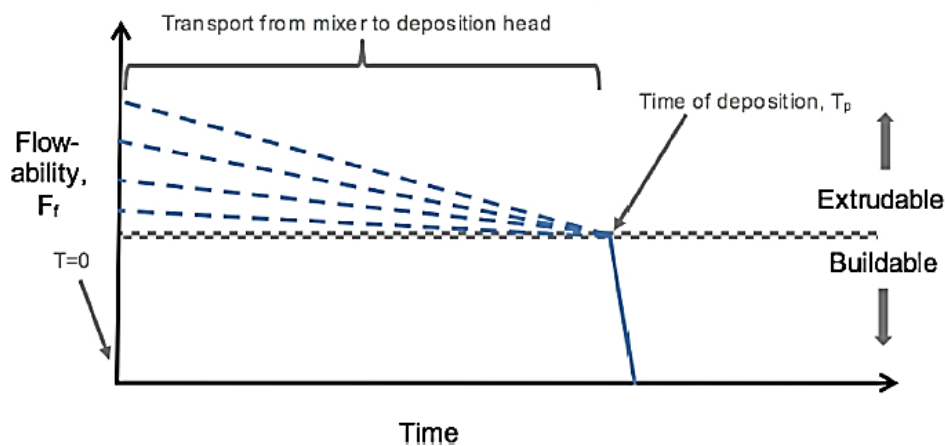


Figure 25 Idealized flowability behaviour of 3DPC (*Soltan and Li, 2018*).

But due to the thixotropic behaviour of 3DPC, the concrete after mixing begins to change phase from liquid to solid in unperturbed conditions. However, this particular group of materials (thixotropic materials) has the property when shear stress is applied, such as stirring-agitation, its viscosity is reduced and the flowability is increasing. Also, when the shear stress is removed, its viscosity is regain (the flowability is decreasing), this process is called rebuilding. Based on these, the proposed printable behavior of 3DPC is presented in Figure 26.

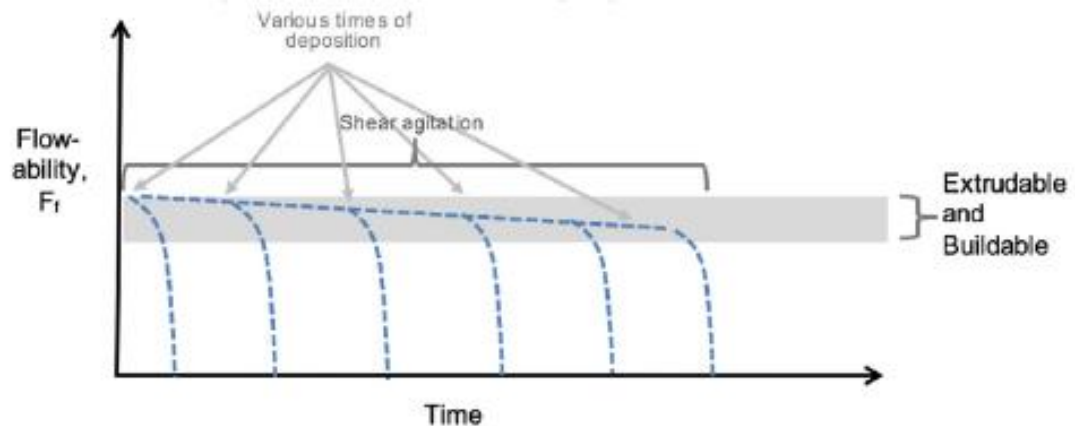


Figure 26 Proposed printable behaviour of 3DPC (*Soltan and Li, 2018*).

2.4. Printing Process

The printing process is an important field of research for 3DPC, because the 3DPC's properties (fresh, hardened and durability) are directly linked to the printing process parameters. Specifically, the parameters that have been recorded in the literature with the most important influence on the properties of 3DPC are:

2.4.1. Agitation System

The agitation system of the printing system has an influence to 3DPC. As mentioned earlier, it is important to control the thixotropic behaviour of the material. To achieve this in an automated extrusion printing system, various agitation methods are used. In conventional agitation methods, rheological state of 3DPC cannot be tested, controlled and modified during the agitation stage. As a result, a large amount of waste material is generated. For this reason, Ghent University (*De Schutter et al., 2018*) proposed a pioneering method of controlling the rheology of 3DPC by applying magnetic nanoparticles. Nanoparticles are controlled by magnetic fields and act as agitators by controlling the thixotropic behaviour of the sample.

2.4.2. Injection Channels

Substantial research is carried out in the field of application of various admixtures (i.e. accelerators, retarders, etc.) on 3DPC during printing stage. This is possible by modification of the printing head with special injection channels (Figure 27) (*Verian, International and States, 2018*). Also, injection channels are used, for the deposition of adhesive agents or other improvement agents aiming to enrich the hardened and durability properties of 3DPC. Major research in this field is carried out by Lafarge in France(*Esnault et al., 2019*).

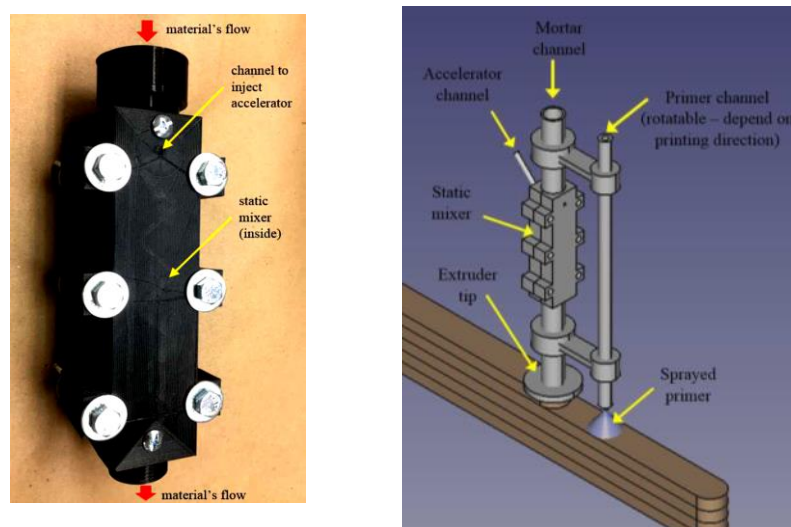


Figure 27 a) Injection channel and b) printing system with injection components(*Verian, International and States, 2018*)

2.4.3. Printing Speed

Printing speed is a parameter that greatly contributes to the structure and printable properties of 3DPC. The printing speed is typically measured in mm/s and determines the number of filaments deposited in the vertical direction and the time gap between them. It has been noticed that during the printing processes with low speed and long time gap, the samples have a lower interlayer bond strength between the layers in vertical direction (*Tay et al., 2019*). This is due not only to the cold joints which are developed, but also to the accumulation of voids (due to trapped air) between the layers (Figure 28). Also, the optimum printing speed must be determined, because at high printing speed, discontinuities on the sample can be observed thus reducing the extrudability (Figure 28) (*Tay, Li and Tan, 2019*).

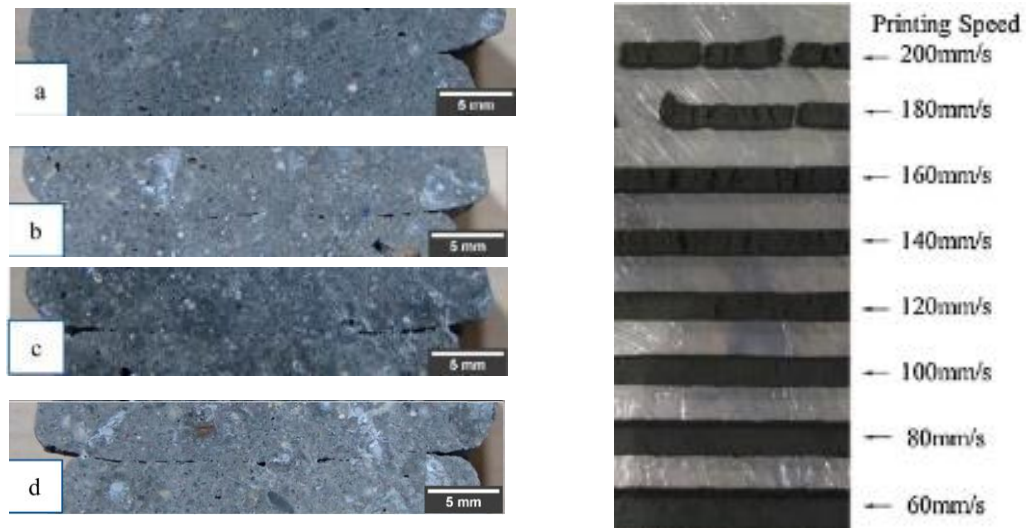


Figure 28 Samples printed at a) 1min gap b)5min gap, c)10min gap, d) 20min gap(right) (*Tay et al., 2019*), effect of printing speed on extrudability (left) (*Tay, Li and Tan, 2019*).

2.4.4. Nozzle Geometry

The geometrical characteristics of the print nozzle affect the properties of the printable 3DPC. The most essential property is the cross-section size of the nozzle. The size of the cross section of the nozzle, besides that it affects the extrudability of 3DPC, also affects the properties of the hardened 3DPC. In more details, in the work of ‘Discrete Element Simulations of Rheological Response of Cementitious Binders as Applied to 3D Printing’ (*Yang, Nair and Neithalath, 2019*) the optimal extrusion rheology is approached in relation to the nozzle section size, using discrete element models (Figure 29). Also, the Purdue University has produced 3D printing cementitious material samples with a 4.3mm nozzle cross section. These samples were examined by X-ray micro-computed tomography (micro-CT) and significant decrease in

porosity was detected (macropores and micropores), substantially altering the mechanical behaviour of the samples (Moini *et al.*, 2019).

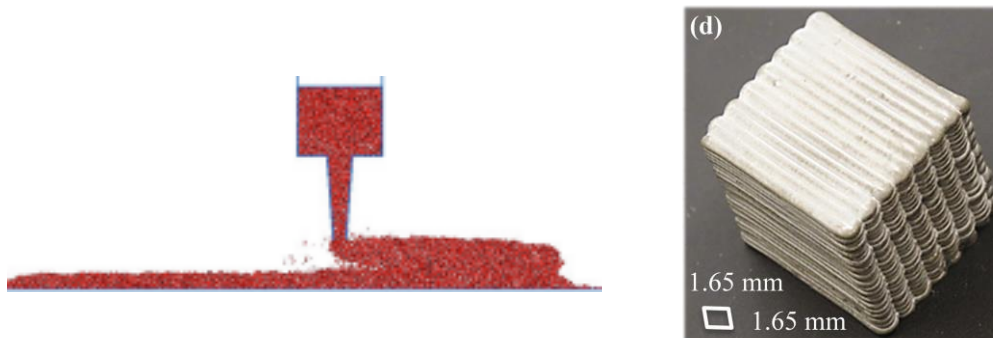


Figure 29 DEM simulation of 3DPC (Yang, Nair and Neithalath, 2019) (left) and 3D printed cement paste sample (right) (Moini *et al.*, 2019).

2.4.5. Printing Patterns

The ability of the printing process to print samples with different pattern-paths using one or more materials, as mentioned above, is a key advantage of additive manufacturing methods in industry. As far as the cementitious materials are concerned, it has been reported that the influence of printing paths on 3DPC properties are very important. Specifically, the report (Hambach and Volkmer, 2017) a significant improvement was observed in flexural (Figure 30), compressive strength (Figure 31) cement paste samples (reinforced with or without carbon fibers), by using different paths patterns.

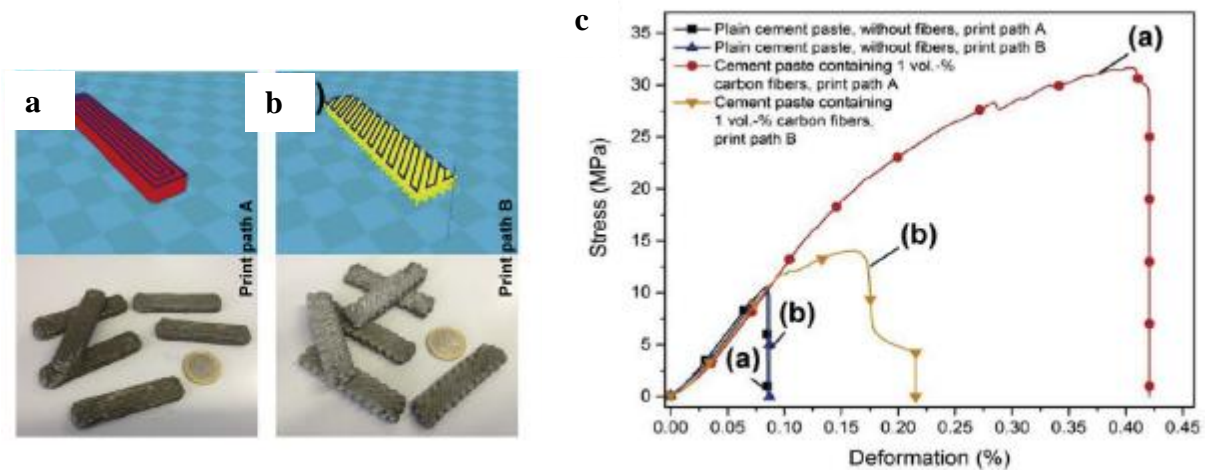


Figure 30 Printing paths (a and b) and 3-point flexural strength test plot (c) for 3D printed samples (with carbon fibres and without carbon fibres) for different patterns (Hambach and Volkmer, 2017).

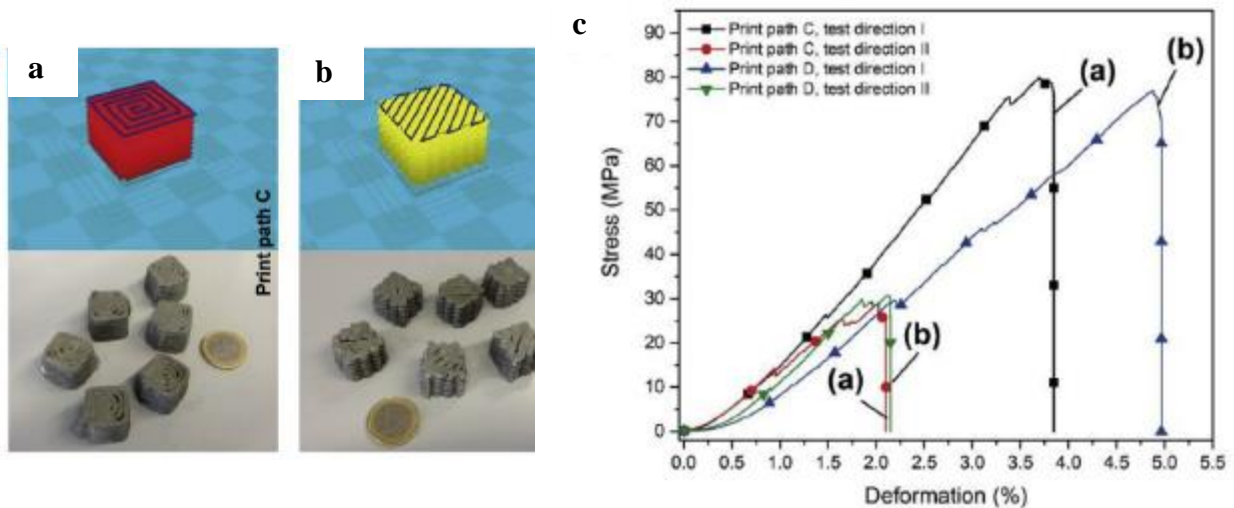


Figure 31 Printing paths (a and b) and uniaxial compressive strength test plot (c) for 3D printed samples (without carbon fibres) for different patterns (Hambach and Volkmer, 2017)

Also, in the same work composite cementitious materials were created, by using carbon-reinforced cement paste, mortar and different printing patterns (Figure 32). It was found that even in this case there were significant changes in the flexural strength of the samples.

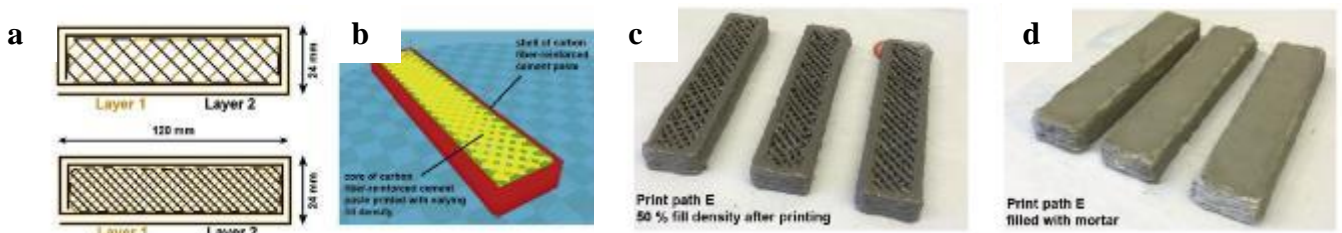


Figure 32 3D printed composite cementitious structures (Hambach and Volkmer, 2017).

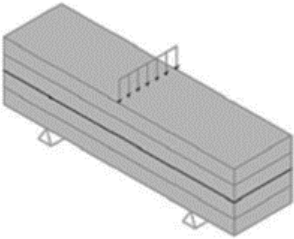

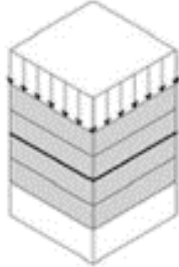
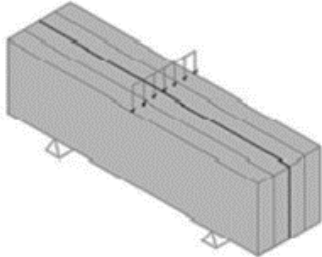
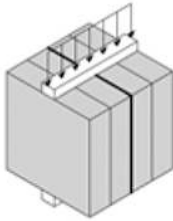
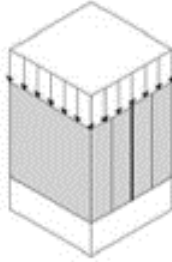
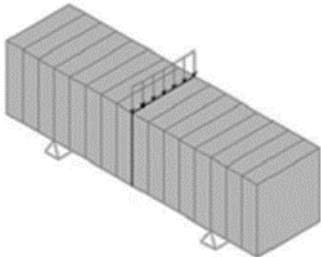
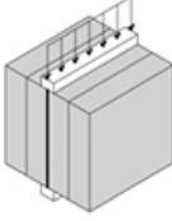
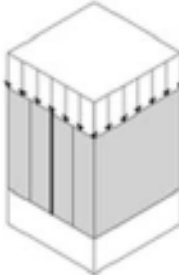
2.5. Hardened Properties

The main hardened properties of 3DPC, which have been extensively studied and recorded in the literature are: flexural tensile strength, compressive strength, splitting tensile strength and interlayer bonding strength.

2.5.1. Flexural Tensile, Compressive and Splitting Tensile Strength

Due to the anisotropic character of 3DPC, the values of flexural tensile strength, compressive strength and splitting tensile strength vary based on the relationship of loading direction to layer orientation. Especially, in literature have been examined three load cases to printed layer orientation (Wolfs, Bos and Salet, 2019)(Table 1).

Table 1 3D Printed layer orientations in flexural tension, tensile splitting, and compression tests (Wolfs, Bos and Salet,2019)

Orientation	Flexural Tension Test	Tensile Splitting Test	Compressive Test
I			
II			
III			

For the effect of layer orientation on the 3DPC mechanical properties, there is no clear picture due to the small number of works and the different approach of each one of them. In more details, in the report of (Wolfs, Bos and Salet, 2019), there is little effect of the layer orientation on the 3DPC mechanical properties for a specific time gap of 15sec (Figure 33). Also, the compressive strength of the cast sample is higher than the compressive strength of the 3DPC sample and Ref.cast sample (approximately 31%).

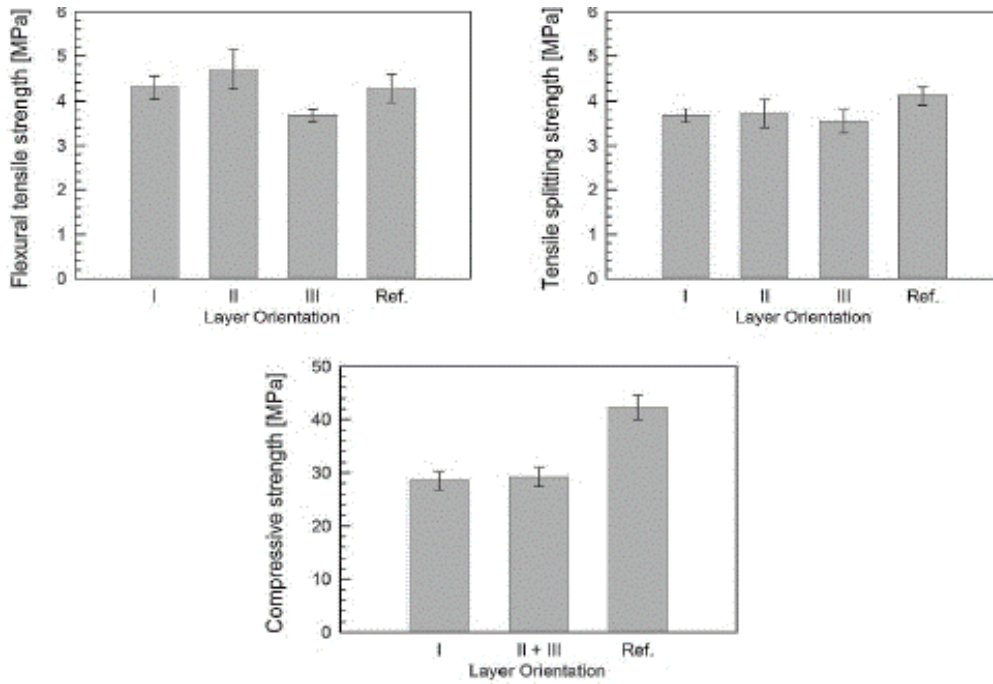


Figure 33 Layer orientation influence on flexural tensile (left) and tensile splitting strength (right) (Wolfs, Bos and Salet, 2019.)

To the contrary, the projects of (Zhang *et al.*, 2019) and (Bong *et al.*, 2019) show a significant influence of layer orientation on the strength properties. (see Figure 34 and 35).

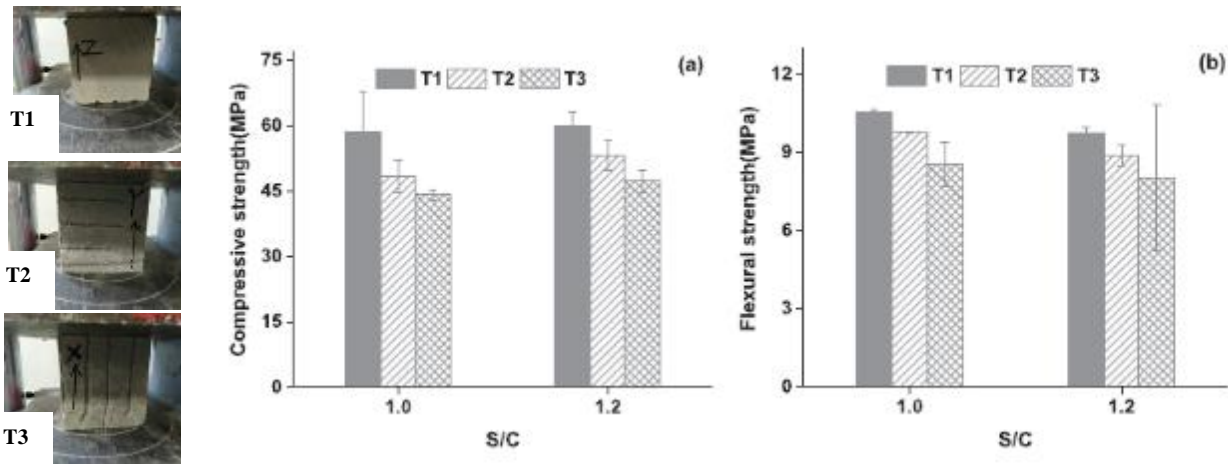


Figure 34 Influence of layer orientation on mechanical properties for different sand/cement ratios (Zhang *et al.*, 2019).

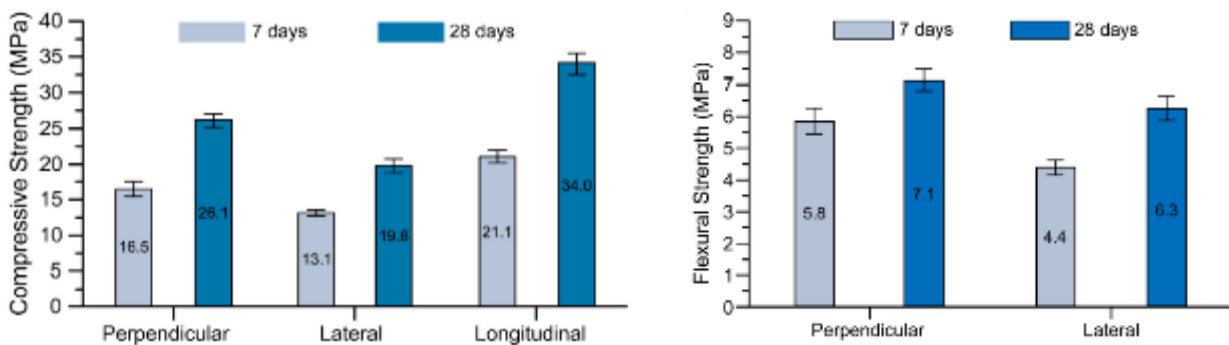


Figure 35 Influence of layer orientation on mechanical properties (Bong *et al.*, 2019)

2.5.2. Interlayer Bonding Strength

One of the governing parameters of the structural integrity of 3DPC is the mechanical interlock between the deposited layers. To measure the interlayer bonding strength of 3DPC, literature reports different testing setups. The most important are:

- Inter-layer bonding strength testing setup uses samples of saw-cut parts of two 3D printed filaments. The samples are stuck with epoxy on the metallic brackets (Figure 36a) and a tensile force is applied (*Putten and Schutter, 2019*).
- Uniaxial tensile strength testing setup, where custom made clamps with two centrally loaded pin connections are used (Figure 36b) (*Marchment, Sanjayan and Xia, 2019*).

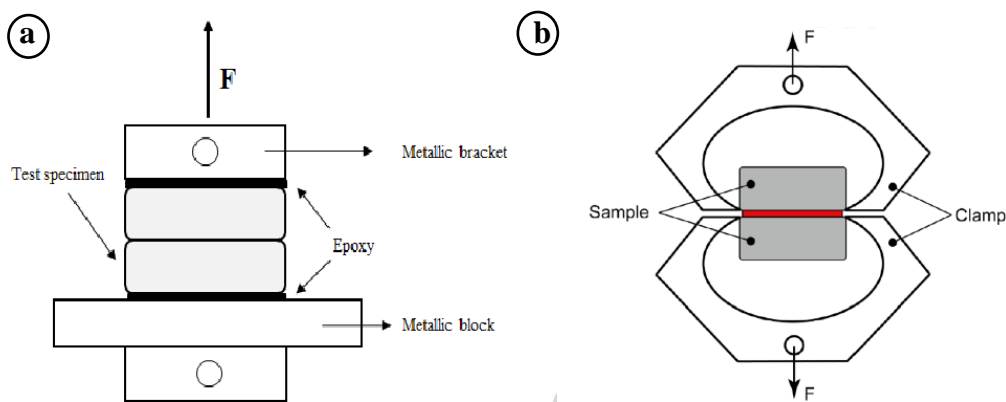


Figure 36 Testing setup for measurement of inter-layer bonding strength

Experimental data suggests that the interlayer bonding strength has a low value and is affected by many parameters mainly from the printing speed. As mentioned previously, the time gap between two filaments which are stacked, is very important and depends on the printing speed. For a long time gap, the bond between the filaments weakens, and a weak interlayer bond appears, which is called cold joint (*Wangler et al., 2016*).

For understanding the interlayer bond, the work of (*Nerella and Mechtcherine, 2017*) uses SEM to investigate the interface between two filaments for different time gaps. The results of this work show that interlayer bonding strength decreases by 9.9% for time gap of 1min, 14.1% for time gap of 10min and 23.1% for time gap of 1 day. This is due to the increase of cold joints along layers and to the presence of cavities due to air entrainment (air voids) (Figures 37 a, b). Also, self-healing phenomena have been observed, in areas where there is no bonding between filaments (silt-like separation), those areas were filled with calcite and / or ettringite over time (Figure 37c).

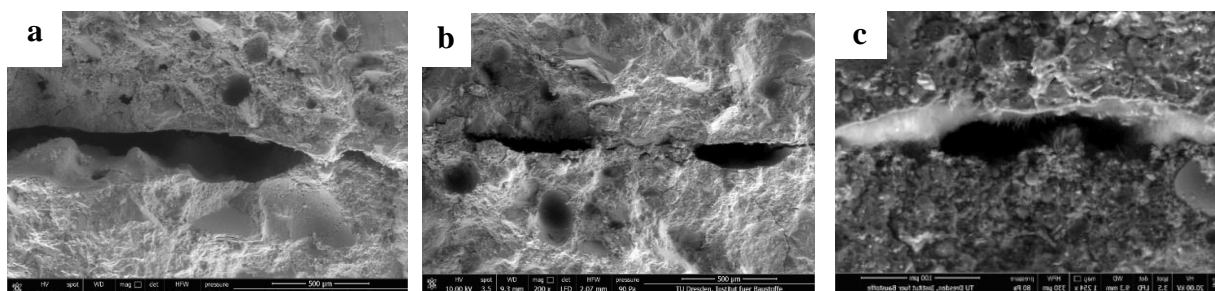


Figure 37 SEM pictures a) cold joints along filaments b) cavities are presented between filaments c) silt-like separation filled by calcite and ettringite -self healing process (Nerella and Mechtcherine, 2017).

To improve the interlayer bonding strength, it has been recorded that either the void number must be reduced or to increase the contact area between the two 3DPC filaments. It has also been found that surface moisture of the layers affects the interlayer bonding strength (Sanjayan *et al.*, 2018). Based on these data, researchers are trying to find ways to improve interlayer bonding strength.

An important approach for improving interlayer bonding strength is the application of adhesive materials between the 3DPC layers. An important example of this application is presented in ‘Method of Enhancing Interlayer Bond Strength in Construction Scale 3D Printing with Mortar by Effective Bond Area Amplification’ (Marchment, Sanjayan and Xia, 2019) where to improve the interlayer bonding strength apply cement paste as an adhesive (Figure 38).

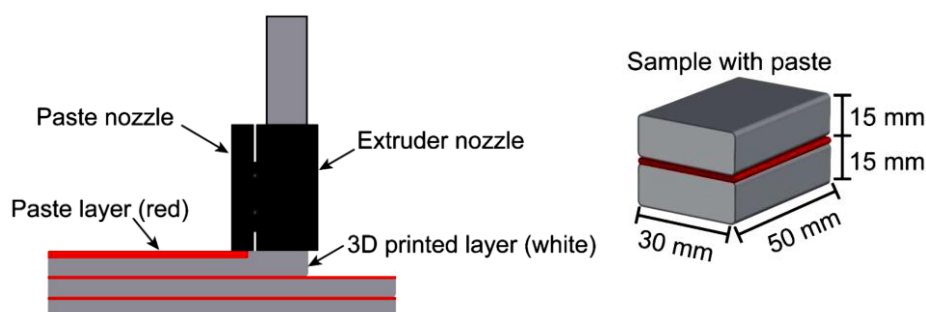


Figure 38 Printing process for 3DPC with cement paste as interlayer bonding strength improvement agent (left) and Sample with paste (right) (Marchment, Sanjayan and Xia, 2019).

The cement paste contains oxides pigments in an amount of 0.25 mass ratio of the OPC weight and is applied in four versions. Paste-Control (contains no admixtures), Paste-Re (contains retarder), Paste-VMA (contains a viscosity-modifying agent) and Paste-SP (contains slump-retention admixture). The samples (Figure 39) were tested in the uniaxial tensile strength testing machine and it was found that the interlayer bonding strength was significantly improved. This is due, to an increase in the effective bond area. The % effective bond area was measured with the help of specialized image analysis and scanning equipment.

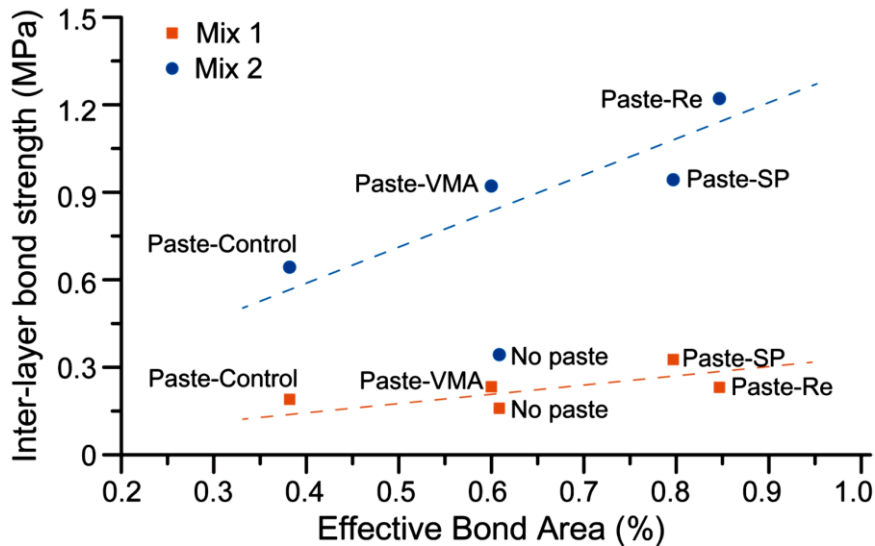


Figure 39 Interlayer bond strength versus effective bond area for Mix1 (cement paste with colour pigments) and Mix2 (cement paste without colour pigments) (Marchment, Sanjayan and Xia, 2019).

A significant improvement is also observed with the use of polymers as adhesive materials. In particular, the work ‘A novel method to enhance the interlayer bonding of 3D printing concrete: An experimental and computational investigation’ (Hosseini et al., 2019) uses Sulfur-Black Carbon polymer (SBC) as an adhesive. In this work, cast samples were created, which in turn, were examined in a three-point bending test, with point load applied on the SBC polymeric interface (Figure 40) similar procedure with that of the tensile splitting test. The results showed a significant increase in interlayer bonding strength (Figure 40).

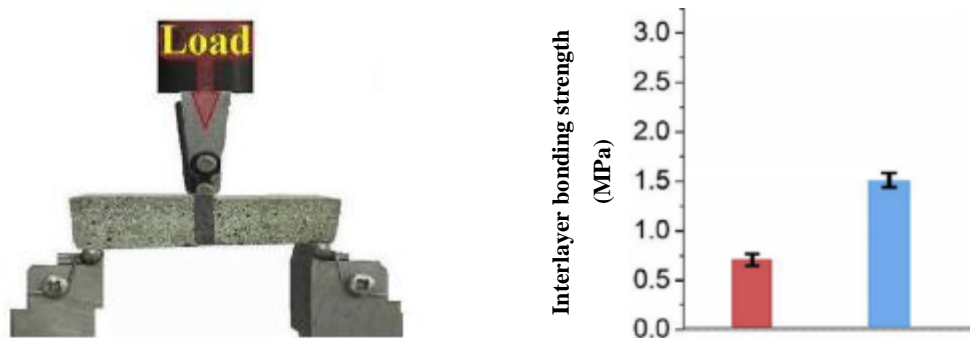


Figure 40 Three point bending test with point load on polymeric interface (left) and interlayer bonding strength outcomes for samples with (blue column) and without SBC (red column) (Hosseini et al., 2019).

The improvement of the interlayer bonding strength is due to the development of a chemical bond, between the polymer and the cementitious material. In more detail, EDS photos (Figure 41) show the development of bonds between calcium, silicon and oxygen (essential compounds of the CSH-hydration product of cement) with sulfur of SBC.

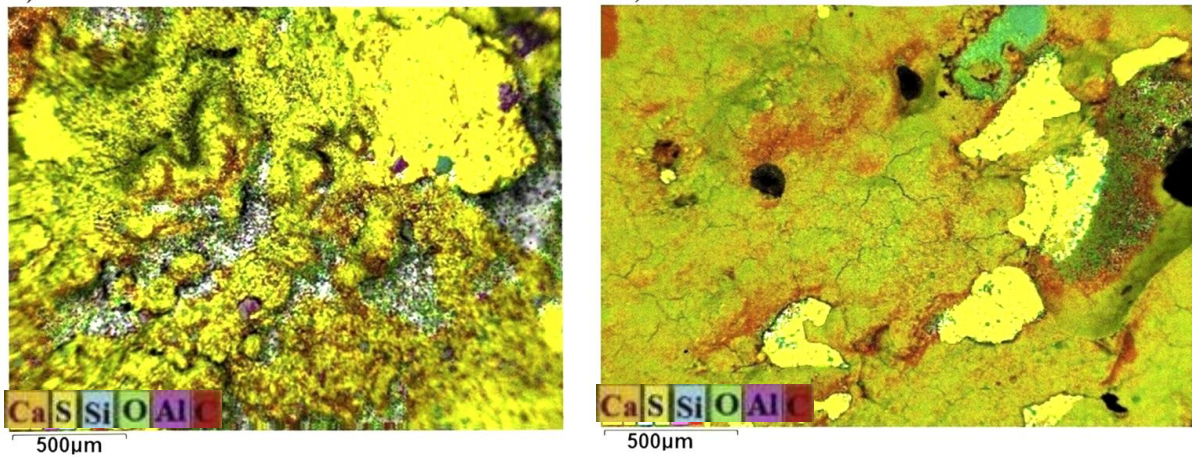


Figure 41 EDS photos of the fracture surfaces of SBC polymer (left) and on cementitious material (right) (Hosseini et al., 2019).

Finally, a significant increase occurs in the interlayer bonding strength with the printing pattern application by which the contact surface of layers is increased. Specifically, in project of (Zareiyani and Khoshnevis, 2017) is to interlocking printing pattern was applied in the interface of the two filaments (Figure 40). The interlocking pattern was applied in four cases, by varying the depth (0, 0.25'', 0.5'' and 0.75'') and holding the width steady. There was a significant increase in the interlayer bonding strength with 0.5'' interlocking depth¹.

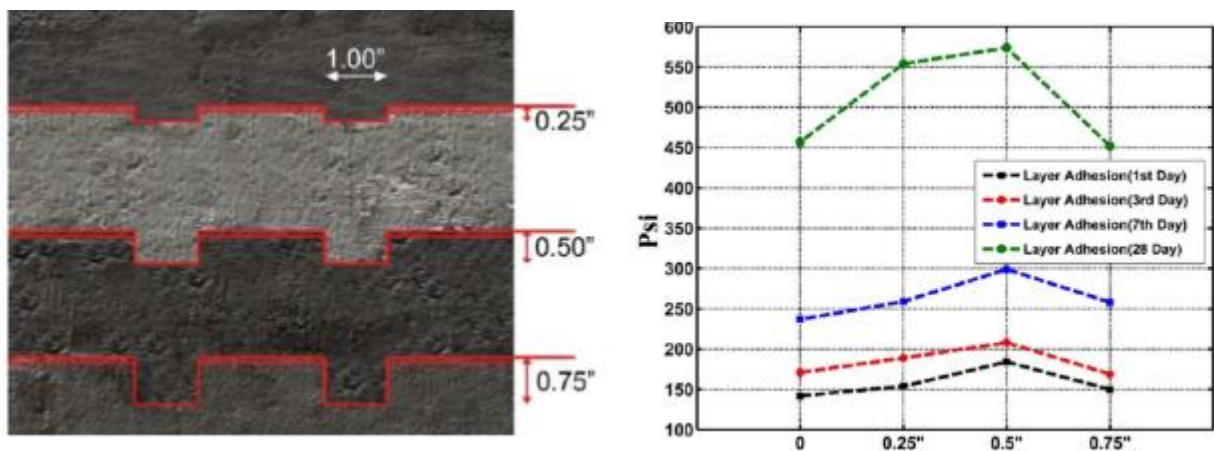


Figure 42 Interlocking patterns (left) and graphs of interlayer bonding strength for different interlocking depths (right) (Zareiyani and Khoshnevis, 2017)

¹ For transformation to SI unites: 1 Psi= 6894.75 Pa and 1''= 0.0254m

- Chapter 3 -

3.1. Introduction

In this work, preliminary experiments were carried out to investigate the behaviour of cementitious materials as printable materials. This chapter describes the materials used, experimental procedures, results related to printable profile of cementitious materials.

3.2. Materials

3.2.1. Raw material

Raw material used in this project:

- Silicate fine aggregates with grain size max. 2mm (Hanson provider).
- Ordinary portland cement, CEM I 52.5N (Hanson provider).
- Fly ash (siliceous pulverized fuel ash) by Drax Power Limited.
- Silica fume used is Elkem Microsilica[®].
- Superplasticizers used are Sika Viscoflow[®] 3000 and Sika Viscoflow[®] 2000.

3.2.2. Samples-Mix Design

In the frame of this work three groups of samples are produced:

- Samples of cement paste (Table 2) are printed in order to get acquainted with the printing device (mortar gun). Also, various parameters (mix design and nozzle cross section) are studied, which affect the printable behaviour (extrudability and buildability) of the samples.

Table 2 Cement paste samples mix design

Samples	water/ binder
CP1	0.30
CP2	0.35

- 3DPC samples enriched with secondary cementitious materials (scm) without admixtures (Table 3). In these samples the influence of the secondary cementitious materials (silica fume and fly ash) on the printable behaviour of 3DPC was studied.

Table 3 Mix design of 3DPC samples with secondary cementitious materials

Samples	Water/ Binder	Sand/ Binder	%OPC	% Silica Fume	%Fly Ash
Control 1	0.20	1.5	100	-	-
Control 2	0.25	1.5	100	-	-
Control 3	0.30	1.5	100	-	-
Control 4	0.35	1.5	100	-	-
SF 5/30	0.30	1.5	95	5	-
SF 5/35	0.35	1.5	95	5	-
SF 10/30	0.30	1.5	90	10	-
SF 10/35	0.35	1.5	90	10	-
SF 15/35	0.35	1.5	85	15	-
SF 20/35	0.35	1.5	80	20	-
FA 5/35	0.35	1.5	95	-	5
FA 10/35	0.35	1.5	90	-	10
FA 15/35	0.35	1.5	85	-	15
FA 20/35	0.35	1.5	80	-	20

- Two groups of 3DPC samples (S2 and S3), where in each group a different kind of superplasticizer was added (S3-Sika Viscoflow 3000[®] or S2-Sika Viscoflow 2000[®]), then the effect of the superplastizers on extrudability, buildability and interlayer bonding strength was investigated. The mix design used for these samples is based on the mix design of the work "Micro- and macroscopic investigations on the interface between layers of 3D-printed cementitious elements" (Nerella and Mechtcherine, 2017) (Table 4).

Table 4 Mix design of 3DPC samples with superplasticizers

3DPC compounds	Content
OPC	55%
Fly Ash	30%
Silica Fume	15%
Superplasticizer	0.75% by weight of binder
Sand/ Binder	0.42
Water/ Binder	1.5

3.3. Experimental Procedure

The experimental procedure applied to the printed samples is divided into two parts the printing process and the experimental methods which were used to examine the printable properties (extrudability and buildability) of the samples. In the case of investigating interlayer bonding strength, cast samples were created simulating the layerwise structure of 3DPC and then the interlayer bonding strength was examined.

3.2.1. Printing Process

A mortar gun (Roughneck® ultimate mortar gun) (Figure 43) was used to print the samples using nozzles with different geometries.



Figure 43 Roughneck® ultimate mortar gun

Initially, a circular cross section nozzle with diameter 3mm was applied for the cement paste samples (CP1 and CP2). It was observed that a not steady flow was produced in the extruded material resulting in an irregular printing pattern (Figure 44). Thus, a circular nozzle with larger, (double diameter 6mm), was used.



Figure 44 Circular cross section nozzle with diameter 3mm (left) and not a irregular printing pattern (right).

The cement paste samples were printed in cubic moulds (50x50x50mm). The purpose of using moulds was the hardened samples to retain the cubic shape without any collapse. The procedure followed is shown in Figure 45. In particular, the first printing process of the first rod started from (0,0,0 mm) to (50,0,0 mm) point with direction of the x-axis. When the first printed rod was completed, the next one was printed in the direction of the y-axis. i.e from (0,6,0 mm) to (50,6,0 mm). When the first layer was printed, the next one was created in the direction of z-axis. i.e. from (0,0,6 mm) to (50,0,6 mm). Finally, the number of rods per layer was 8 and the number of the layers was 8.

The printing parameters printing speed, printing pressure and printing angle were not identified in this stage due to the manual printing process.

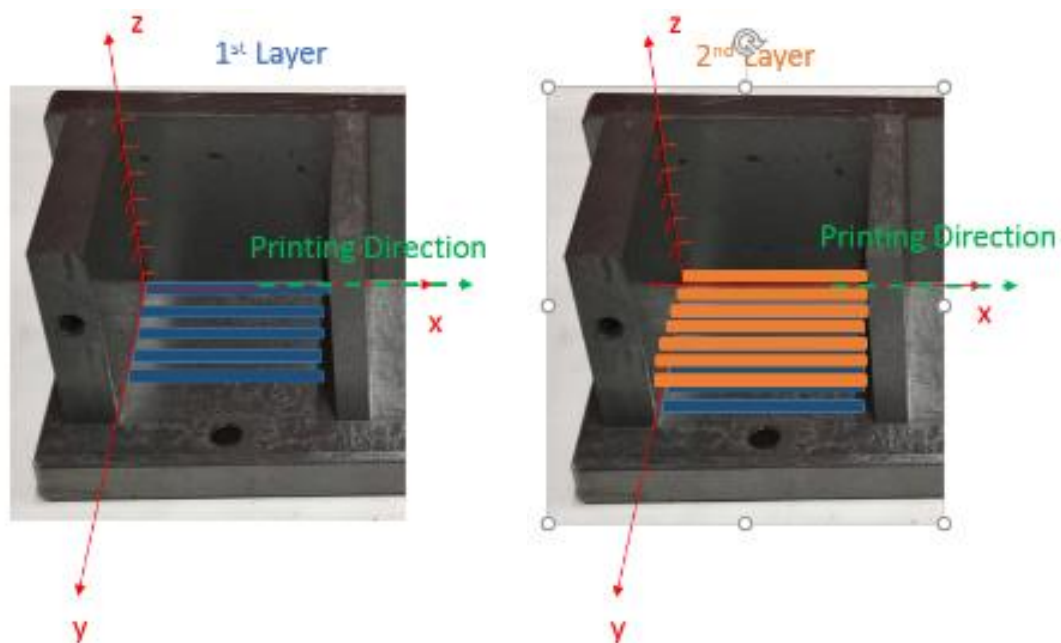


Figure 45 Printing process of cement paste samples.

For the 3DPC samples with scm, a rectangular cross section nozzle was used to print the samples (dimensions of nozzle: width 35mm and height 8mm). The samples were printed in moulds with dimensions (40x40x160mm) - prism shape (Figure 46). The printing process followed had a direction from left to right and from bottom to top (bottom up). The samples were printed in confined and semi-confined conditions. The term semi-confined is the case where one side of the sample has no mould support. Three samples, two confined and one semi-confined, were printed for the SF20 / 35, FA20 / 35 and Control 4 mix design. It is important to note that the printing speed and the printing angle could not be precisely determined due to

the manual process and recurrent fillings of the mortar gun. But, an estimation can be made for printing speed and printing angle 6mm / sec and 45° respectively.

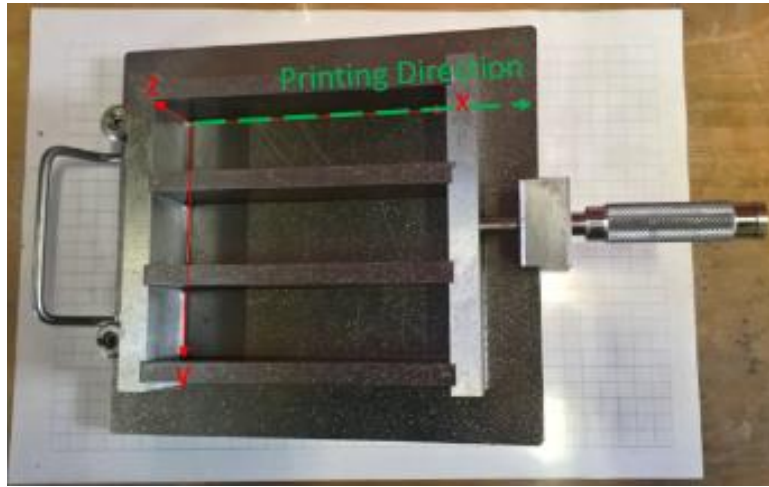


Figure 46 Printing process for 3DPC samples with scm

3.2.2. Experimental Methods

Qualitative and quantitative methods were used to determine extrudability.

Specifically, for printed cement paste samples (CP1 and CP2), the extrudability was qualitatively determined by observing the texture of the samples and recording significant results. With the quantitative approach, the printability limit and consistency of the samples were measured using a Vicamatic 2 by Controls Group (Figure 47).



Figure 47 Vicamatic 2 by Controls Group

For 3DPC samples enriched with scm, extrudability was qualitative determined while in the case of 3DPC samples with superplasticizers, a flow table (Figure 48) was used to compare the effect of different superplasticizers on the workability of the samples.



Figure 48 Flow Table

To determine buildability, a stacking plate apparatus was created by which samples with superplasticizers were examined. This experimental apparatus simulates the weight of the subsequent layers loaded onto the sample using plexiglass containers fitted with sand (Figure 49).



Figure 49 Stacking plate apparatus

Initially, the sample is casted in the mould with dimensions (50x50x50mm) and it is removed after the printability limit (Figure 50a). The 3DPC sample is then weighted and its weight is recorded. (Figure 50b). This weight is the weight of each subsequent simulation layer placed

upon the sample. The weight of each layer is simulated by placing sand in the plunger containers. The container-plunger is then placed upon the sample. The plunger presses the sample and the height reduction is measured by using a ruler. (Figure 50c). Subsequent simulation layers are added at fixed time intervals (time gap-printing speed) and the height reduction is measured again. If the height reduction value exceeds 10% of the original height, then the specimen is assumed to have collapsed for a specified number of layers. This method determines the stability regarding the time gap in terms of height reduction for a specific time gap.

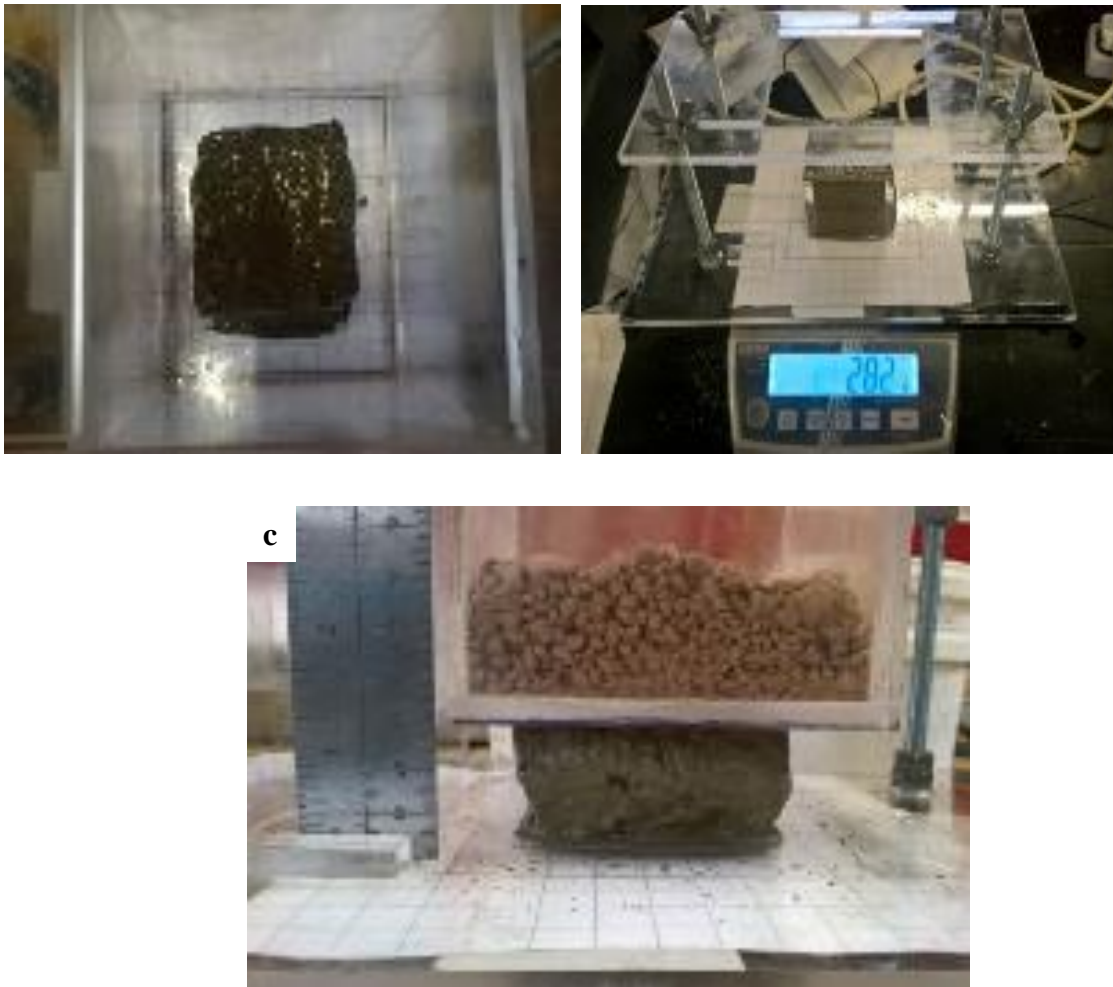


Figure 50 a) sample in stacking plate apparatus b) measurement of sample weight c) collapse of the sample when the plunger (one simulation layer) was placed.

3.2.3. Interlayer Bonding Strength – Cast Samples

For the samples with superplasticizers, the mortar gun was not used. The samples were casted in modified moulds with separators, simulating the layerwise structure of 3DPC. Separators were placed in order to create interface in the mortar samples. Specifically, the samples were casted into moulds (40x40x160mm) and separators were placed in the half-length of the moulds (40x40x80mm). The purpose of this process is the investigation of the influence of different parameters (mix design, time gap and the weight of the layers) on interlayer bonding strength. The experimental process that took place follows the stages:

1. Initially, the mortar samples were casted with the separators (Figure 51). The separators were then removed at intervals of 5 min, 10 min and 20 min from the time they were casted. The purpose of this first step is to simulate the time gap between the layers and research its influence on the interlayer bonding strength.



Figure 51 Prism moulds with separators.

2. The samples were then placed, inclined with inclination angles of 30° (Figure 52) and 90°. The purpose of this procedure was to study the influence of the weight of the subsequent filament (half part of the sample) on the interlayer bonding strength.



Figure 52 Mould with sample with 30° inclination.

3. Finally, after 1 day of setting the samples unmoould.

3.4. Results

3.4.1. Fresh Properties

The extrudability of the CP1 and CP2 samples was examined. Good extrudability was recorded for both samples. It is important to note that there is a small spread of the rods when the cement paste is extruded directly after the end of the mixing procedure. The texture of the material resembles a bleeding scenario (Figure 53a). If the paste is left to rest for 15min (Figure 53b) the consistency of the extruded material is noticeably better. The rods retain their extruded shape and the bleeding resemblance does not exist. Probably, the last observation happens as a result of good saturation of the cement particles and the initiation of the hydration process.

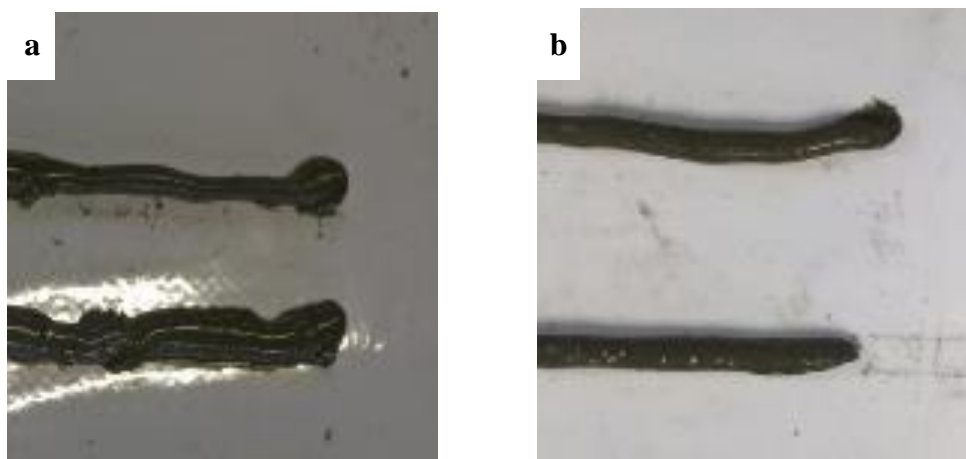


Figure 53 CP2 sample texture a) immediately after mixing procedure and b) 15 min after mixing procedure.

Also, to determine printability limit (printable window), the Vicant (Vicamatic2-Controls Group) device was used to determine the initial setting time. For this purpose, the Vicant device was programmed with parameters different from those defined by the EN196-3 standard. The results of these measurements are shown in (Figure 54). There are some differences in the values between the measurements of the modified process applied (CP1 and CP2) and measurement according to protocol EN 196-3 (CP1 EN 196-3). Probably, these differences are the result of varying laboratory conditions (Table 6).

Table 6 Initial setting time results

Samples	Initial setting time
CP1	01:17:30
CP2	02:27:30
CP1 EN 196-3	02:30:00

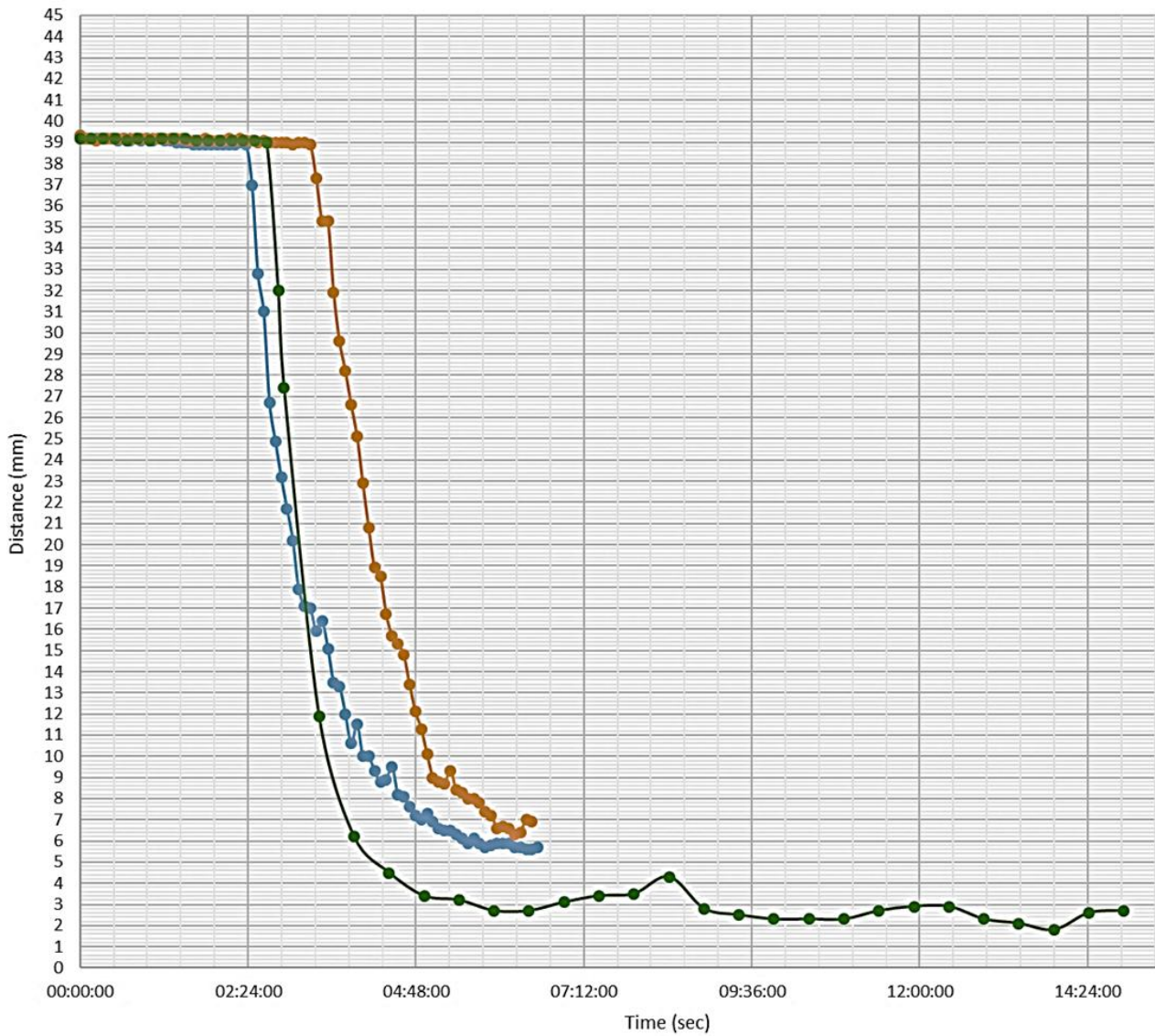


Figure 54 Setting time plot for CP1, CP2 and CP1 EN 196-3.

Also, the consistency of cement paste samples was examined. For measurement of the consistency, the Vicamatic2 device was used with a special probe to measure consistency (Figure 55). The consistency of the CP1 and CP2 samples was measured and found to be the same (Table 7).

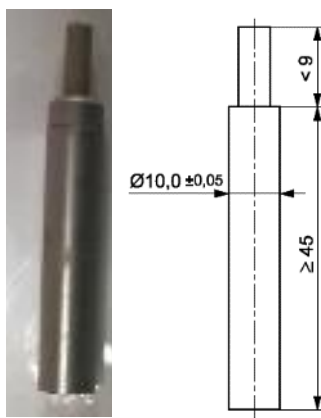


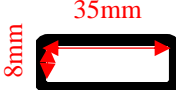


Figure 55 Consistency Probe

Table 7 Consistency results for CP1 and CP2

Samples	Consistency
CP1	39.2mm
CP2	39.2mm

The 3DPC samples with scm were printed with different types of nozzles (Figure 56), and by the observation process they were separated as extrudable (✓) and non extrudable (x). This separation was based on the retention of the shape (red guidelines on photos, (Figure 56) in addition with the absence of imperfections and discontinuities (Figure 56). According to these criteria, samples containing secondary cementitious materials were extrudable (Table 8). These results show that an important parameter is the water / binder. Specifically, the samples Control 1 and Control 2 did not become extrudable, because the samples had water/binder 0.20 and 0.25 respectively.

Table 8 Extrudability results for different nozzle geometries

Samples	 35mm 8mm	 diam. 10mm	 diam. 6mm
Control 1	x	x	x
Control 2	x	x	x
Control 3	✓	✓	✓
Control 4	✓	✓	✓
SF 5/30	✓	✓	✓
SF 5/35	✓	✓	✓
SF 10/30	✓	✓	✓
SF 10/35	✓	✓	✓
SF 15/35	✓	✓	✓
SF 20/35	✓	✓	✓
FA 5/35	✓	✓	✓
FA 10/35	✓	✓	✓
FA 15/35	✓	✓	✓
FA 20/35	✓	✓	✓

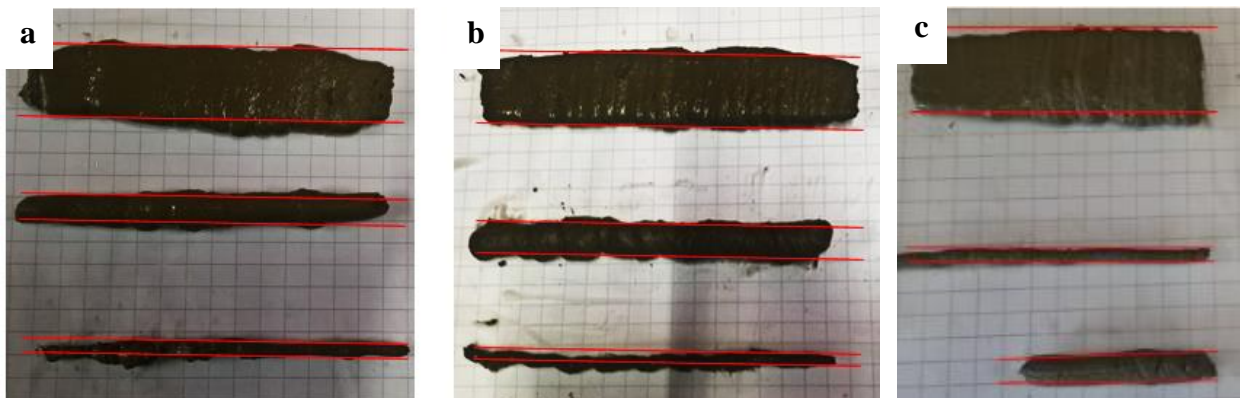


Figure 56 Extrudability of a) Control3 sample b) SF10/35 sample and c) FA20/35 sample.

The workability (as a extrudability parameter) of the samples with superplasticizers (S2 and S3) was measured using a flow table (Figure 57). It was found that the sample S3 has a higher flowability rate compared to the S2 sample by 4.6%.

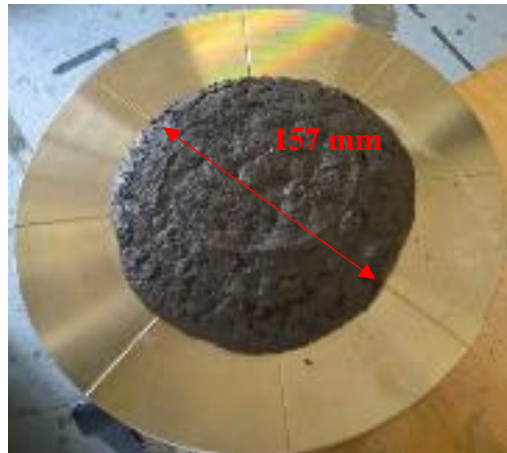


Figure 57 Workability of S3

The extrudability of the samples was then examined qualitatively. Using the mortar gun and nozzles with cross section rectangular (dimensions of nozzle: width 35mm, height 8mm and circular diameter of 6mm) S2 and S3 samples were printed. These samples were then tested, and it was found to have a good shape stability after extrusion, not discontinuities, but a few defects were observed on the surface of the samples (highlighted by red circles on photos, Figure 58).



Figure 58 Extrudability texture of S3 (left) and S2 (right).

Finally, the buildability of the S2 and S3 samples was tested using the stacking plate apparatus. As mentioned in a previous paragraph, this setup mimics the multilayer concept of 3DPC and measures the buildability in terms of height reduction (stability) regarding to time gap. Samples S2 and S3 were tested and exhibited similar buildability. That is, for a time gap of 10min, they can withstand 10 simulation layers and the height reduction is less than 10%.

3.4.2. Hardened Properties

The cement paste samples were cured for 7 days in water and then were examined. Initially, CP1 and CP2 samples and the filaments-rods held the internal parallel pattern (orange lines on pictures, Figure 59 a,c,d,f). On the other hand, they showed a lot of imperfections. In particular, there are dimensional changes on rod shapes due to the weight of the subsequent layers (yellow circles on pictures, Figure 59 b,e). Also, surface defects were observed in both of the samples in the form of air pockets trapped between the boundaries of each layer (red circles on pictures, Figure 59 c,e,f). The size of the defects increases from CP1 to CP2, as it was expected. The size of these defects ranges from 2mm to 3mm for CP1 and 3mm to 5mm for CP2.

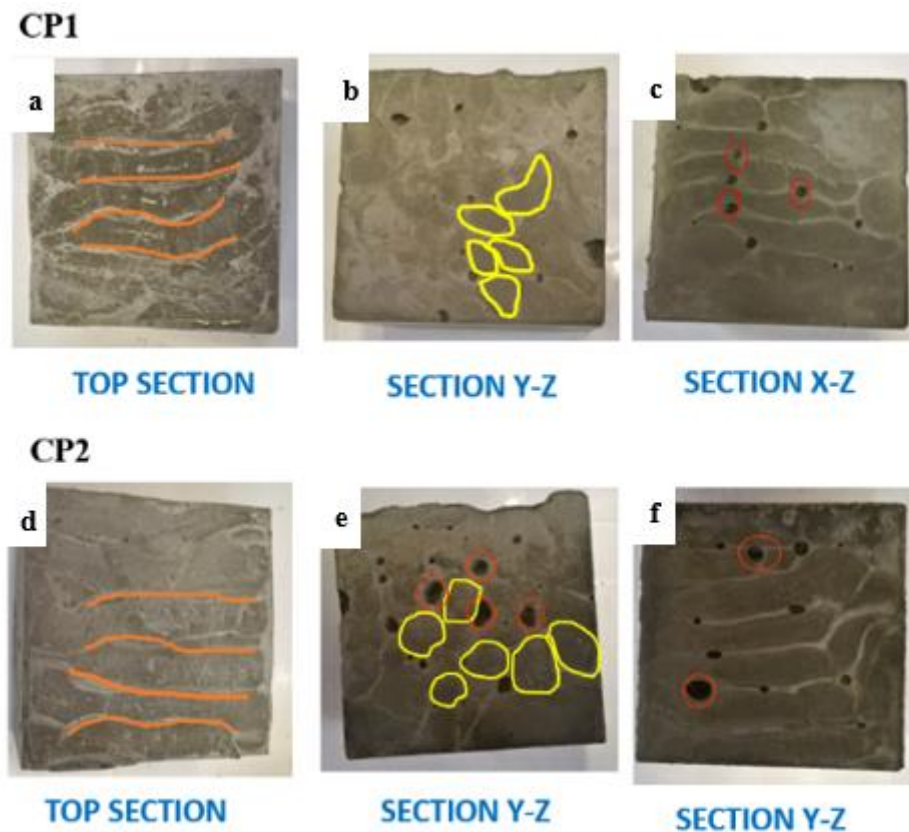


Figure 59 Hardened cement paste samples

The samples with scm were cured for 7 days and tested as hardened 3DPC samples. The observations on the samples are as follows:

- The number of layers differs between the samples. Specifically, it is noticed that the layers of the sample with fly ash are more (6 layers) (Figure 60a) than the samples with silica fume (4 layers) (Figure 60b). This is due, to the fact that samples with silica fume have better buildability than the samples with fly ash. As a result, fly-ash filaments do not withstand the

weight of the subsequent layers and become compressed. The samples with silica fume have better buildability because they have a better pozzolanic behaviour. Thus, the samples with silica fume gain faster strength through the hydration process (due to the value of hydration products) compared to the samples with fly ash. Also, important factors for the variation of the number of layers, can be considered to be the non-constant printing angle, printing speed and printing pressure.

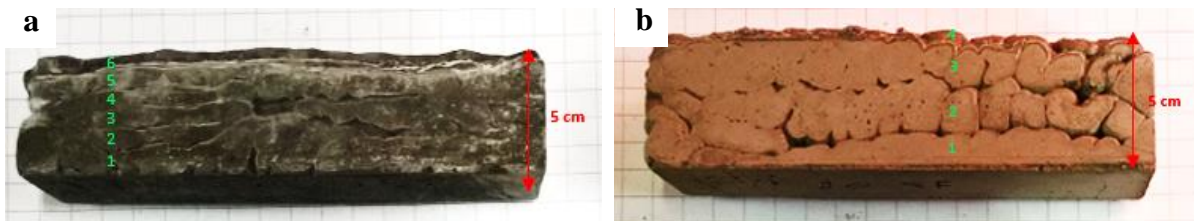


Figure 60 a) Different number of layers between FA 20/35 b) and SF 20/35.

- In addition, the parameters of non-constant printing angle, non-steady printing speed, and non-steady printing pressure contribute to an unsteady formation of the layers. Therefore, waves and irregularities on the shape of the layers were observed on the surface of the samples as a result of these parameters (Figure 61).

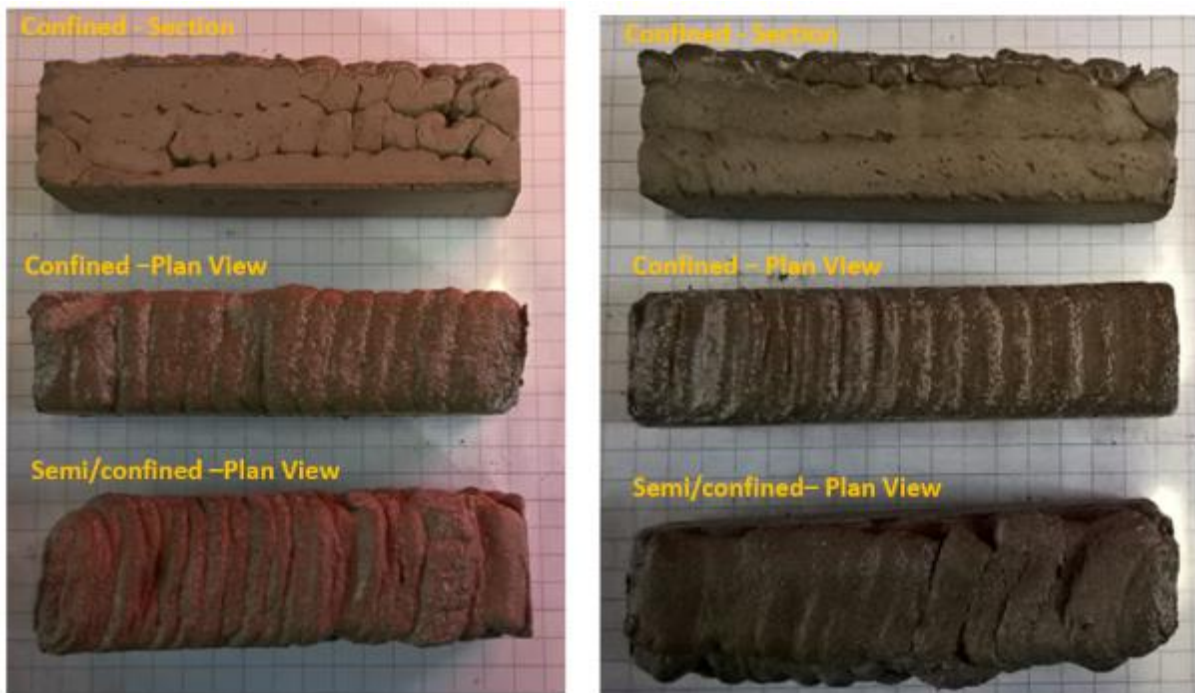


Figure 61 Influence of unsteady printing speed, printing angle and printing pressure on SF20/35 (left) and Control4 (right).

- For semi-confined samples, better stability was observed in the case of the samples with silica fume and without scm as compared to samples containing fly ash (Figure 62). It would be expected that fly ash would increase flow and reduce the buildability. On the other hand, silica fume reduces flow but increases buildability.



Figure 62 Stability of FA20/35 (left) and SF20/35 (right).

- In most of the samples, there are visible defects such as voids on the layers, gaps between layers, and mixing of the layers.

The samples with superplasticizers were separated at the interface area during the unmoulding process, which area had been created using separators (Figure 63). This area was separated without making any kind of bond, which would result in the presence of cold joints. An optimization of the experimental process is required.

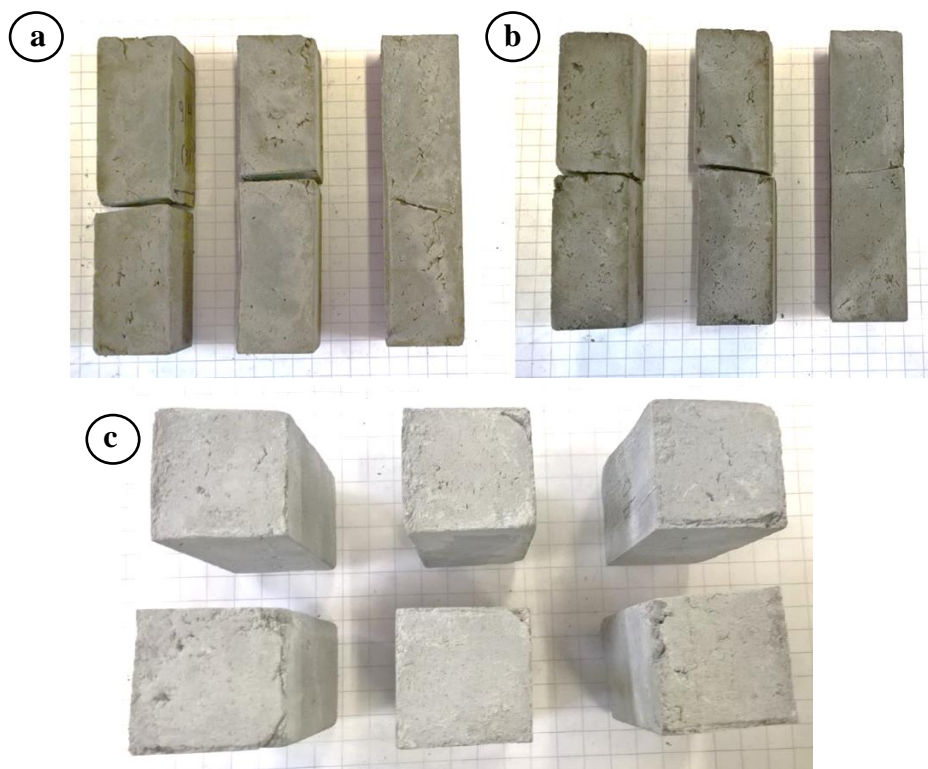


Figure 63 a) Samples cured in 90° b) Samples cured in 30° and c) Observation of cold joints in interface area of samples.

Discussion

The construction sector is the industry with limited adopted technological innovations. In recent years, efforts have been made to change the construction industry to be more sustainable through the use of digital technologies. One of the most important technology which promises to transform the construction industry is the additive manufacturing method. This new method attracts the interest of researchers and stakeholders in construction. A rise in the number of research works and applications has been noted in recent years.

Additive manufacturing methods in construction use a variety of materials, as a main the 3D Printing Concrete 3DPC. This material is used by extrusion additive manufacturing techniques and has significant differences from the existing monolithic types of concrete. In particular, it is an anisotropic material with a layer-wise structure, whose properties are associated with features of the printing process and the printing system. Research on this new type of concrete is constantly developing. Researchers are trying to understand the printable properties of this new type of concrete and develop methods to improve these properties.

In the literature there are several research approaches on 3DPC. This new uncharted research field presents numerous mix designs of 3DPC, different printing processes and printing parameters, non-standard terminology of the 3DPC properties and several experimental approaches for the identification of 3DPC properties.

In this study, literature review and preliminary experiments were carried out with the purpose to explore the feasibility of experimental procedures in order to determine some important 3DPC properties (extrudability, buildability and interlayer bonding strength). As part of this work, three groups of cementitious material samples were created (cement paste samples, 3DPC samples with secondary cementitious materials and 3DPC samples enriched by different type of superplasticizers) by using as a printing device, a mortar gun.

The bibliography does not mention a standard terminology and experimental procedure for the investigation of the printable properties of 3DPC. In the present study, the terms extrudability and buildability were adopted for printable properties. Several qualitative and quantitative experimental approaches have been investigated in order to determine these properties. Specifically,

- The qualitative approach and quantitative approach were applied to determine extrudability.

In the qualitative approach, a filament of every mix design was extruded by a mortar gun. Filaments were considered as extrudable when they keep their shape, they have not discontinuities and imperfections on their surfaces. Based on these criteria, it was found that the shape and size of the nozzles have minor impact on the extrudability of filaments. In addition, it observed that w / c ratio has an influence on extrudability. In particular, filaments with low w / c with values of 0.20 and 0.25, do not make it possible to print them. Also, cement paste filaments, which were printed directly after the mixing process, showed acceptable extrudability. Although, the texture of the filament resembles a bleeding scenario due to pure mix saturation. Also, It was noticed that the bleeding scenario disappeared when the paste rested for about 15min (Figure 53).

In addition, a flow table and a Vicant setting time experimental device (Figure 47) were used to determine workability and consistency respectively, as quantitative testing methods of extrudability. The results showed that 3DPC samples with different superplasticizer additive are extrudable when the workability values have variation up to 4.6% between them (Figure 57 and 58). Also cement paste samples with different w / c ratio (0.30 and 0.35) had the same consistency and were extrudable (Table 7).

- Qualitative and quantitative experimental methods were applied to determine buildability.

In the qualitative approach, a number of samples were printed with different printing procedures. Specifically, samples of cement paste were created consisting of rod-shaped filaments which were printed in cubic shape moulds - confined conditions. In the initial stage of the experimental process, familiarity was gained with the printing device (printing angle, printing speed, printing pressure etc.) and elementary observations on buildability were recorded. In more detail, it was observed that the dimensional changes on the printed filaments are due to the weight of the subsequent filaments, the parallel printing pattern was maintained as it was and interlayer defects occurred due to the printing process (Figure 59).

In continuation several 3DPC prism shape (40x40x160mm) samples with secondary cementitious materials were printed in confined and semi-confined conditions (Figure 62). The buildability qualitative experimental approach was selected for these group of samples. The object of this experiment was to determine the influence of secondary cementitious materials on buildability. As a result, it was noticed that the samples with silica fume showed better buildability compared to others (Figure 60). This is because silica fume has a better pozzolanic behaviour compared to fly ash. It is important to note that imperfections appeared in the samples like voids on the layers, gaps between layers, and mixing of the layers (Figure 61). These defects were related to the non-steady printing parameters process with a clear effect on the buildability of the samples.

For the investigation of the effectiveness of buildability quantitative experimental approach, a stacking plate experimental apparatus was designed and built (Figure 50). The buildability of 3DPC samples with superplasticizers was examined by using the stacking plate. The outcomes of this experimental process showed that the samples with superplasticizers were buildable. Specifically samples S2 and S3 for a time gap of 10min, they can withstand 10 simulation layers and the height reduction was less than 10%. The advantages of this method, compared to the qualitative approach, are the absence of imperfections on the samples as occurred in the qualitative approach and the significant reduction in the amount of 3DPC waste.

Another group of properties, under investigation, is the mechanical properties of 3DPC. The research interest focuses on the influence of the layer-wise structure of 3DPC on these properties. Current research notices that the interlayer bonding strength is the weakest property of 3DPC and it is influenced by a number of parameters of the printing process. Based on that, several adhesive materials (polymers and cementitious materials) have been used as improvements of the interlayer bond and they have showed satisfactory initial results (Figure 39 and Figure 40). Nevertheless, the research on this field is still on an elementary stage.

In the context of this study, the influence of time gap between layers in interlayer bonding strength it was investigated. For this purpose, an experimental setup by separators and prism moulds was created (Figure 51). Then it was observed that the layers of the samples were separated without to develop any bond for printing speed - time gap 5min, 10min and 20min (Figure 63) . Therefore, a further optimisation of that process is required.

In literature, references are made about the sustainability behaviour of additive manufacturing method in construction, concentrated mainly at the level of applications. In more details, for the construction of complex structural elements by using additive manufacturing method is achieved with less amount of materials, a lower construction cost (Figure 23), and better environmental impact (Figure 4), compared to the classic construction methods. This part of the research is limited and a further systematic investigation is required.

An important part of this research project is the study of sustainable behaviour of these new printable cementitious materials. The literature mainly mentions that these new materials are more sustainable than traditional construction materials due to the application of digital fabrication methods in construction, by achieving a reduction in the amount of used materials, and the reduction of the quantity of waste materials, consequently the overall sustainability index of these materials is improving through LCA. Unfortunately, no report has been found investigating the sustainability behaviour of printable construction materials in terms of mix design, printable, mechanical and durability properties and comparing it to the traditional monolithic cementitious materials.

After the completion of this preliminary stage of the research program, the following key remarks have derived:

1. There is a wide variety of research approaches in the literature on the investigation of this new material. To date, no standardized experimental procedures for investigating the properties of 3DPC have been adopted, as there are for the monolithic-traditional types of concrete. Therefore, there is a difficulty in collecting data, analyzing it and drawing conclusions. An important example of this non-systematic experimental process is that in most studies the reference to the printing parameters is omitted. As a result, the influence of the printing parameters on the material properties were not considered at the level of the analysis of the results.
2. It is also important to mention that in the literature there are examples of the application of 3DPC in the construction of structural elements or entire construction systems without specifying the fundamental properties of the material, such as compressive strength, flexural strength etc. Therefore, there is a gap in the research and development of 3DPC and questions arise about the effectiveness and advantages of these applications compared to traditional methods.

3. The printable properties of 3DPC take a significant part of the current research. As mentioned, for the investigation of these properties, qualitative and quantitative experimental approaches have been used. In this work, it was found that the qualitative approach for the extrudability yielded satisfactory results. Also, the quantitative approach for buildability, by using a stacking plate apparatus, yielded satisfactory results as well. Based on these results, an effective experimental process of determining extrudability and buildability for a defined open time can be adopted for further development of this research project.
4. The 3DPC, is a layer-wise cementitious material whose properties are affected by the capabilities of the printing system and the printing parameters. In this study, cementitious materials were printed by using a mortar gun, as a printing device. The usage of the mortar gun did not provide the ability to accurately determine printing parameters such as printing speed, printing angle, printing pressure etc., consequently the samples showed a number of defects that affected their properties. On the other point of view, through this process, remarks were noted related to the degree of easiness of each sample (mix design) that can be extruded through a mortar gun - pumpability. Specifically, for the samples with silica fume less printing pressure was applied on the mortar gun trigger compared to samples with fly ash. This observation showed the interaction between mix and interior walls of the mortar gun. This is an important parameter for determining printing pressure, the type of material that the printing system has made and the mix design of the 3DPC.
5. Interlayer bonding strength is a weak property of 3DPC, research has shown that it is influenced by many parameters of the printing process, and it is a source of creation of several interlayer defects. These imperfections degrade the mechanical behaviour of the samples and might be the cause for degrading factors of durability properties such as permeability, sorptivity etc. As mentioned in the literature, there is a gap in the investigation of the durability properties of 3DPC and in the sustainable behaviour of 3DPC (key objective of this research project) subsequently. Based on that, in this study, the interlayer bonding strength in relation to printing speed was investigated. For this purpose, an experimental setup with separators was designed to avoid these imperfections made by the printing parameters. The results showed that further optimisation of this experimental process is required.

Based on the outcomes of this initial stage of the research program is proposed the future work to be divided into two phases:

Phase 1 - *Investigation of the optimal mix design in terms of sustainability and printability.*

More extrudability and buildability experiments should take place in order to investigate the impact of more sustainable additives (i.e. recycled silicates) on the printable profile of 3DPC. The optimised mixed design should be selected in terms of embodied CO₂ emissions and material cost (sustainability criteria). In more details, for the samples that they have acceptable printability, an optimisation index should be created. This index should be a function of the embodied CO₂ emissions and material cost. This mix design must have the lowest optimisation index and it will be considered as the *optimal sustainable design mix*.

Phase 2 - *Investigation on the hardened properties of 3DPC.*

In this phase, it is proposed the further investigation of hardened properties of 3DPC samples with optimised mix design. These prism shape samples (40x40x160mm) will be printed by using a printing device with calibration capability of the printing parameters. The samples will be different in terms of printing speed and layer thickness. The investigation of these samples will take place in two experimental parts:

1. *Mechanical properties.* Identification of compressive strength, flexural strength and interlayer bonding strength of 3DPC and monolithic samples (with the same mix design) will be performed.
2. *Durability properties.* Sorptivity test (capillary porosity) and permeability test will be performed for 3DPC samples and the values will be compared with the cast monolithic samples with the same mix design.

Afterwards, a combination with microstructural analysis techniques such as, Scanning Electron Microscopy (SEM), the influence of printing speed, layer thickness and layer orientation on the hardened properties of 3DPC will be examined. Based on the data results, optimisations of the processes will be suggested and conclusions about 1) the sustainable behaviour of 3DPC in terms of mix design, printable properties, mechanical and durability properties, 2) the advantages and limitations of 3DPC by comparing with traditional monolithic concrete types will be drawn.

Conclusions and Recommendations

The interest in the applications of the digital fabrication methods in several industry sectors is continuously growing. The number of applications of additive manufacturing methods in construction is rising in the last years. This interest creates the need to determine the effectiveness of these applications in terms of structural capability and sustainability. These applications are based on the type of printing device, the printing technique and the printing construction materials used. The research on printable construction materials is a research field in the elementary stage. This report presents the preliminary research work done on the properties of new printable cementitious material, the 3DPC. The main conclusions which have been drawn are the following:

1. The research on the application of additive manufacturing method in construction is at an early stage. There is a small number of research references and a standard methodology for the investigation of essential structural and sustainability behaviour of applications has not been determined. Thus, there are difficulties at this stage for conclusions to be drawn about the effectiveness of these new construction methods compared to the traditional construction methods.
2. The 3DPC is a new cementitious material whose properties depend to a large extent on the the printing device and its printing parameters capability. The influence of printing parameters on 3DPC samples was found in the literature and it was verified in this stage of the research project. In order to develop this research program effectively, the optimisation of the printing process is needed.
3. The extrudability and buildability of cementitious materials is mainly affected by the printing parameters, mix design and printable window. The most effective methods which have been observed, at this stage of research, are the qualitative experimental approach for determining the extrudability and the quantitative approach by using the stacking plate apparatus for the buildability.
4. There is a number of references in the literature for the mechanical properties of 3DPC. The research interest is mainly focused on the interlayer bonding strength and the parameters which have affected this weak mechanical property. Efforts are made to improve this property by using adhesive materials between the printed layers of 3DPC. In general, more research needs to be done on the mechanical properties especially on the investigation of the influence of the orientation of layers on the mechanical

properties, the influence of printing parameters on these properties and their long term behaviour.

5. In the context of this work, no study in the literature has been identified that examines the durability properties of 3DPC. Thus, this research task should explore the influence of the layer-wise structure of 3DPC on durability properties and the type of materials to be used as an adhesive to improve interlayer bonding strength. For instance, the choice of adhesive materials should be based on their long term behaviour in the atmosphere (i.e. CO₂ emissions) and water environment.
6. In the literature there is a limited number of research works related to the sustainability of additive manufacturing method in construction, especially for the sustainability behaviour of 3DPC. More research is required in this area.

No.	Paper	Water/ Binder	Sand/ Binder	Sand	Clay ²	Cement ³	Silica Fume ⁴	Fly Ash	Admixtures ⁵	Fibers
1	(Le et al., 2012)	0.37	1.5	2mm	-	CEM I 52.5	not specified	not specified	1%SP, 0.5%R	Polypropylene
2	(Wei et al., 2016)	0.39	1.7-4.2	not specified	-	not specified	vary	vary	1.9%SP	Polypropylene
3	(Zareiyani and Khoshnevis, 2017)	0.50	vary	natural sand 4.75mm	-	CSA + OPC type I	-	-	-	-
4	(Nerella and Mechtcherine, 2017)	0.42	1.5	not specified	-	not specified	15%SF	30% FA	0.75%SP	-
5	(Kazemian et al., 2017)	0.43	vary (mainly 2.30)	max size 2.36mm	ANC 1.75µm	ASTM C150 type II	densified SF	-	VMA,HRWRA	Polypropylene
6	(Rushing et al., 2017)	vary	vary	vary	Bentonite	Type I/II	vary	vary	SP	Metal
7	(Soltan and Li, 2018)	vary	vary	vary	ANC	CA + Type I	GS + MS	Class F	HRWRA, HPMC	PVA (12 mm)
8	(Putten and Schutter, 2019)	0.5	2	max.size 2mm	-	CEM 52.5 N	-	-	SP	-
9	(Liu et al., 2019)	vary	vary	vary	vary	ASTM I, Grade 42.5	undensified SF - Grade 940	Class F	SP	-
10	(Demyanenko et al., 2018)	vary	0.5	not specified	-	CEM I 42.5H	-	-	-	-
11	(Panda and Tan, 2019)	0.45	1.35	not specified	-	CEM I + ASTM C 618	vary	vary	SP	-
12	(Rahul et al., 2019)	vary	vary	quartz sand	Nanoclay 1.5-2 µm	IS 12269	SF	Class F	SP,VMA	Polypropylene
13	(Zhang et al., 2019)	0.35	vary	max.size 1mm	2% Nanoclay	not specified	2% SF	-	HRWR	-
14	(Marchment, Sanjayan and Xia, 2019)	0.36	0.5	fine aggregate	-	AS 3972, type GP	SF	-	SP,R,VMA,SRA	-
15	(Tay et al., 2019)	0.46	1.2	not specified	-	ASTM I, Grade 42.5	undensified SF	Class F	-	-
16	(Panda, Lim and Tan, 2019)	0.30	0.83	not specified	0.5%ANC	OPC	Microsilica	Class F	-	-
17	(Wolfs, Bos and Salet, 2019)	0.5 *water to fines	not specified	siliceous aggregate with max.size 1mm	-	CEM I 52.5R + Limestone filler	-	-	Rheology modifiers	Polypropylene

² ANC-Attapulgit Nanoclay

³ CA-Calcium Aluminate Cement,

⁴ GS-Ground silica, MS-Microsilica

⁵ SP-Superplasticizer, R-Retarder, VMA-Viscosity Modifying Admixture, HRWRA-High Range Water Reducing Admixture, HPMC- Hydroxypropylmethylcellulose, SRA-Slump Retention Agent

Master of Science by Research –Appendix / Mix Design of 3DPC

No.	Paper	Water/ Binder	Sand/ Binder	Sand	Clay ⁶	Cement ⁷	Silica Fume ⁸	Fly Ash	Admixtures ⁹	Fibers
18	(Chaves et al., 2019)	vary	vary	vary	-	CEM I 42.5 + Limestone powder	-	vary	SP, VA	PVA
19	(Tay, Li and Tan, 2019)	0.5	1.7	max.size 2.36mm	-	ASTM Type I, Grade 42.5	undensified SF	Class F	-	
20	(Ma and Kawashima, 2019)	0.34	vary	max.size 2.36mm	Palygorskite nanoclay	Class H oil well cement	-	-	SP	-
21	(Khalil et al., 2019)	vary	vary	0.2mm	-	CSA + CEM I 52.5	-	-	SP	-
22	(Kazemian et al., 2019)	0.45	vary	max.size 2.36mm	ANC	ASTM C150 Type II	vary	-	VMA, HRWRA	Polypropylene
23	(Bao et al., 2019)	vary	vary	not specifiend	ANC	CA + CEM I	vary	vary	VMA, HRWRA	PVA
24	(Wangler and Flatt, 2018)	0.42	vary	max.size 2 mm	-	CEM I 52.5 R	0.50wt% suspension	Class F	SP,HRWRA	-
25	(Putten and Schutter, 2019)	0.37	2	max.size 2 mm	-	CEM I 52.5N	-	-	SP	-
26	(Yu and Leung, 2019)	0.30	0.20	not specified	-	CEM I 52.5N	Silica sand	FA	SP, VMA	PVA
27	(Tay et al., 2019)	0.46	1.2	not specifiend	-	ASTM I, Grade 42.5	undensified SF	Class F	-	-

⁶ ANC-Attapulgit Nanoclay

⁷ CSA-Calcium Sulfoaluminate Cement,

⁸ GS-Ground silica, MS-Microsilica

⁹ SP-Superplasticizer, R-Retarder, VMA-Viscosity Modifying Admixture, HRWRA-High Range Water Reducing Admixture, HPMC- Hydroxypropylmethylcellulose, SRA-Slump Retention Agent

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