

**EXPERIMENTAL DESIGN OF NOVEL GEOPOLYMER
MIXTURE FOR CONSTRUCTION 3D PRINTING**

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**Submitted in fulfillment of the requirements
for the degree of Master of Science
in Mechanical & Aerospace Engineering**



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April 2023

DECLARATION

I hereby, declare that this manuscript, entitled “*Experimental Design of Novel Geopolymer Mixture for Construction 3D Printing*”, is the result of my own work except for quotations and citations, which have been duly acknowledged.

I also declare that, to the best of my knowledge and belief, it has not been previously or concurrently submitted, in whole or in part, for any other degree or diploma at Nazarbayev University or any other national or international institution.



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Abstract

Additive manufacturing has become a crucial tool for modernizing various industries, including construction engineering, and has recently led to significant interest in the application of 3D printer technologies. 3D printing of concrete-based material is a relatively modern approach that enables the construction of objects' creation without formwork and offers numerous benefits. However, the implementation of 3D printing technologies in the building industry is restricted because of technological weaknesses, including the need for mechanical control and appropriate printing materials.

The primary aim of the research is to design and develop novel geopolymer mixture for construction 3D printing utilizing locally sourced raw materials. The proposed methodology focuses on exploring the components and parameters of the entire structure necessary for successful extrusion design, determining the ideal proportions and mixture of geopolymer for the extrusion process, and assessing the microwave heating methods of geopolymers' potential for industrial-scale production. The main results of this research study are the development of an optimal extrusion design for 3D printed geopolymer materials, guidelines for the pre-duction of geopolymers, and the evaluation of the mechanical properties for various construction applications. The contribution to knowledge lies on its proposed methodology, which is an enabler endeavor toward 3D construction printing among building companies in Kazakhstan, an economically affordable 3D construction printing system using geopolymer, and encouraging the industrial production of the manufacturing of geopolymers with microwave assistance.

The industrial benefits include increasing the use of geopolymer as an eco-friendly substitute for regular Portland cement, lowering carbon dioxide emissions, and using fewer resources. Furthermore, this study results can provide significant economic and architectural advantages to the construction industry by enabling the creation of structures with complicated designs and reducing labor costs and formwork.

Keywords: Additive manufacturing, Extrusion design, Geopolymer, Construction industry.

Acknowledgments

I would like to take this opportunity to express my deepest appreciation and gratitude to Professor Md. Ali and Professor Essam Shehab, my thesis supervisors, for their invaluable support, guidance, and encouragement throughout this journey. Their mentorship and expertise have been indispensable in shaping the outcome of my research.

Professor Md. Ali, with his vast knowledge and experience, has provided me with insights, direction, and motivation throughout the entire research process. His unwavering commitment to excellence, coupled with his patience and understanding, has been instrumental in helping me to overcome the various challenges that I have encountered along the way.

The success of this thesis work was also largely contributed by Professor Essam Shehab. His exceptional knowledge has been of immense value in shaping the direction of my research. His constructive feedback, guidance, and suggestions have been invaluable in helping me to refine my ideas and to present them in the best possible way.

I would like to thank my parents for instilling in me a love of learning and for their unconditional love and support. My siblings have been my constant companions, cheering me on through the ups and downs of my academic journey. To my friends, thank you for being there for me, for encouraging me, and for believing in me.

Lastly, I would like to thank the faculty and staff of Nazarbayev University, whose dedication and hard work have made it possible for me to pursue my academic goals. Their commitment to excellence has been an inspiration to me, and I am grateful for their support.

List of Publication

- [1] A. Baigarina, E. Shehab, and M. H. Ali, "Construction 3D printing: a critical review and future research directions," *Progress in Additive Manufacturing*, <https://doi.org/10.1007/s40964-023-00409-8>, pp. 1-29, 2023.

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List of Abbreviations & Symbols

FA	Fly Ash
GGBS	Ground granulated blast-furnace slag
SF	Silica fume
OPC	Ordinary Portland Cement
XRF	X-ray fluorescence

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Chapter 1 – Introduction

1.1 Research Background

Additive manufacturing is a crucial tool for the modernization of industries such as medicine, food, automotive, and military [2]. Construction engineering's interest in applying 3D printer technologies has recently risen significantly [3]. Compared with traditional technologies, concrete 3D printing is the newest approach that allows the creation of construction objects without formwork. In addition, implementing AM in this particular sector has numerous benefits, such as i) freedom construction - the production of new structures; ii) mechanized construction - the accomplishment of automotive construction, which minimizes labor costs and formwork; and iii) eco-friendly construction – the analysis of construction materials, which reduces the consumption of materials [4]. However, only a few accomplished tests have led to production, and 3D printing projects are still imperfect [5].

A method of construction using cement molding technology, in which steam is used to connect the material in layers, was proposed by Pegna et al. [6], but this method was not interpreted conceptually. The innovative work of contour Crafting conducted by Khoshnevis et al. [7] demonstrated the applicability of AM method in the construction sector. Several years later, another AM technique motivated by stereolithography appeared, known as particle bed printing [8, 9]. After that, a dynamic casting approach was designed, which uses the technology of forming a slip with a robotically moving formwork [10]. Nonetheless, an extrusion-based process is nowadays the most widely employed technology for concrete AM in the construction sector [11].

1.2 Problem Statement and Motivation

The extrusion-based method of AM is the primarily used approach in the building sector, enabling construction elements with complicated designs. Moreover, this technique can use conventional building materials, including plaster, cement, or geopolymers. However, in comparison with other areas in the building sector, AM methods are mostly adopted gradually, and their implementation is restricted because of technological weaknesses [12]. Namely, in the building sector, research that should be focused on mechanical control and appropriate printing materials is required [13]. Moreover, there are no specific standards or guidelines for the production of geopolymers for 3D printing technologies. Therefore, this research's main objective is to investigate an optimal extrusion design to control the movement of the material and to determine the appropriate system mixture.

3D printers using geopolymers are one of the rapidly developing areas of additive technologies. This is mainly because industrial waste, including red mud, slag, or fly ash, might be used as a precursor to geopolymer [14]. Accordingly, developing extrusion design for 3D printed geopolymers, to some extent, will minimize carbon dioxide emissions and energy consumption by replacing Ordinary Portland cement (OPC) and allow geopolymers to be considered an ecologically alternative option [15].

1.3 Aim and Objectives

The primary aim of the research is to design and develop novel geopolymer mixture for construction 3D printing utilizing locally sourced raw materials.

The main objectives are to:

1. Identify components and parameters of the entire structure necessary for the successful extrusion design;
2. Determine the ideal proportion and mixture of geopolymer for the extrusion process;
3. Investigate the use of microwave curing to accelerate the formation of the geopolymer concrete structure.
4. Validate the results of the study by testing the mechanical properties of geopolymer structures.

The primary research questions are as follows:

- What types of extrusion designs are available for 3D-printed geopolymers?
- What are their advantages and challenges?
- What component ratios are necessary to obtain geopolymers for 3D printing?
- What kind of methods exists to microwave the heat of geopolymers?

1.4 Scope and Constraints

The primary scope of the thesis is shown in Figure 1. The scope of this research covers the following aspects: firstly, an examination of the existing extrusion designs; secondly, the development of a novel extrusion design specifically tailored for the 3D printing; thirdly, the preparation of a geopolymer mixture. The fourth aspect concerns the use of microwave heating to enhance the properties of the geopolymer. It is essential to mention that this research work does not encompass an evaluation of other additive manufacturing techniques or a comprehensive assessment of various materials commonly used in 3D printing.

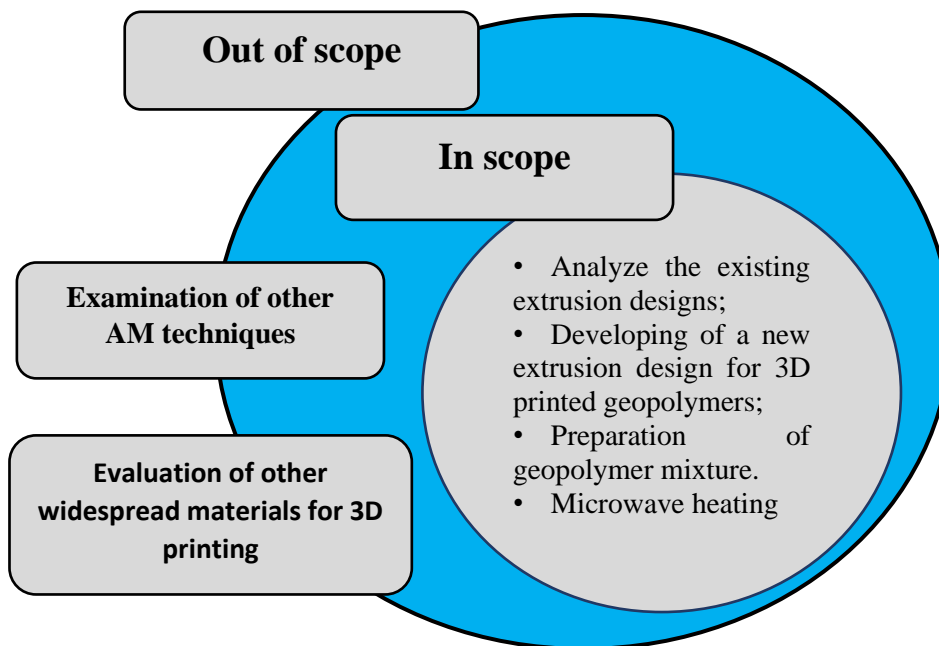


Figure 1. Scope of the research

Although several restrictions might have occurred throughout the study process:

- The costly price of additional systems.
- An insufficient number of scientific publications and approaches to extrusion design for 3D-printed geopolymers.
- The need to acquire additional knowledge, and computer software.

1.5 Thesis Structure

The chapters of the thesis research are as follows: Introduction, Literature Review, Research Methodology, Experimental Results, Conclusion and Future Work. Each chapter has a specific purpose and is described in detail below.

Chapter 1: Introduction

- Research Background, Motivation
- Definition of Aim and Objectives
- Determination of the Scope and Constraints
- Overview of the research work structure

Chapter 2: Literature Review

- Overview of existing 3D construction printing technologies
- Examination of the Extrusion Printing process
- Evaluation of 3D Printing based materials for construction
- Review of investigation of geopolymer as a building material
- Analyzing of Microwave heating process to improve material properties

- Identification of the Research Gap of existing extrusion-based printers and geopolymer compositions

Chapter 3: Research Methodology

- Explanation of the adopted research structure
- Determination of geopolymer design and preparation of the material
- Development of 3D printable material
- Solidification of prepared material through microwave heating
- Identification of the geopolymer with relatively best properties and validation

Chapter 4: Experimental Results

- Illustration of the outcomes of compressive strength
- Representation of the outcomes of flexural strength
- Explanation of setting time results
- Depiction of microwave-assisted solidification

Chapter 5: Conclusion and Future Work

- Summary of the thesis research
- Contribution to the knowledge
- Identification of Future work directions

Chapter 2 – Literature review

2.1 Introduction

AM has been implemented in the building industry for more than a decade. The main aspects of this area have been reviewed in this chapter and illustrated in Figure 2. Some essential benefits of AM are reduced waste and labor, automation, freedom of design, and the creation of complicated designs. Initial research on 3DCP began at Loughborough University with a focus on the development of mixtures [16]. Afterward, experiments at the Technical University of Eindhoven examined its potential and drawbacks in more detail [17]. For example, the Chinese company WinSun advantageously produces a one-story house [18] and in Norway, they build a bridge for bicycles [19]. Additionally, the US Army Corps began printing both municipal and military buildings simultaneously [20] After that, a few further massive printed constructions were built in various locations throughout the world [21], [22], [23].

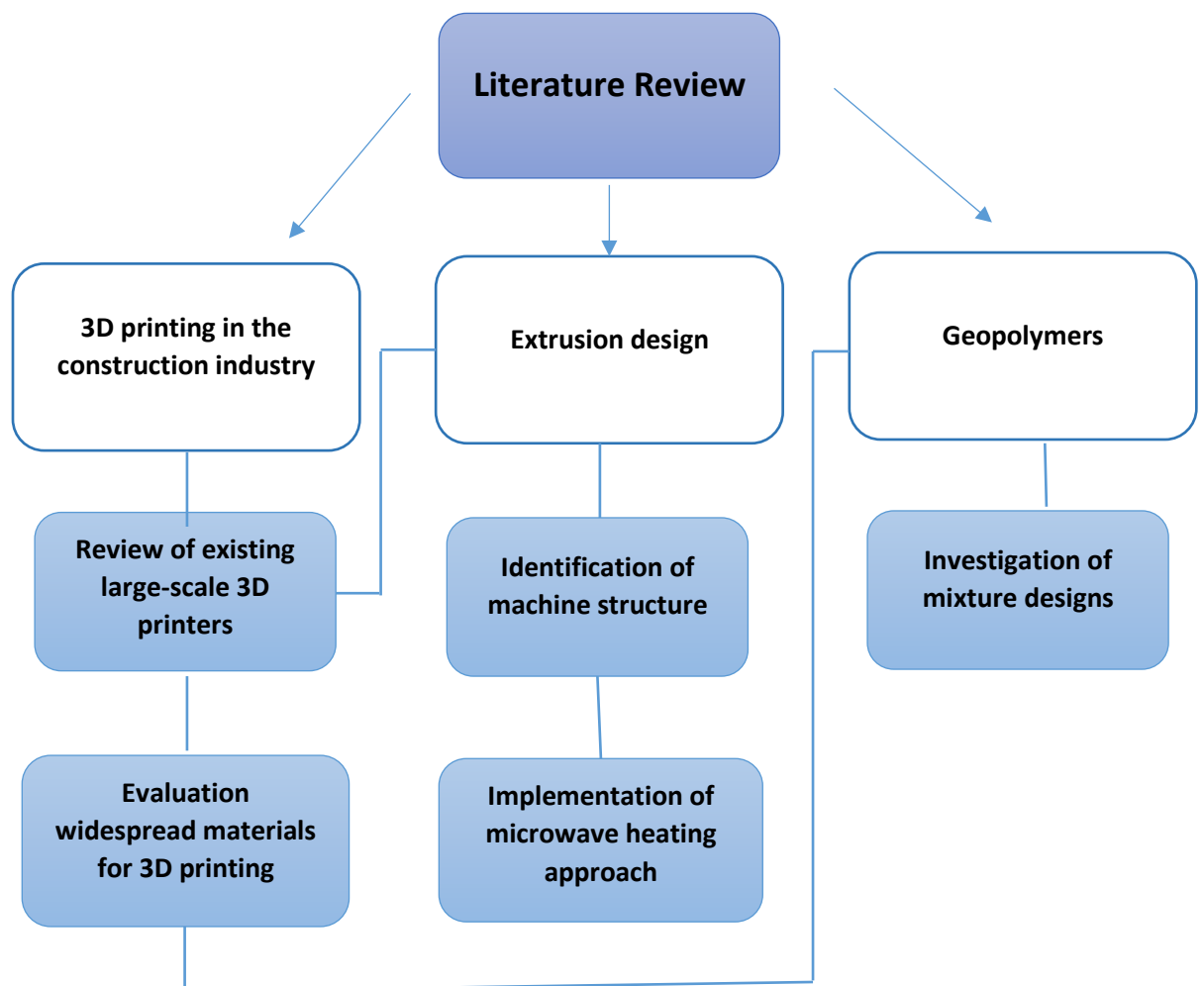


Figure 2. Literature review areas

Geopolymers can benefit from a variety of current technological developments that have been made for the additive manufacture of cementing materials. Extrusion-based 3D printing techniques and powder-based AM methods are the two basic groups into which they can be categorized [24], as illustrated in Figure 3. The 3DCP extrusion design is identical to the Fused Deposition Modeling (FDM) process, which produces a concrete component by extruding the cementing material out of a nozzle. This kind of method is intended for use in on-site construction. However, a considerable limitation of extrusion printers related to the restricted range of concrete that is applicable for printing. Traditional concretes are not appropriate for 3D printers based on extrusion, and OPC parameters restrict their use for 3D printing [25]. OPC could be replaced by a geopolymer obtained from industrial waste, such as slag or fly ash. The geopolymer also offers outstanding benefits over OPC, including higher strength, improved fire and sulfate resistance, and CO₂ emissions from products that are nearly 80% lower than OPC [26].

To expand the horizons of AM implementation, researchers around the world are trying to solve the problem associated with a limited assortment of concrete suitable for printing. In addition, there is a demand for novel approaches for obtaining better properties of 3D-printed geopolymers for construction applications.

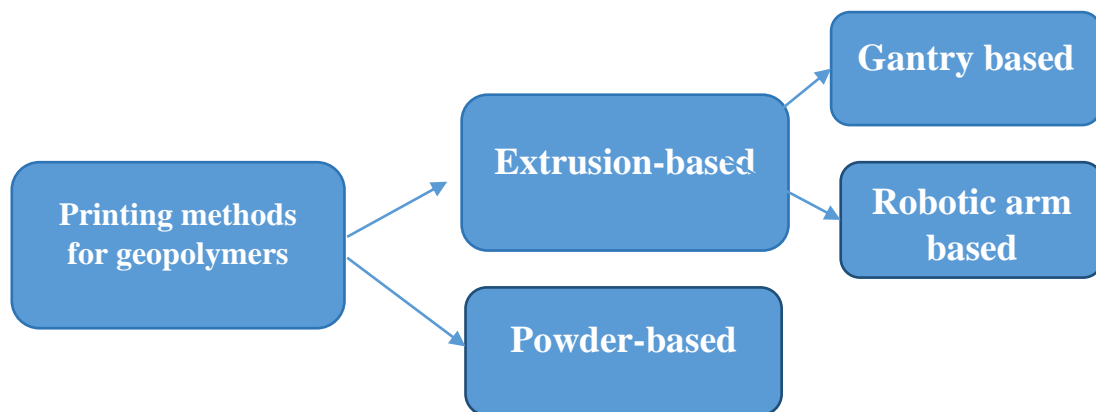


Figure 3. Printing techniques for 3D geopolymer printing

2.2 Widespread 3D construction printing technologies

Currently, this sector is dominated by three popular large-sized AM technology types: D-shape [29], Concrete Printing (CP) [28], and Contour Crafting (CC) [27]. Every technology has a

few special features. The three techniques all have some printing-related qualities in common and are designed for printing concrete. Table 1 lists the three techniques' principal applications, hardened features, and materials.

The University of Southern California (USC), which was the first to realize the potential of AM, developed contour crafting. The additive manufacturing process known as contour crafting (CC) makes use of the surface-forming characteristics of troweling to create smooth, accurate, flat, and free-form surfaces. Structures may be built effectively and sustainably, responding to external conditions, thanks to the versatility that contour crafting allows in the building of concrete shapes, which previously very difficult to do with the conventional method of pouring [30].

3D concrete printing is a well-known technique that may reduce the supply chain in the building industry. Engineers at Loughborough University were successful in their first attempt to manufacture concrete printing. Reducing the nozzle's nonprinting movements in this process speeds up printing [31]. Pre-packaged mortars were used as a building material, and gypsum was used as a support material, by optimizing variables such as flow rate, rheological properties, size, and shape [32].

The D-shape approach was created by Enrico Dini and displayed at the London Civils 2007 exhibition [29]. The major use of this D-Shape 3D printing technique is as an enormous Z Corp printer. The technique was developed for scaling up 3D printing procedures so that massive, reasonably priced objects the size of constructions may be manufactured [33].

The similarities, as well as the distinctions between all three techniques, were examined by Lim et al. [16]. Despite their additive construction, they have been created for a variety of materials and implementations and offer unique advantages. While both CP and CC are built on the extrusion of a cement-based substance, the former allows for the creation of a smooth surface finish with the addition of consecutive layers, while the latter has a lower resolution. Additionally, CC was created as a crane tool for in-situ and on-site purposes, whilst CP and D-shape were developed for off-site applications [16].

Table 1: AM technologies and their main characteristics [16].

Method	Build Material	Essential parameters			Mechanical properties	Flexural strength	Compressive strength	Application	Advantages	Disadvantages
		Layer thickness	Nozzle diameter	The number of nozzles						
Contour Crafting	Concrete	13 mm	15 mm	1		Unknown	Unknown	Large-scale structures	Smooth surface	Molding requirements = extra process
Concrete Printing	Concrete	4 - 6 mm	9 - 20 mm	1	The force was applied from the top of the printed surface	12—13 MPa	100-110 MPa	Structure, Formwork	Minimal printing process	Limited print size by printed frame
D-Shaping	Sandstone	5 mm	0.15 mm	300 nozzles that are 20 mm apart from each other		235-242 MPa	14—19 MPa	Formwork	High strengths	<ul style="list-style-type: none"> • rough surface • removing unused materials • relatively slow process

2.2.2 Contour-Crafting in the use of construction

The Contour-Crafting method's central idea (Figure 4) is around the use of extrusion and filling for generating large designs [34, 35]. For instance, the CC approach uses the 3D printing head to extrude concrete [36]. Additionally, the technology makes sure that trowels are fastened to the nozzle's side [37]. It is claimed that contour crafting offers potential applications in two different industries: (1) inexpensive housing or insulated urgent housing; and (2) building designs with complex forms that would be pricey to build using traditional methods.

But according to Bhardwaj et al., using contour crafting to build gigantic buildings necessitates using enormous gantries, which creates other problems. Compared to this, cables would be much easier to move and deploy [38]. So, scientists look into and evaluate a cable-based technique for contour sculpting. The concept of Cable-Suspended Contour Craft Construction (C4) was established [39]. It was made composed of a rigid frame and an end effector hanging from 12 cables (four uppers and eight lowers). The eight bottom cables were also split into four parallel pairs. Using the robot's available workspace and the related cable tension, the scientists approximated the largest size at 44 m*44 m*40 m over the given cubic frame construction having

a length of 50 m. They looked into statics and kinematics, but only provided evidence of translational motion [39].

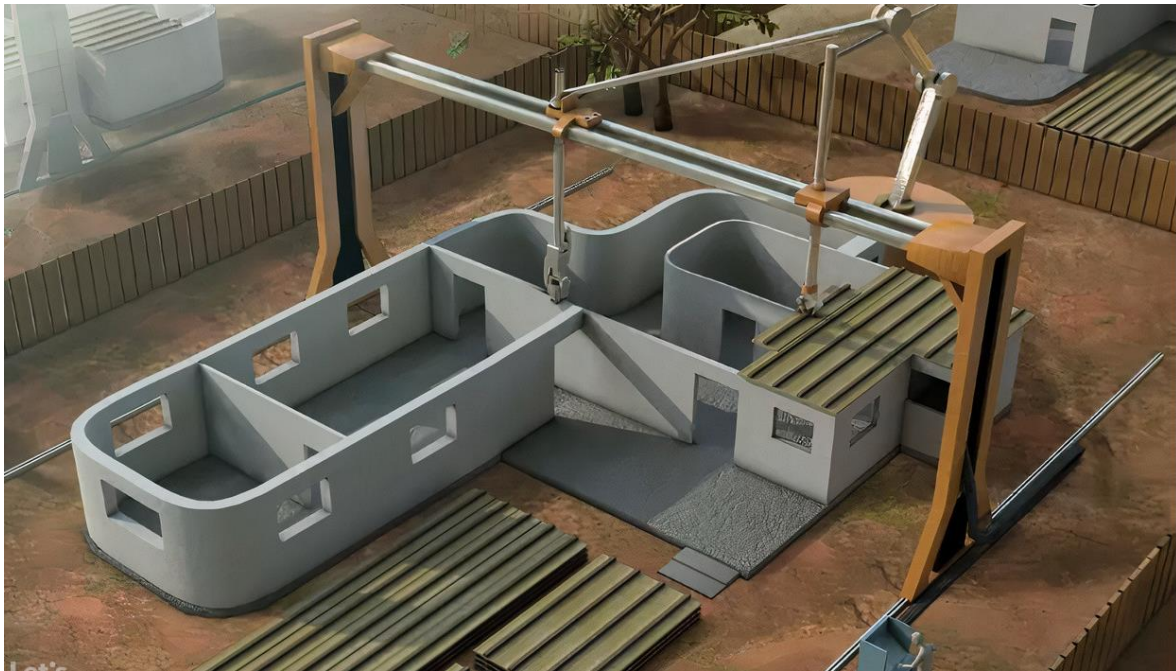


Figure 4. Contour Crafring

However, alternative technologies exist that are identical to CC, like BetAbram [40], WinSun [41], ICON [42], and CONPrint3D [43].

2.2.3 Current state of D-shape in construction

The first attempt to create a conglomerate structure using additive manufacturing was undertaken in Buti, Italy, in 2008. The ephemeral shell was made using a 3D printer by sending a 3D data file to a computer, where it was converted into an STL file. The structure of the 3D-printed conglomerate was finally revealed after extracting the unconstrained combination and shell [9]. D-shape can produce architectural structures that are up to 6 by 6 m in size utilizing a powder-bed printing system and a production gantry [44]. This method's main idea is based on 3DP and aims to create architectural artifacts by using an inorganic bond with a layer thickness of between 5 and 10 millimeters. The printing medium is thought to be comparable to marble [28]. Instead of cement-like ingredients, this procedure makes use of powder layers and adhesives. As in a conventional 3D printing procedure, it involves a powder deposit that is selectively solidified using a binder [45].

The present multi-size, multi-particle, or multi-binder AM device is being produced by D-Shape. The printhead's hydraulic tools can currently be used to pump water or concrete mortars. The particle layering apparatus can now spread particles of various sizes, from powder particles to a few millimeters, evenly [9]. The most popular aggregate particles have sizes between 0.2 and 4 mm. Layers are typically five millimeters thick [9].

The D-shape technique outperforms alternative 3D printing methods of construction and traditional constructive procedures [46]. While the technique does provide architects greater creative freedom, it also comes with a lot of material requirements because the entire build surface needs to be covered in sand or other particles to reach the top of the print. Before the print can be used, laboriously remove the extra powder substance that preserves its framework throughout the curing process [44].

2.2.4 Worldwide approaches of AM in the building sector

A 200 m² cluster of homes were profitably printed in 2014 by WinSun, a Chinese company (Figure 5). The company used a big scale equipment to produce large-scale building components [47]. The 120-foot-long printer that produced the structure is 20 feet tall, 40 feet wide, and mobile. The material that is extruded from the printer's nozzle is made of concrete, fiber content-reinforced polymers, mortar, and glassy fiber-reinforced gypsum connected by a unique ingredient that fully enables the concrete to cure in a few days. 50% of this material is composed of repurposed construction waste [48]. The construction industry partners' interest in this area of application makes this technology a viable advance even though WinSun hasn't supplied specific information on the mechanical qualities of their printed buildings. Dubai, for instance, projects that by 2030, 30% of its buildings will be printed [49].



Figure 5. WinSun [41].

One example of how AM technology, as seen in Figure 6, makes it practicable to produce a house on-site to reduce building costs is Apis Cor [50]. The 3D printer's weight of 2 tons and overall dimensions of 4 by 1,6 by 1,5 meters allow for an expansion of the production surface to 131 m². Users can utilize several coordinated 3D printers to create large structures like houses

[51]. A San Francisco business created a house utilizing a 3D printer in Russia at the end of 2016 in just one day. The house has a 38 square meter space, and it cost \$10,134 in total. The printer has a maximum height and breadth of little more than 3 and 5 m, respectively [52].



Figure 6. Apis Cor [50].

A two-story house was printed totally on-site by Hua Shang Tengda in China (Figure 7) [53]. Off-site Given that transferring and installing 3DCP on-site will cost an enormous amount of money and work, it might not be the ideal choice [54]. The delivery cost may exceed the savings resulting from modular construction of homes, particularly for a dwelling, based on a similar statement. The two-story house was constructed right there on the spot, but it only took a month and a half to complete the job from start to finish, comprising plumbing and other fittings. Contrarily, an equivalent structure constructed conventionally would take roughly 6-7 months to complete.



Figure 7. Hua Shang Tengda [53].

The Dutch company CyBe Construction (DEWA) 3D printed the drone research and development center to serve the Dubai Electricity and Water Authority. The 168 m² structure was produced on-site in about three weeks employing a CyBe RC 3Dp device. The portable 3D printer from CyBe's main goal is to provide a dependable method for creating cement in three dimensions on a building site [55]. Figure 8 shows the computerized layout of this laboratory.

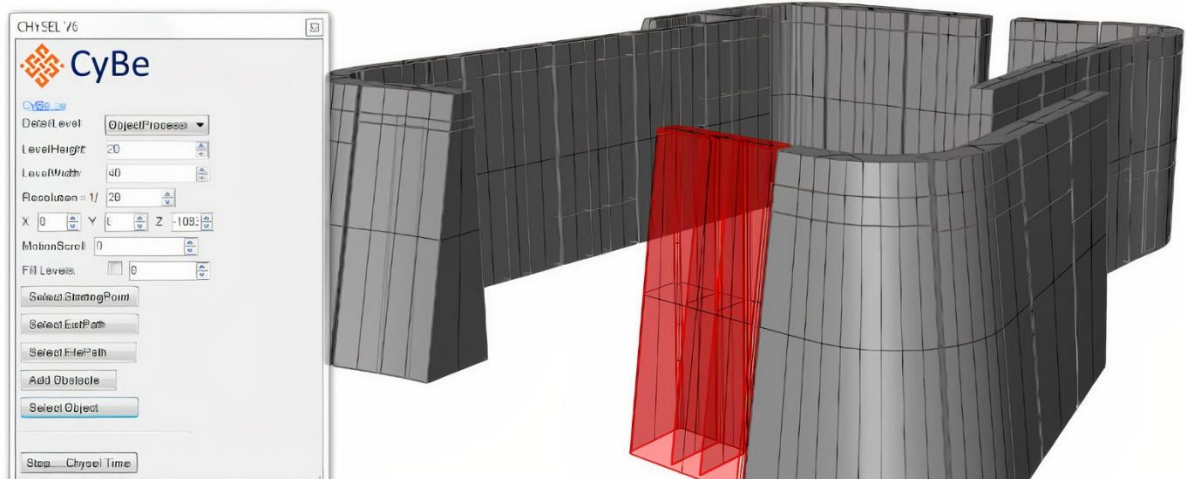


Figure 8. The Dubai laboratory's digital design [55].

The Eindhoven University of Technology and the Dutch construction firm BAM Infrastructure Group produced and constructed a 3D-printed concrete cycling bridge in the Netherlands in September 2017 [19]. In June 2017, the preparation and design phase of the manufacturing process got underway. The bridge measured 8 meters in length and 3.5 meters in width, making it the first of its sort in the Netherlands. The printed components of the bridge were

created using strengthened and reinforced concrete. Cycling enthusiasts can use the bridge to pass through Gemert's Pelshe sewer loop [56] [57].



Figure 9. Gemert's 3D-printed bicycle bridge [58]

Table 2 summarizes 3D-printed worldwide models with the advantages and limitations of the different technological methods.

Table 2: Summary of worldwide models

No	Manufacturer	Name of object	Place	Size of the printed model	Year of completion	Advantages	Limitations	References
1	WinSun	Series of houses	China	200 m ²	2014	<ul style="list-style-type: none"> • 30% time decrease compared to similar buildings constructed utilizing conventional techniques • able to save construction and labor expenses by around 80% compared to conventional on-site construction techniques • 30–60% reduced material waste compared to conventional concrete placement 	<ul style="list-style-type: none"> • only able to produce building components, not complete systems • traditionally built foundations, reinforcing, and MEPF retrofitting will always be required 	[48] [47]
2	Apis Cor	Residential House	Russia	38 m ²	2016	<ul style="list-style-type: none"> • reduces costs by up to 40% compared to conventional production • only two people are required to deliver and control the material; else, the process is fully automated • 3D printers do not produce excessive manufacturing waste • Setup takes less than 30 minutes 	building components' scale and size may restrict 3D printing to a single technique.	[52] [51]; [59]

3	Hua Shang Tengda	Two-story house	China	400m ²	2016	<ul style="list-style-type: none"> • can resist earthquakes of a Richter magnitude of up to eight • the increased speed 	choosing the right concrete mix to meet structural requirements for earthquake resistance presents obstacles.	[53] [60]
4	CyBe	Laboratory	UAE	168 m ²	2017	<ul style="list-style-type: none"> • the on-site 3D printing process • mortar sets in five minutes • it has less CO₂ emission and waste 	Not Found	[55] [57]
5	The Eindhoven University of Technology & BAM Infrastructure group	Bridge	Netherlands	28 m ²	2017	<ul style="list-style-type: none"> • printed for public usage • whereas concrete is often poured in, there is no need for shutters • no release of CO₂ emissions 	the printing process is not certified	[56] [57] [19]

2.3 Extrusion Printing

The widely used type of AM in the building industry is an extrusion-based method, which involves depositing material from the nozzle layer upon layer to build the product [61]. This method relies on layer-by-layer extrusion of geopolymer paste through a nozzle to produce the final product. An arrangement for shifting the nozzle concerning the platform across the designated coordinates is implemented by gantry-type 3D printers. However, other types of printers use a kinematic system modeled by a robotic arm to move their mechanical components. This type of 3D printer can cover a vast working area, making it possible to produce free-form components accurately. In contrast to gantry systems, it is more challenging to achieve the smooth movement needed for printing in this kind of process [24].

Figure 10 depicts the usual 3D concrete printing (3DCP) procedure for the building sector. Two techniques can be identified when looking at the complete 3D printing process. First, there is the software part, followed by the hardware part. The objects are initially modeled in a 3D program like AutoCAD or Solid Works before being exported to another program for slicing. The entire object is then produced as a G-code so that the printer may use it and complete the task [62].

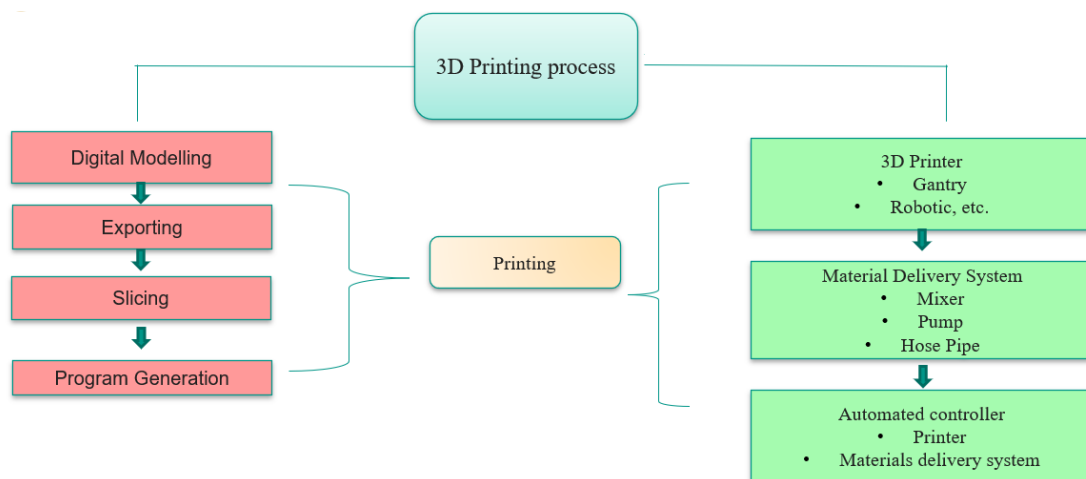


Figure 10: The 3D printing method for the building sector [62]

2.3.1 3D Printer Structures

The primary extrusion-based 3DPG printing procedure is demonstrated in Figure 11. Typically, the pumping system transfers the material to the extruder where it is deposited sequentially until the desired form is obtained. Controlling the quality of each procedure is crucial for the successful on-site printing of a geopolymer combination [61].

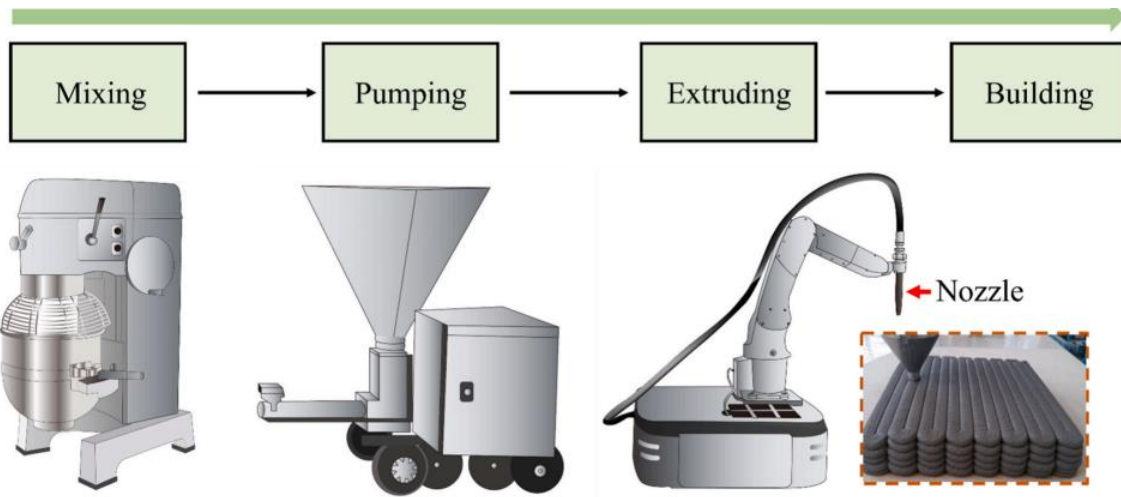


Figure 11: Printing stages for 3DPG [61]

The configuration of the AM apparatus used operated for study is typically briefly described in studies. It is not properly explained how the machine architectures were created or how the components were chosen. To provide probes for the examination of quality and other attributes are shown in research by Jo et al. [13], Muthukrishnan et al. [63], and Arunothayan et al. [64], used printers are routinely scaled. The same essential elements are used in all printer configurations. In a tank or other container, the new mixed material is kept. The material is pumped to the moving print head and nozzle using a pump.

Figure 12 illustrates a setup for printing concrete in three dimensions at a laboratory scale, with the size 1* 1* 1 m [13]. The moving frame that adjusts to the X and Y axes has the nozzle installed on the Z axis in order to print concrete structures. A servo motor is necessary for the linear movement along each axis. Frames might be impediments that restrict the workspace, but they typically stiffen the entire system and ensure precise movement. The dispensing system is a 3D printer's most important technological component for producing adequate and dependable

concrete volumes. According to the relevant material qualities, such as their compressive strength and stacking capacities, extrusion control in the dispenser system should be increased [13].

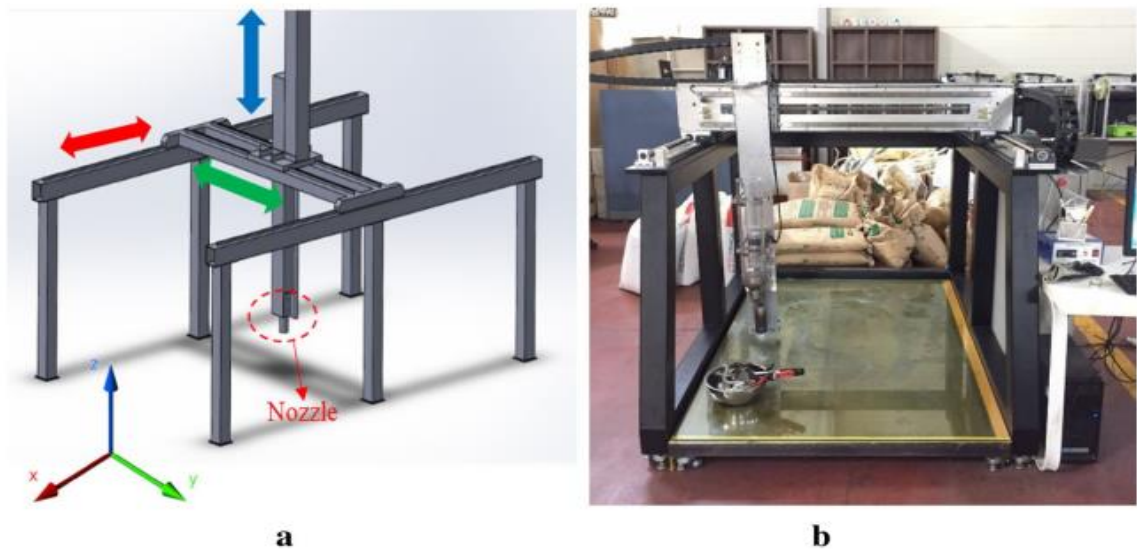


Figure 12. a) Schematic representation of 3d printer; b) developed 3d printer

A screw-type extruder made by Mutukrishnan et al., installed on a 3-axis rail system, allows [63] a 3D printer to move precisely (Figure 13). The dimensions of this printer are $2.4*2.0*2.1 m^3$. In this apparatus, the extruder comprises an auger, agitator, and nozzle for the material to be continuously sheared, displaced positively, and deposited under control. An auger with a speed motor is added to change the extrusion rate. Between 0 and 2.5 rad/s can be selected as the auger's rotational speed [63].

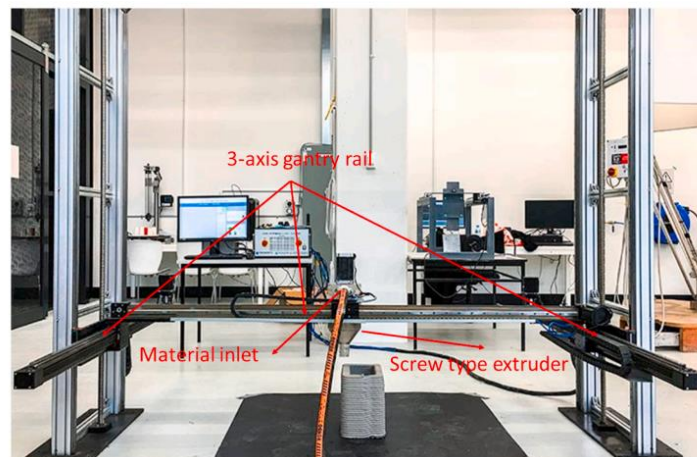


Figure 13: 3D printer used in Mutukrishnan's work [63]

In the study done by Arunothayan et al. [64], a custom-created printer was employed to imitate the extrusion type 3DCP method, as illustrated in Figure 14. The printer employed an extruder with a 45° -inclined rectangular head measuring 30 mm (W) by 15 mm (H). A metal

cylinder of 600 mm in length and 50 mm in diameter served as the extruder. The printer motor managed the printer's movement on the Cartesian plane.

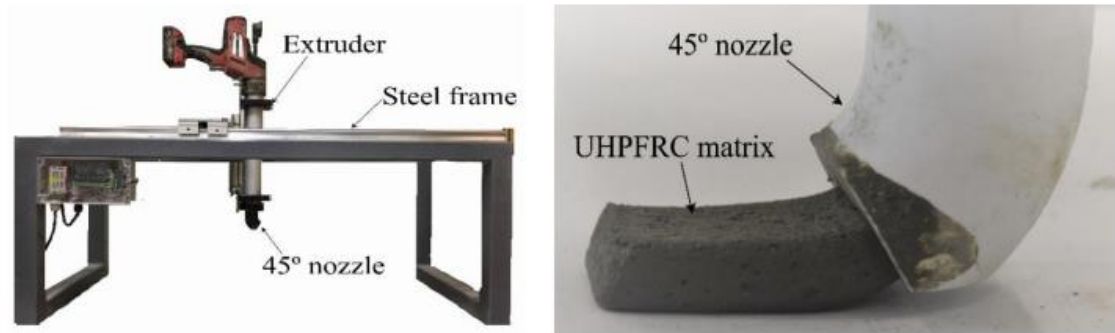


Figure 14: 3D printer used in the study by Arunothayan et al. [64]

The gantry printer used by Lao et al. [65] offers one degree of freedom for rotation and three more for translation (Figure 15). The gantry was used as a nozzle placement device to direct the nozzle's movement following the printing trajectory.



Figure 15: Gantry printer used by Lao et al. [65]

2.3.2 Mixers and pumps

The mixing process, which involves the suitable materials being added to the mixer in the proper order, the power and time of the mixing must be carefully handled for the final product of concrete material. Mixers in concrete production can produce in batches or continuously. Depending on the dimensions of the mixer, batch mixers can combine a specific volume of material. The printed material is typically mixed gradually. Typically, it is impossible to mix all the materials required for an object's printing at once. As the material reservoir empties, new, fresh material is mixed, causing a difference in the printed material age; as a result, the pressure must be managed appropriately [17].

Studies of pumps utilized by concrete production and 3D printing applications were reviewed to identify references to the various types of pumps used or that could be employed in

the 3D printing of geopolymers. Pumps transport material from the mixing to the nozzle [66]. Large-scale pumps are used in concrete construction. Reciprocating pumps and high-capacity twin-piston pumps are the most popular types of concrete pumps [66].

There was no mention of using the pumps in 3D printing applications. Concrete pumps must be able to carry abrasive substances contained in the pumped material. In cases of severe wear, maintenance of the pump should be provided [67]. The most common types of pumps used in the 3D printing of concrete are volumetric pumps [11]. Even though they have a pulsating flow, some studies use peristaltic pumps, although a constant flow is usually required for optimal extrusion [3].

2.3.4 Printer heads and nozzles

The nozzle's principal function is to distribute material among layers evenly and give the ejected layer its shape. Four different nozzle shapes—round, elliptical, rectangular, and square—are typically employed for extrusion [66]. The dimensions of rectangular nozzles usually ranged from 9x6 to 38x15mm [66], and the round nozzle hole usually ranges from 6 to 50 mm. The extruded thread is usually visually checked for deformations, splits, and breaks to determine the extrusion quality [11]. The nozzles are often made of steel or similar hollow material and most often have a round or rectangular shape [17]. Extrusion technology has also been used to create printer nozzles made of plastic. Figure 16 shows that the printer nozzle can be made of PVC plastic. The orifice's form and how it impacts the extruded filament's quality have become the primary research areas for nozzles. The surface finish, mechanical strength, and compactness of the nozzle shape can all be enhanced [68].



Figure 16: Nozzle with a 45-degree angle on its rectangular opening [69]

2.4 3D Printing based materials for construction

In large-scale construction, three-dimensional printing technology is used to produce formally complicated building enclosures. For the comfort of the occupants, energy effectiveness, and material durability, the enclosing structure of the building—which serves as a barrier between the inside and the outside—is crucial. It serves four main functions: distribution, finish, control, and support [70]. Consequently, 3D-printed building enclosures must do each of those functions. According to recent research, concrete, and polymer represent the two main building materials utilized in 3D-printed structures, as indicated in Table 3 [71]. The findings of the research conducted to comprehend the materials frequently utilized in construction engineering are compiled in this part.

Table 3: 3D Printing based materials for the construction industry

Material	Advantages	Limitations	References
<i>Concrete</i>	<ul style="list-style-type: none"> • superior material handling and positioning flexibility; • low-cost access to necessary materials • quicker construction; • flexibility in design 	<ul style="list-style-type: none"> • rate of vertical construction • extrusion-based horizontal technology • the use of reinforcement • Subpar surface luster 	[72]; [73]
<i>Polymer</i>	<ul style="list-style-type: none"> • low weight • process adaptability • high rate of use • simple production method 	<ul style="list-style-type: none"> • absence of structural attributes • Young's modulus for 3D printing qualities that are not satisfied 	[74]; [75]; [76]
<i>Geopolymer</i>	<ul style="list-style-type: none"> • ecologically sound building products • strong compressive capacity • high adaptability • minimal drying shrinkage • better sulfuric acid attack • enhanced resilience to acid and fire attacks 	<ul style="list-style-type: none"> • the time being set slowly • strengthening requires heat curing. 	[77]; [78]; [79]

2.4.1 Concrete material

As reported by Wangler et al.[72], approximately 2 billion tonnes of consumption per year, concrete is the second most utilized substance in the world, after water. The most frequent justification for the extensive concrete execution is the availability and affordability of necessary resources. Half of the expenditures associated with concrete construction can be attributed to scaffolding and molds. Traditional concrete methods promote simple, repetitive formwork to reduce costs. Concrete additive manufacturing processes have advanced significantly as a result of the numerous opportunities for significant savings in costs they offer. Concrete printed with additives is more advanced and utilized in construction than metal or polymers printing [80]. One of the key elements influencing its appeal as a building material is its natural transformation coming from a liquid into a solid form, allowing it to flow and refill molds and, following hardening, support loads. Additionally, concrete offers exceptional material handling and installation flexibility, both of which the world of digital manufacturing has not yet fully tapped into [72].

The "third industrial revolution" of digital fabrication uses digital modeling and technology to create unique materials [81]. It encompasses 3D printing, a branch of digital manufacturing that, despite recent emphasis, is still evolving in the fields of design and construction [16, 82]. The fundamental advantage of 3DCP lies in how elaborate, uncommon shapes and details may be swiftly created by using a printer that also functions as a pump, hosepipe, and nozzle. There are still certain property problems with 3D printing because it is different from conventional production. In contrast to traditional construction, where the intended form of the structure is attained by flowing concrete into a predetermined formwork, 3D printing achieves the desired shape by extruding concrete layer by layer. As a result, newly formed concrete parameters and printing settings, such as printing time and speed, have a significant impact on the hardened qualities of 3D-printed objects. While 3DCP eliminates the requirement for formwork to hold the concrete layers and necessitates substance to be forced to the nozzle for extrusion, the rheology of the new characteristics of printing material is critical [80].

One of the main disadvantages of printed materials is that, unlike cast specimens, which are presumed to have isotropic characteristics in all directions, printed materials exhibit combined isotropic and anisotropic properties in some directions. Additionally, factors like material flow properties, printing speed, and time intervals between consecutive layers all significantly affect the final 3D-printed goods [83]. It is expected that material suitability will be more important in the horizontal plane than in the vertical plane in 3D printing because the final product is constructed layer by layer [84].

When concrete is in a plastic state, it is easy to deal with, enabling the extrusion process considerably simpler. However, because the hydration of concrete happens gradually over time, it will eventually reach its setting condition. In this circumstance, concrete extrusion is challenging. Additionally, the concrete's hardening qualities may change significantly if it is disturbed following it has attained its plastic phase. Large-scale 3D printing of concrete initially produces prints of acceptable quality, but over time, the hydration process speeds up and the matrix solidifies (converting plastic to a setting state). The water in the mixture could evaporate over time, lowering the printing quality.

As a result, the bottom layers, which were printed much earlier, may have created connectors and concrete layers of lower quality than the upper layers. When the object is larger and printing takes longer, this phenomenon is unquestionably true. For the same reason, the mechanical properties of the specimens obtained from different positions of the item would similarly differ greatly [62].

2.4.2 Composition of cement-based materials

A cement paste is created when water and a cementitious substance are combined during the hydration process. A combination of calcium, silicon, and aluminum oxides creates typical portland cement. When clay and limestone (a calcium source) are heated, the result is termed clinker, which is then ground with a sulfate source to produce Portland cement and comparable compounds (most commonly gypsum). The majority of a concrete mixture comprises fine and coarse particles. The most common materials used are sand, gravel, and crushed stone. Portland cement and water quickly gel when combined because the gel is made of tangled chains of interlocking crystals. These continue to change over time, and the initially fluid gel frequently helps in placement by making it easier to work. As the concrete hardens, the crystal chains link together to produce a strong framework that binds the aggregate particles together. More cement reacts with the leftover water during curing. The mixture used in the 3D printer is comparable to the mixes typically used but differs in composition. Traditional methods range widely in their use, from hand tools to heavy industry, but they all lead to concrete placement in a formwork where it cures into a final shape. No formwork or mold is used when printing concrete in three dimensions [85].

Numerous academics have suggested various mixed design processes for the 3D printing of concrete. To give the proper and diverse combinations of cementitious materials, numerous investigations have been conducted [86]. Industries and academic institutions all around the world are looking into this specific method to accredit various construction applications [87]. For instance, in the work of Ma et al. [88] every kind of aggregate and cement-like component was chosen based on their nozzle size's extrudability criteria. Supplement cement-like substances

(SCMs) and fibers were added to suit the demands of several qualities, including shrinkage, buildability, strength, and setting time.

Additionally, inconsistent performance demands for concrete include high fluidity balanced by superplasticizer. A similar idea was presented in the study by Malaeb et al. [89], arguing that the mixed design must achieve the objectives of new attributes. Furthermore, Figure 17 shows the proportion of components for 3D-printable concrete that Rehman and Kim [90] examined.

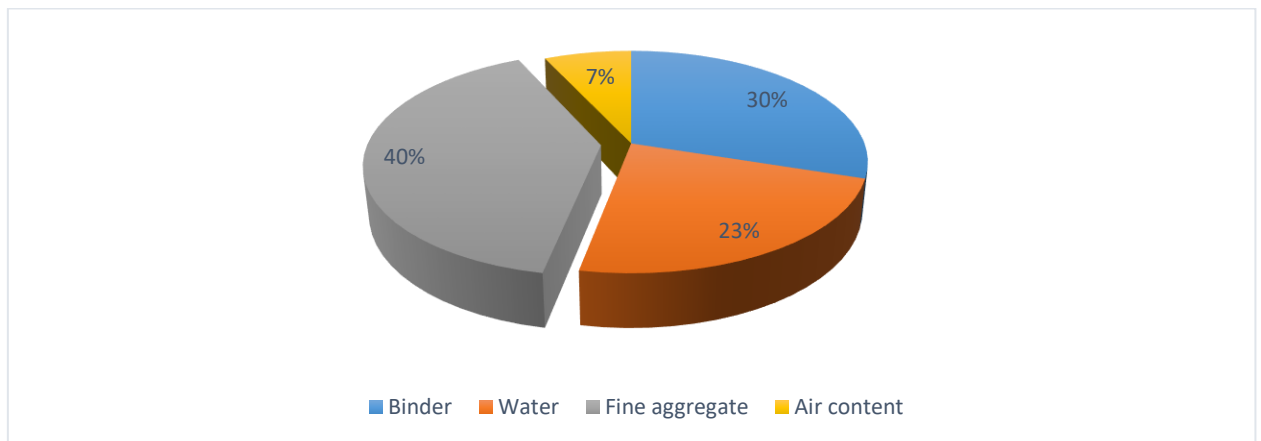


Figure 17: Composition of materials used in 3DPC by Rehman and Kim [90]

Because the concrete mix design affects the end product's structural performance, special consideration must be given whereas 3D printing cementitious concrete [86]. Since there is no formwork or standard, the right composition of the material represents one of the main points [91]. Additionally, the material needs to have its rheology properly regulated so that it can be pushed through an extruder and maintain its final form after being extruded out [92]. The conditions for 3DPC printability, particularly are directly related to the operations of the machine and the printer itself, must be met by mixed design technologies [93]. Fresh concrete or mortar is typically applied in layers, one on the highest point of the other [86]. The following problem is that the layer that was printed must support the subsequent ones [91]. According to Lužanin et al. [94] the complexity of creating a digital model must be paired with the importance of the material and printer.

2.4.5 Polymer-based materials

Significant progress in polymer print technology has been made recently, and several research domains are currently moving toward the production of planned objects [95]. Due to their low melt and cure temperatures, superior liquid or softened flow characteristics, and chemical stability, polymers are quite common [96]. Particle extrusion and polymer printing are made

possible by 3D printing techniques, which support designs with a variety of layouts, topologies, and responses [97].

It is important to note that polymers have not gained as much traction in the construction sector as cementing materials because they are mostly used with other materials for architectural or aesthetic reasons [98]. The shortage of mechanical strength and functioning of 3D-printed polymer items, despite their intricate geometric shapes, poses a substantial barrier to their widespread adoption. A viable solution to these problems is to combine various substances to accomplish the desirable mechanical and functional properties. As a result, recent years have seen a lot of interest in the development of composites that are compatible with existing printers [99]. Additionally, the majority of printed polymers are inappropriate for strongly loaded components due to their comparatively low strength and stiffness. However, AM has made use of polymer composites with enhanced mechanical properties, such as ABS with carbon fiber reinforcement [71, 100, 101].

Extrusion is the most significant and popular technique in polymer processing. It is frequently employed in the manufacture of films, sheets, pipes, and profiles in addition to in specific technological processes including compounding, mixing, and granulation. These processes are carried out on the majority of polymer materials, primarily through extrusion [102]. Extrusion machines carry out a variety of crucial tasks, including transporting polymers, melting metals, creating pressure, combining materials for chemical homogenization, and finally, producing products [102]. In polymer extrusion, the material which is formed is typically applied layer by layer. After being heated, the nozzle releases a filament-shaped polymer using a typical material extrusion setup [103]. Up till now, extensive research on an extrusion technique has been done in books and articles: Rauwendaa [104], Gogos and Tadmor [105], Chung [106], Agassant, et al. [107], Wilczyński [102].

Furthermore, a variety of AM techniques, such as FDM, SLA, SLS, and inkjet printing, can be used to create polymers [99]. For the production of polymer composites, particularly the most popular thermoplastics like PLA (Polylactic acid), PC (Polycarbonate), and ABS, fused deposition modeling (FDM) printers are widely utilized [99].

2.5 Geopolymer and it's potential as a building material

Understanding the mix proportions of geopolymers is the first and most important research topic according to the research questions. Large quantities of cement [108] and tied-up formwork result in significant resource waste and CO₂ emissions [109]. Therefore, coming up with a novel and creative construction strategy is encouraged with the purpose of achieving architectural, economic, and environmental advantages [86]. Technology for making cement is improving, but

using conventional raw materials, such as plain Portland Cement (OPC), has drawbacks. OPC is the fourth most important producer of CO₂ with an annual production of about 0.8 tons [110]. Utilizing geopolymers is an alternate method [111]. Fly ash and slag are two locally available materials that are used to create the material geopolymer, which has qualities that are superior to OPCs [112, 113].

The words "geopolymer," "inorganic polymers," "hydro ceramics," "alkali-bonded ceramics," and "alkali-activated types of cement" are also commonly used to refer to amorphous alkali aluminosilicates [114]. Despite the variety of that language, these terms describe a class of synthetic materials that share the same chemical [78].

The physicist Glukhovsky created the first structures made of alkali-activated slag in Ukraine in the 1950s. He identified the potential of binding synthesizers employing solutions of aluminosilicates and alkali metals and gave the concrete the name "soil silicates" as a result. In Mariupol, Ukraine, two nine-story residential complexes were constructed using this material in the 1960s [115]. Nevertheless at the previous center, the term "geopolymer" was first used in the late 1970s to describe a collection of source materials produced by interacting an alkaline solution with an aluminosilicate powder [111].

It is interesting that geopolymer research is currently seeing a resurgence as a result of intensified environmental issues and rising raw material costs. In order to avoid large-scale dumping and contribute to the solution of the issue of cement shortages, developing nations, in particular China and India, are seeking a way to recycle rising volumes of industrial waste [116]. For the production of geopolymer, secondary sources created from industrial waste can be used in place of primary raw materials. Therefore, growing industrial countries are very interested in developing geopolymers for widespread application [117].

2.5.1 Geopolymerization

Geopolymerization takes place while aluminosilicate materials dissolve in an alkali activator solution at either low or high temperatures. This results in an amorphous stage and a three-dimensional silicoaluminate network structure (Figure 18) [118] [119]. Even while specialists have different hypotheses regarding how reactions happen during geopolymerization, most concur that there are three main steps to the process [78, 118, 119]:

- The evaporation of aluminosilicate materials in the concentrated alkali solution leads to the creation of free silica and the alumina tetrahedron unit.
- The inorganic geopolymer materials gel stage is produced by material transfer, solidification, and condensation reaction involving alumina and silica hydroxyl. The structure is now releasing water as a consequence of the hydrolysis process.

- When the gel phase solidifies, a three-dimensional network of silicoaluminate condenses to form a geopolymer [120].

The structure of the initial mixture is essential for the precise use of 3D printing techniques with geopolymers. In addition to the aluminosilicate source and alkaline activators, it can be determined by accelerators, plasticizers, and other elements that provide geopolymers with the essential properties needed for 3D printing [24]. Table 4 provides a summary of some of the many studies that have been done so far to develop the optimum 3D printing method for geopolymers.

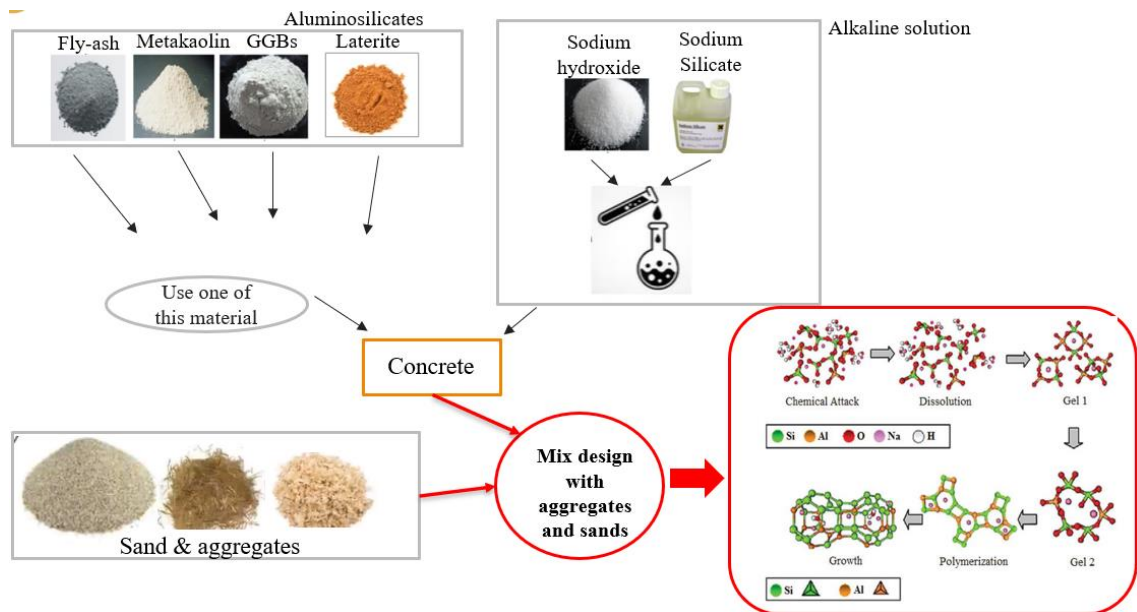


Figure 18: Geopolymerization [121] [122]

Table 4: Various technologies and components of geopolymers for 3D printing

References	3D printing machines		3D printable geopolymer components		
	Technique	Parameters of the printer	Aluminosilicate source	Activators	Aggregates
[123]	Material extrusion	Speed of the printer: 20 mm/s	fly ash+blast-furnace slag +silica fume	Na ₂ SiO ₃ ; NaOH	Sands with different grain sizes
[91]	Material extrusion	Diameter of the pipe: 25 mm; Speed of the gantry: 70 mm/s;	fly ash+blast-furnace slag +silica fume	K ₂ SiO ₃	Fine river sand
[15]	Material extrusion	Speed of the printer: 120 mm/s;	fly ash+blast-furnace slag +silica fume	KOH + K ₂ SiO ₃	River sand
[69]	Material extrusion	Dimension of the nozzle: 25 × 15 mm:	fly ash	NaOH, Na ₂ SiO ₃	Silica sand
[124]	Material extrusion	Diameter of the nozzle: 15 mm; Speed of the printer: 60 mm/s;	fly ash+blast-furnace slag +silica fume	penta sodium metasilicate	Silica sand
[125]	Material extrusion	Dimension of the nozzle: 30 × 15 mm:	Fly ash +blast-furnace slag	NaOH + Na ₂ SiO ₃	Fine and coarse quartz sand

2.5.2 Properties

Setting the properties for 3D geopolymers to be printed is a multi-parameter process heavily influenced by internal and external factors. Internal factors of source materials include the type and ratios of other components, such as the activator and precursors of aluminosilicate, as well as the printing process and curing conditions. The following subsections examine how these factors affect the printed geopolymers' new and hardened state properties (Figure 19).

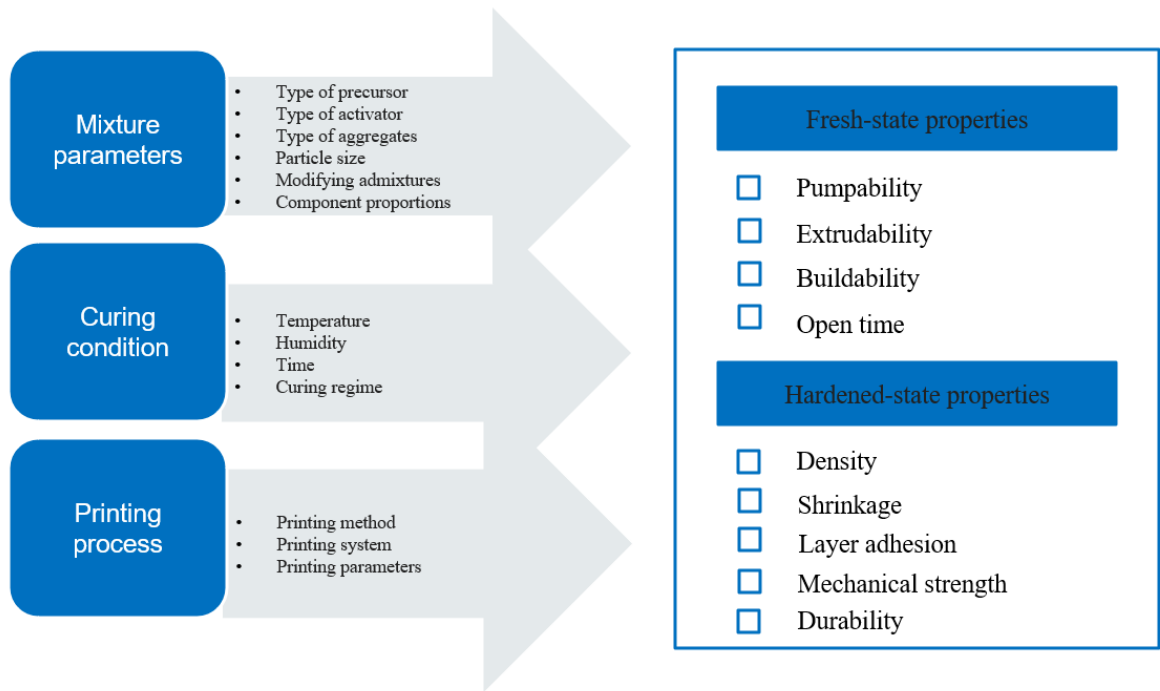


Figure 19: Factors affecting the properties of geopolymers suitable for 3D printing

2.5.3 Fresh-state properties

An essential quality of a new geopolymer combination is pumpability, describing the capabilities in terms of under pressure, change into an AM technology. The pumpability is related to rheological characteristics, mainly plastic viscosity and yield stress. High amounts of these characteristics typically result in declining pumpability, yet simultaneously, they get better buildability. Various organic and inorganic designing concrete mixtures can be utilized to control viscosity. However, their profusion may also negatively impact the ability to construct. Therefore, to obtain a high-quality geopolymer material, it is crucial to maintain a proper balance in both pumpability and buildability when setting rheological parameters. Internal variables like source material type and activator content significantly impact the viscosity of the geopolymer mixture [24].

Extrudability refers to a geopolymer mixture's ability to flow easily through the printing head's nozzle and produce a continuous filament [24]. A fresh geopolymer mixture's "open time" is the duration for which it has the right consistency for printing. The buildability of a fresh geopolymer mixture is a crucial component of the 3D printing process. It affects how well the laminated filament can maintain its shape without undergoing severe deformation or destruction. In addition, significant yield strength is required for the polymer mixture.

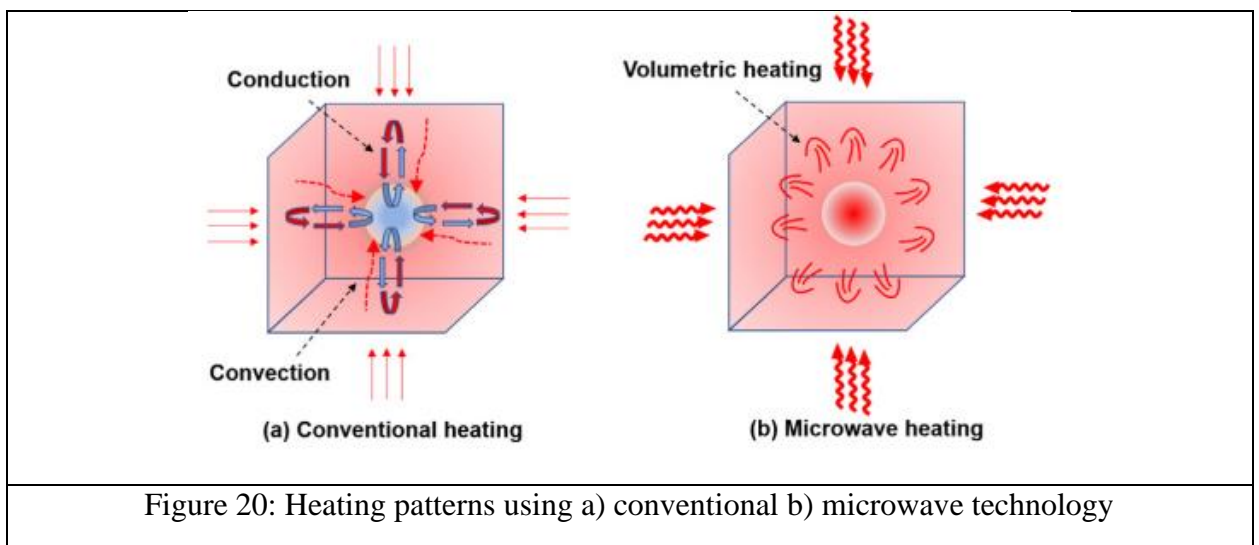
The mixing abilities of adjacent layers are crucial since their insufficiency might result in the creation of "cold seams". Microwave heating technology can increase the ability to create 3D-

printed geopolymers. The polycondensation effect among the filaments, which also increases the strength of the interlayer bond, is the reason for this effect.

2.6 Microwave heating process

This section's main objective is to summarize the state of microwave-curing method-based research and implementation in the geopolymer. A relatively recent field of study is the implementation of microwave heating to enhance the characteristics of geopolymer materials. Microwave heating offers several benefits, including quick speed for heat generation, high energy penetration, clean procedure, and precise electronic control; hence there is increased interest in this sector. Traditional heating techniques also have many drawbacks. For instance, it takes them a while to build up the strength needed for a water cure. Additionally, because of the non-uniform hydration products that result from concrete's natural thermal insulation, produced concrete can reach various temperatures under high-stream and temperature-heating conditions [126].

Microwaves are electromagnetic radiation with frequencies that vary from 300 MHz and 300 GHz that produce dielectric heating. In the chemical, food, and medical industries and among other processes, MW irradiation is often used. Microwaves are superior to conventional heating methods due to fast reaction, enhanced reaction, low heat loss, and excellent energy efficiency. In addition, traditional heating methods create a "skin effect", which consists of rapid water evaporation from the external surfaces of the processed products and is effective for surface heating (Figure 20a). Because of the high-temperature gradient, it is difficult to achieve uniform heating. Heated materials lose a significant amount of energy due to thermal conductivity and convection. Instead, microwave heating causes an increase in temperature due to heat absorption by polar molecules, creating localized heating points that do not depend on position and are, therefore, helpful in obtaining uniform volumetric heating (Figure 20b) [127].



It is challenging to understand how microwave solidification affects the properties and qualities of geopolymer products. Geopolymerization, another name for creating geopolymers,

also comprises the steps of dissolution, speciation equilibrium, gelation, reorganization, polymerization, and hardening [128].

2.7 Research Gap Analysis

Despite the extensive research conducted on the topic of an extrusion system for 3D-printed geopolymers, there are still some areas that have not been fully explored. Consequently, the following research gap has to be filled:

- The insufficient investigation of the characteristics of geopolymers could lead to economic and architectural advantages, as well as their ability to provide environmentally-friendly benefits.
- There is a lack of mixture design for 3D printed geopolymer materials using locally sourced raw materials.
- There is a need for research that specifically focused on appropriate printing materials for the extrusion-based method of AM.
- The absence of specific standards for the production of geopolymers for 3D printing

Chapter 3 – Research Methodology

3.1 Introduction

AM technologies have revolutionized the field of building engineering by providing a novel approach to building structures. The operating of AM technology in construction is an emerging field, and it has the potential to offer several benefits, including increased speed of construction, reduced costs, and enhanced sustainability. However, a thorough understanding of the materials used, the printing methods, and the end products' qualities are necessary for the successful application of this sort of procedure in construction.

A detailed comprehension of the materials, and the printing process is necessary for the introduction of 3D printing techniques in the building industry. The current study intends to investigate how geopolymer materials might be employed in construction using 3D printing techniques and to propose a method for producing such structures. The stages taken in conducting this study are detailed in depth in this chapter, and the findings will help create a system for printing geopolymer materials in the field of building engineering.

3.2 The Adopted Research Methodology

In this chapter, the methodology for this investigation is thoroughly explained, and Figure 21 includes the relevant stages. The material has been created for use in 3D printing by implementing the procedures listed below.

- 1. Data collection:** A thorough examination of the literature on 3D printing in building engineering, the essential properties of geopolymer materials, and key parameters of 3D printing machines have been conducted.
- 2. Geopolymer Mixture Preparation:** The appropriate materials and determine their precise chemical composition have been selected. The design for three different types of mix and perform various testing methods was investigated. The most suitable geopolymers will be thoroughly tested, and the values will be measured according to the standards employed to assess their appropriateness for 3D printing.
- 3. Molding and Demolding:** The mold was printed for this study using an Interprint I3 V2 3D printer. Rhino® and Grasshopper® are used to generate toolpaths for this study.
- 4. Microwave solidification:** The casted geopolymer and extruded layers are transferred to a domestic microwave oven and heated for a certain period.
- 5. Results Comparison and Validation:** The compressive and flexural strengths of casted and 3D-printed geopolymers are contrasted.

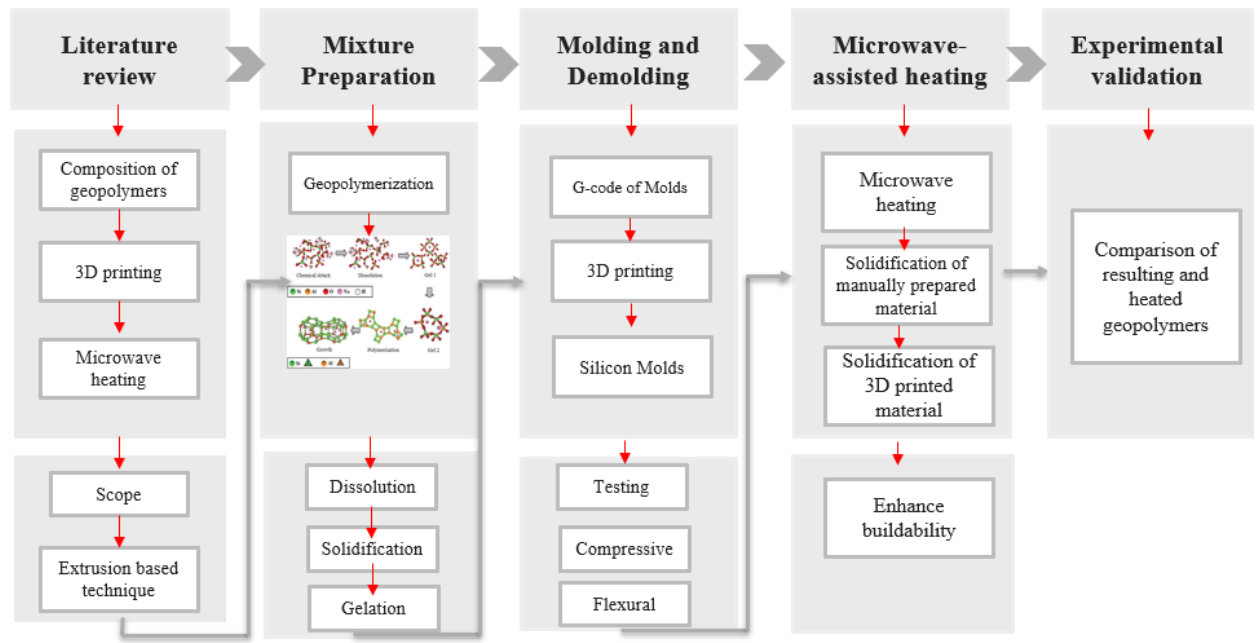


Figure 21: The Research Methodology Stages

Various materials and technologies are used to create and test geopolymer material. The materials used in creating geopolymers for 3D printing and the methods used in testing and evaluating new materials are established on a systematic methodology that verifies the influence of each ingredient in the material.

3.2 Geopolymer Mixture Preparation

This study used raw materials from nearby power plants, which are industrial by-products with a limited end user. The development of geopolymer for 3-D printing purposes uses fly ash, metakaolin, sand, and ground granulated blast-furnace slag (GGBS). The mixture uses sodium silicate solution as the reagent. The geopolymer binder formulation requires materials of FA class F from the Ekibastuz Power Plant and bottom FA class F from the Karagandy Power Plant. Aluminum and silicon are obtained from metakaolin. The literature analysis revealed that river sand is typically used as an infill for 3D printable geopolymers. Table 5 provides a summary of the key resources used in this research study.

Table 5: List of raw materials

Raw materials	Minimum, %	Maximum, %
Fly Ash class F	60	96.7
GGBS	1.67	30
Metakaolin	1.67	30
River sand	20	70
Sodium hydroxide	2.9	5
Sodium silicate	25.9	35

Numerous mixture configurations are possible using the usual precursors for geopolymers and activators. Data on the geopolymer compositions for extrusion AM used in various experiments were acquired to establish the most suitable combinations. Particularly interesting were the ratios of silicon dioxide (SiO_2) used in mixes. The most common 3DPG binder type is a combination of low calcium FA, GGBS, and silica fume. These three major ingredients are utilized to create printable geopolymers (SF). In this work, X-ray fluorescence (XRF) (Figure 22) was operated to analyze the utilized components and pinpoint their specific chemical composition before creating the mix design. Table 6 lists the chemical components of the materials in the form in which they were employed in this study.



Figure 22: XRF Axious Max [129]

Table 6: XRF data on the chemical components of FA and GGBFS (%)

Compound	FA (%)	GGBFS (%)
SiO ₂	56.247	36.142
Al ₂ O ₃	22.428	10.081
CaO	1.843	35.430
Na ₂ O	0.266	0.371
MgO	0.259	9.296
SO ₃	0.214	2.817
Cl	0.000	0.008
K ₂ O	0.529	0.783
TiO ₂	0.996	1.251
Cr ₂ O ₃	0.000	0.021
MnO	0.058	0.306
Fe ₂ O ₃	3.238	0.342
Co ₃ O ₄	1.241	0.000
CuO	0.295	0.000
ZrO ₂	0.025	0.025
BaO	0.098	0.000
CeO ₂	0.000	0.054

Two different kinds of metakaolin were used in this study, one from Austria (Metaver I) and the other from Kokshetau. The chemical composition of Metaver I has illustrated in Table 7.

Table 7: Chemical composition of Metaver I

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O ₃
51-56	40-44	<2.0	<2.0

To prepare another metakaolin, kaolin from Kokshetau was used. When using the furnace, as shown in Figure 23, kaolin was fired until the temperature reached 750 ° C, then kept at the same temperature for 2 hours, after which it solidified in the furnace for 12 hours.



Figure 23: Furnace for kaolin

Immediately after the kaolin was extracted from the furnace, a ball mill was used for grinding, as shown in Figure 24.



Figure 24: Balling mill for metakaolin

Testing was conducted on the two main materials that were determined to be the most effective for 3D printing applications. The mixing proportions of geopolymers by using metakaolin Metaver I are given in Table 8. The mixture's initial ingredients were assessed with a precision of 0.1 grams. Before starting to mix all the materials, the sodium silicate was prepared in advance for up to 24 hours by adding mixing sodium hydroxide to the sodium silicate solution. Then, sodium silicate was poured into metakaolin and stirred for 5 minutes at a speed of 150-200 rpm. GGBFS was added and mixed at the same speed for exactly 3 minutes. Finally, river sand was introduced and mixed at a speed of 100 to 150 rpm for an additional 5 minutes. The second design was prepared using the same method as the previous one, but before adding GGBFS, ash was added and mixed for 10 minutes.

Table 8: Proportions of the mixes with Metaver I

Materials	Mix proportions (gr)	
	G1	G2
Metakaolin (Metaver)	64.88	47.2
FA class F	-	184.8
Bottom ash class F	-	-
Sodium Silicate	100	140
GGBS	20	20
Sand	200	130
Water	3.8	10

The same procedure was done for metakaolin from Kokshetau. The mixing proportions are demonstrated in Table 9.

Table 9: Proportions of the mixes with metakaolin from Kokshetau

Material	Mix proportions (gr)	
	G1	G2
Metakaolin (Kokshetau)	106.49	102.54
FA class F	-	184.4
Bottom ash class F	-	-
Sodium Silicate	100	140
GGBS	20	20
Sand	200	130
Water	3.8	10

3.3 Molding and Demolding

When the material has been cured for 1, 3, and 7 days, testing has been done for both the fresh and the hardened conditions. The test procedures mentioned in this research study are categorized in Table 10.

Table 10. Test procedures for 3D printing geopolymers materials

Testing	Preparation	Specimens	Quantity	Measurements
Compression	1, 3,7-day hardened samples	50x50x50 mm cube	3 samples for each day	Loaded til fracture
Flexural	1, 3,7-day hardened samples	40x40x160 mm	3 samples for each day	Loaded til fracture
Setting time	1,2-hours hardened samples	70x70x70 mm	1 sample	

Compressive and flexural strength tests were carried out using the machine shown in Figure 25. Compression tests were conducted under ASTM 349, the hardened concrete compressive strength test standard procedure. 50x50x50 mm molds (Figure 26a) were operated and filled with the material. Samples were set up for 3, 7, and 28 days of room-temperature drying.

Testing for flexural strength was done following ASTM C293/C293M. This test method involved the use of a straight beam with a load at a central point to identify the flexural strength of geopolymers samples. The dimensions of the beam molds are 40x40x160 mm (Figure 26b).



Figure 25: Compression and flexural testing machine [130]



a)



b)

Figure 26: Test specimens for a) compressive; b) flexural testing

Evaluating the mechanical properties of geopolymer materials is a critical step in assessing their suitability for various applications. In this context, the setting time is an important parameter to measure, as it indicates the time required for the geopolymer to achieve its maximum strength.

By measuring the penetrating resistance of mortar that has been sieved from the geopolymer mixture, the setting time method allows for the estimation of the time at which geopolymer with a slump larger than zero will set. Geopolymer mortar is created by starting with the creation of an alkaline solution about a specified ratio. Dry mixtures of fine aggregate and fly ash were then fully blended with the addition of an alkaline activator. Once the liquid was poured, the setting time was measured at room temperature until the initial and final setting times had been established. The setting time of the geopolymer was determined using the Resistance Apparatus, as shown in Figure 27, which measures the penetration of a needle into the geopolymer paste over time. Penetrometer testing following ASTM C403 was implemented for this sort of testing. According to the penetration resistance, the initial setting period's pressure should be 3.5 MPa, and the final setting time should be 27.6 MPa [131].



Figure 27: Penetration Resistance Apparatus




The mixtures of numerically most suitable geopolymers are thoroughly tested, and the values are measured according to the standards employed to identify the suitability of the material for 3D machines. Mixtures of geopolymer materials are selected and evaluated using numerical values for further testing.

Essential parameters of newly printed geopolymers are extrudability, shape retention, and buildability. In concrete printing, extrudability describes a substance's ability to pump freely inside an extruder without clogging or restricting the pipe flow. Due to the association between these phenomena and the freshness attribute of the material, numerous research studies have detailed it concerning well-known Bingham variables and other flowing features. Although several standard methods, including the flow table test and the drop test [132], were employed in the past to measure

flow characteristics, in this work the extrudability of an independently created geopolymer mortar intended for 3D printing purposes was investigated using data from a rheometer. Shape retention is another essential component of 3D printing. The material after extrusion should maintain its shape following the extruder measurements.

Buildability is a difficult problem in 3D printing, and to solve it, the newly formed material should return to its pre-extrusion viscosity and yield stress levels before the subsequent layer begins to collapse upon it. The buildability of a combination of geopolymers depends on the set speed, the interval between extrusions, and the ability to bind the layers of the thread together. An organized process flow of the methods mentioned for 3D printing material in a laboratory is shown in Figure 28 and the main characteristics of the printer are illustrated in Table 11.

Table 11: 3D printer characteristics [133]

<p>Concrete 3D printer</p> 	Build volume, WxDxH, cm	100X190x180
<p>Mixing print head</p> 	Nozzle speed, mm/sec	200
<p>Ram extruder</p> 	Ram extruder volume, l	5
	Feed rate, l/min	up to 1

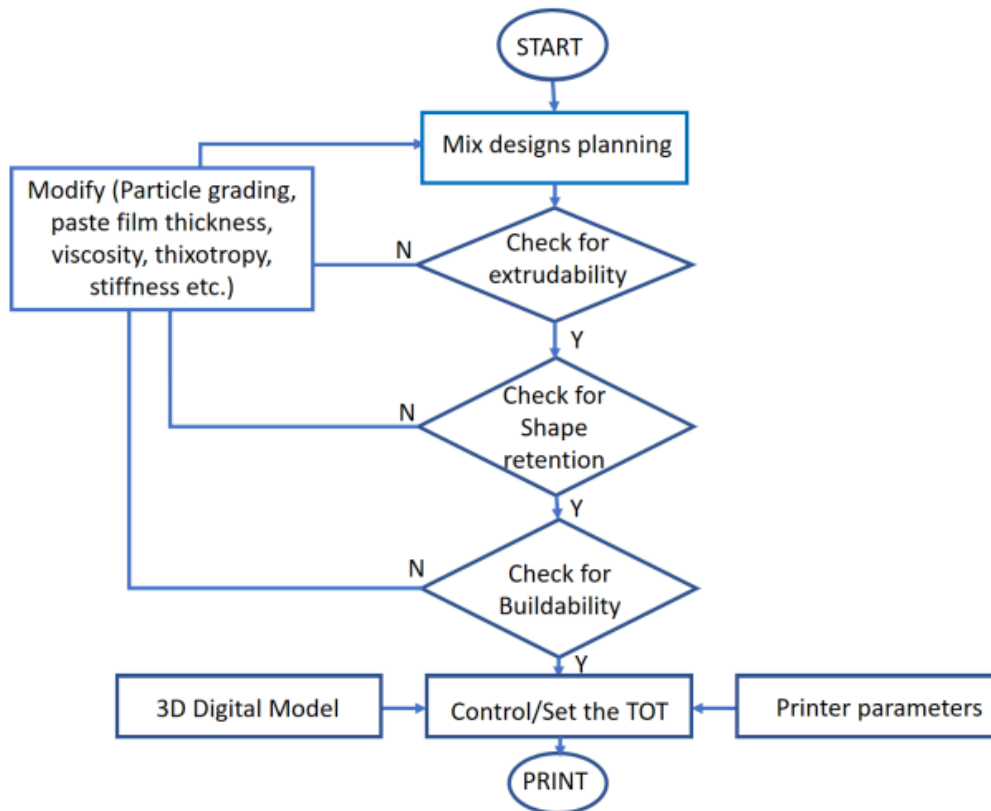


Figure 28: Flow chart of a laboratory experiment to test a geopolymer

3.4 Microwave-assisted Heating

A new technique has been proposed that makes use of a domestic microwave oven to assess the mechanical characteristics of 3D-printed items made of geopolymer. Testing was constructed using Rhino/Grasshopper software and fabricated using an Interprint I3 V2 3D printer (as depicted in Figure 29). To obtain results for microwave heating, 3D models of the matrix for compressive (50 x 50 x 50), as shown in Figure 30) and flexural (40 x 40 x 160), as shown in Figure 32. The main characteristic of the printed demonstrated in Table 12, the fabricated matrices were filled with silicon to obtain the desired molds. The final mold for compressive strength testing is presented in Figure 31, while the mold for flexural strength testing is illustrated in Figure 33.

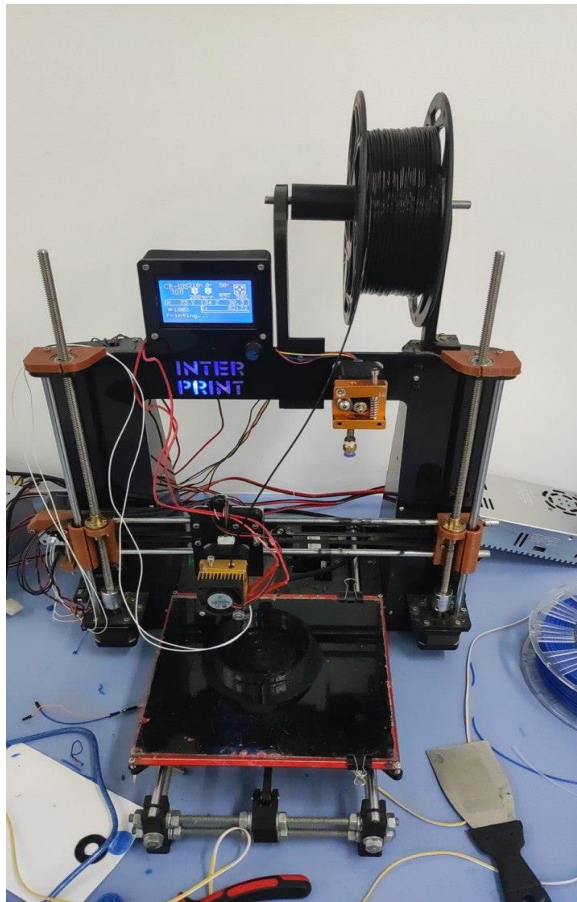


Figure 29: 3D printer used for matrix printing (Interprint I3 V2)

Table 12: The characteristics of the printer [134]

Working area	200*200*200
Nozzle	0.4 mm
Movement speed:	100 mm/s
Print speed:	80 mm/s
Printing material:	PLA, ABS, FLEX

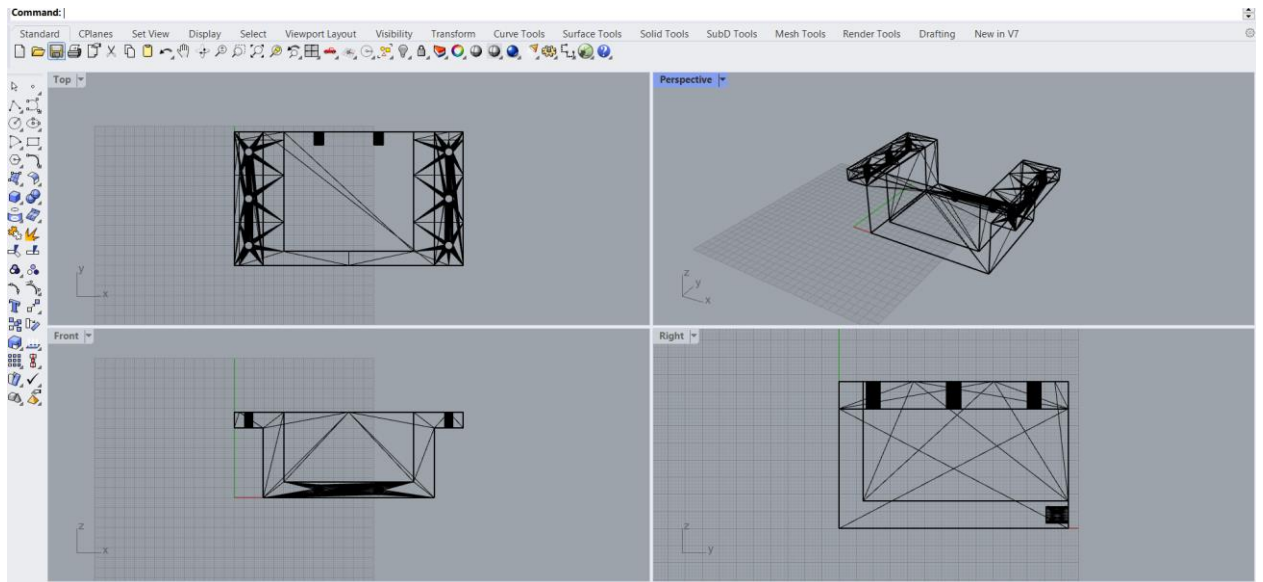


Figure 30: Model of the mold matrix for compression testing

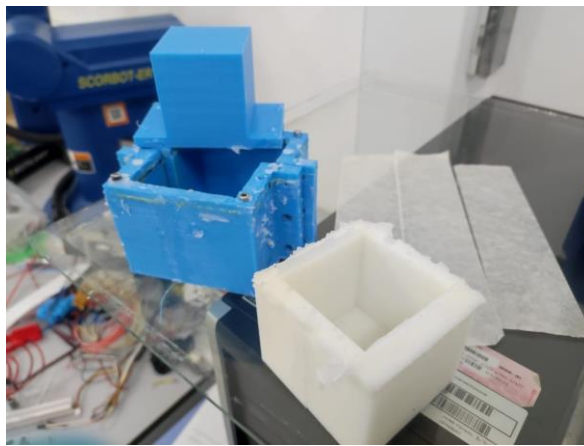


Figure 31: Printed matrix and silicone mold

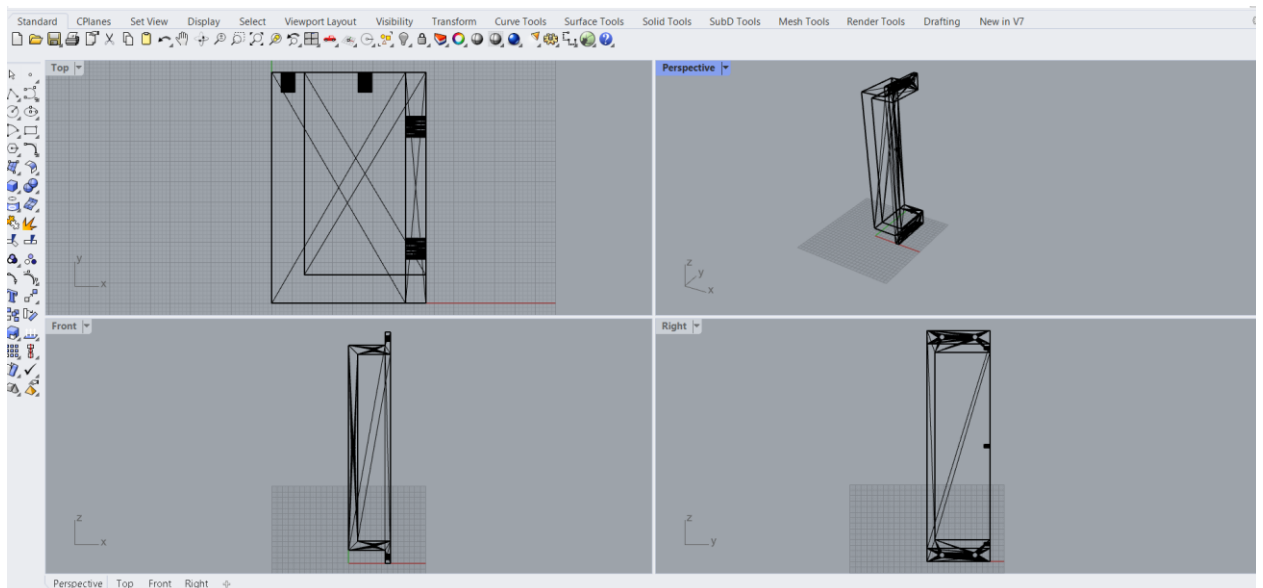


Figure 32: Model of the mold matrix for flexural testing

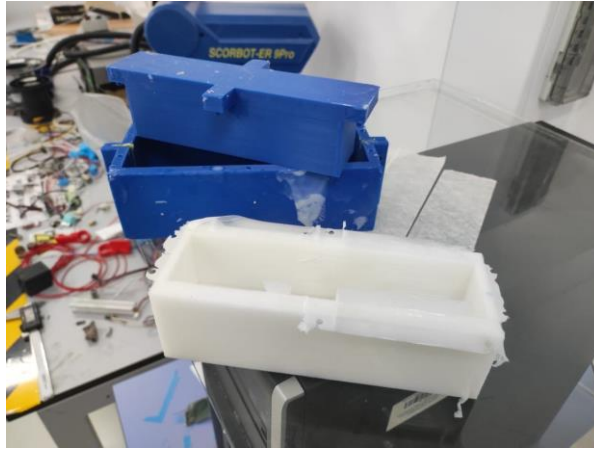


Figure 33: Printed matrix and silicone mold

The geopolymer is transferred to the oven and heated for a certain period, and their flexural and compressive strengths are assessed. It is worth highlighting that for all samples, the cycle time—the amount of time among the application for every manufactured geopolymer – was 10, 20, 30, and 40 seconds. According to the work done by Sun et al. [127] to 40 seconds, 600 W, 700 W, and 800 W are the most suitable installations for analyzing the fly ash-based geopolymer. Therefore, the power of 600 watts, 700 watts, and 800 watts was chosen for this experimental part. It ensures consistency in the printing process and allows for direct comparison of the prepared objects. The employing of a microwave oven for heating the geopolymer layers offers several advantages over traditional heating methods, such as convection or radiant heating. Microwave heating is a fast and efficient method of heating, which can reduce processing time and energy consumption. It also allows for precise control of the heating process, which is essential for maintaining the structural integrity of the printed objects. In addition, microwave heating can be easily scaled up for industrial purposes, making it a promising technology for large-scale AM technology.

The proposed method offers a simple and efficient way of evaluating the mechanical characteristics of 3D printing for geopolymers. The samples are heated using a household microwave oven, which is a quick and effective technique to simulate printing conditions. The results are encouraging for the potential of geopolymer composites for 3D printing purposes.

3.5 Results Comparison and Validation

In addition to investigating the properties of fresh 3D printing geopolymer materials, there has been a remarkable focus on testing the hardened characteristics of these materials. The primary focus of hardened property testing has been on compressive, and flexural testing. Compressive strength testing assesses the material's resistance to crushing forces, while flexural strength testing evaluates the material's capacity to withstand bending forces.

The findings of these studies are not only valuable in understanding the material properties of 3D printing geopolymers but also in comparing the results with similar investigations conducted by other researchers. Overall, the investigation of both fresh and hardened 3D printing geopolymer materials is critical in advancing the area of 3D printing and expanding its implementations in various industries. The findings of these studies contribute to a deeper understanding of the material properties of 3D printing with geopolymer, which can aid in the improvement of the printing method and the creation of novel applications.

3.6 Summary

The methodology for a research study on the creation of 3D-printed geopolymer material is described in this chapter. The study used raw materials from nearby power plants, which are industrial by-products with limited end-users. The main materials implemented in this research study are fly ash, metakaolin, sand, and GGBS.

The study consists of five main stages: data collection, material preparation, 3D printing of the material, microwave solidification, and results from comparison and validation. The first stage involved a thorough examination of the literature on AM techniques in construction engineering, the essential properties of geopolymer materials, and key parameters of 3D printing machines. In the material preparation stage, the appropriate materials were selected, and their precise chemical composition was determined.

Various mixture configurations were tested for their suitability for 3D printing, and several tests were conducted according to ASTM standards. The third stage involved 3D printing of the material using a cavity pump on a 3D-printing machine, and Rhino® and Grasshopper® were used to calculate and generate toolpaths for the study. The fourth stage involved microwave solidification, while the fifth stage involved comparing and validating the outcomes of casted and 3D-printed geopolymers in terms of mechanical strength and setting time,

CHAPTER 4 – Experimental Results

4.1 Introduction

The created geopolymers were put to the test using the test techniques covered in the methodologies section for 3D printing applications. Each specimen in each test was tested in the same way. In this chapter, the collected data are visually displayed together with estimated average values and dispersion. For each variation in each test, measurements were taken at least three times. The same graphs' average values were computed and displayed. Error bars were set up to compare the overall average to the highest and lowest recorded values.

In general, four types of geopolymer were created, and a brief description of the basic composition and the name of each geopolymer is shown in Table 13.

Table 13: Designations of created geopolymers

Main components	Name
Metaver I metakaolin-based geopolymer	A
Fly ash-based geopolymer with Metaver I	B
Kokshetau's metakaolin-based geopolymer	C
Fly ash-based geopolymer with Kokshetau's metakaolin	D

4.2 Compressive strength results

The compressive strength of geopolymer over a period of seven days has been assessed mechanically using a variety of experiments. The geopolymer specimens were cured for one day, three days, and seven days and the compressive strength was measured using appropriate instruments. Figure 34 and Figure 35 display the compression test results.

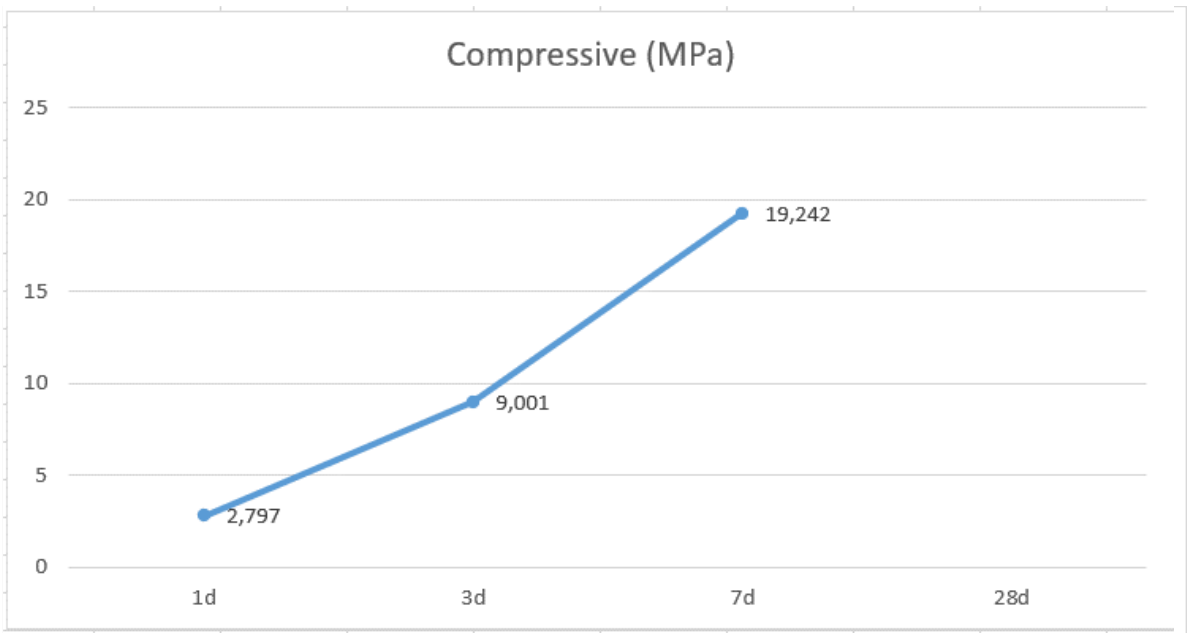


Figure 34: Compression strength of geopolymer A after 1, 3, and 7 days of drying

After one day of drying, the compressive strength was found to be 2.797 MPa, indicating a relatively low strength. However, after three days this value significantly increased to 9.001 MPa, suggesting that the geopolymer was gaining strength over time. Finally, at the end of seven days of curing, this number reached 19.242 MPa, indicating a remarkable increase over the preliminary findings. This data highlights the importance of extended curing periods in enhancing the mechanical properties of geopolymer, which is critical for its successful application in construction and other industries.

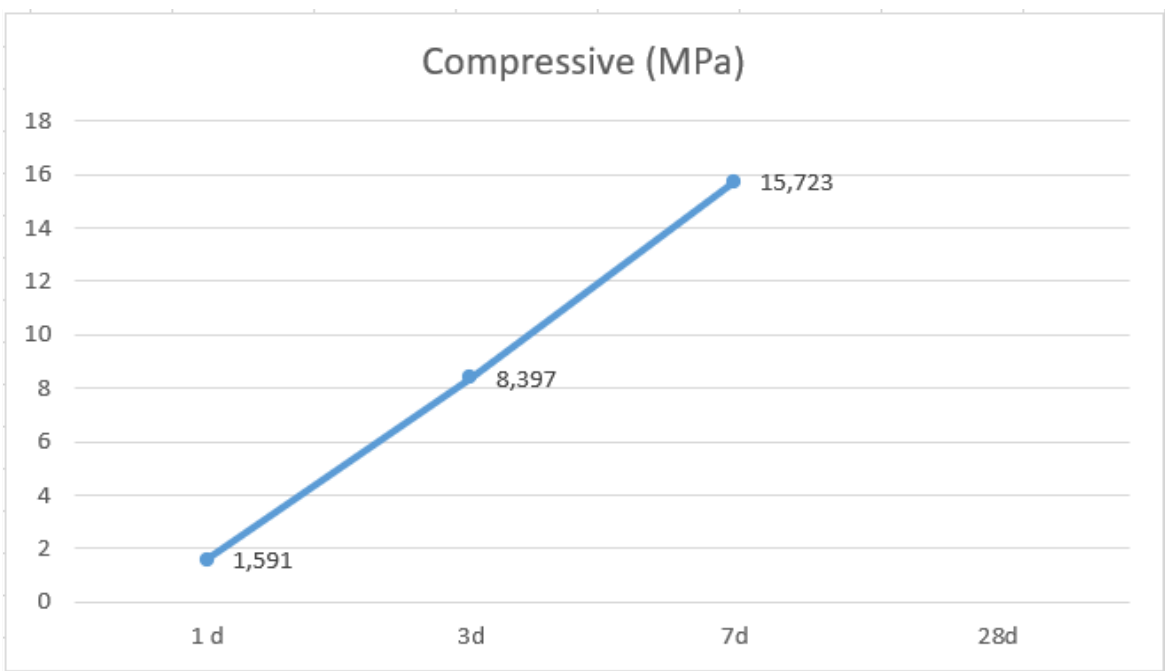


Figure 35: Compression strength of geopolymer B after 1, 3, and 7 days of curing

The compressive strength of specimen B was 1.591 MPa following one day of drying. However, as the curing process continued, this value improved significantly. For example, by the third day of curing, the compressive strength increased to 8,397 MPa. This tendency to increase strength persisted during the curing period, and by the seventh day, the compressive strength reached 15.723 MPa.

The compressive strength of geopolymers containing metakaolin (Kokshetau) and Fly Ash was evaluated after 1, 3, and 7 days of ambient temperature curing. Figure 36 and Figure 37 display the compression test results.

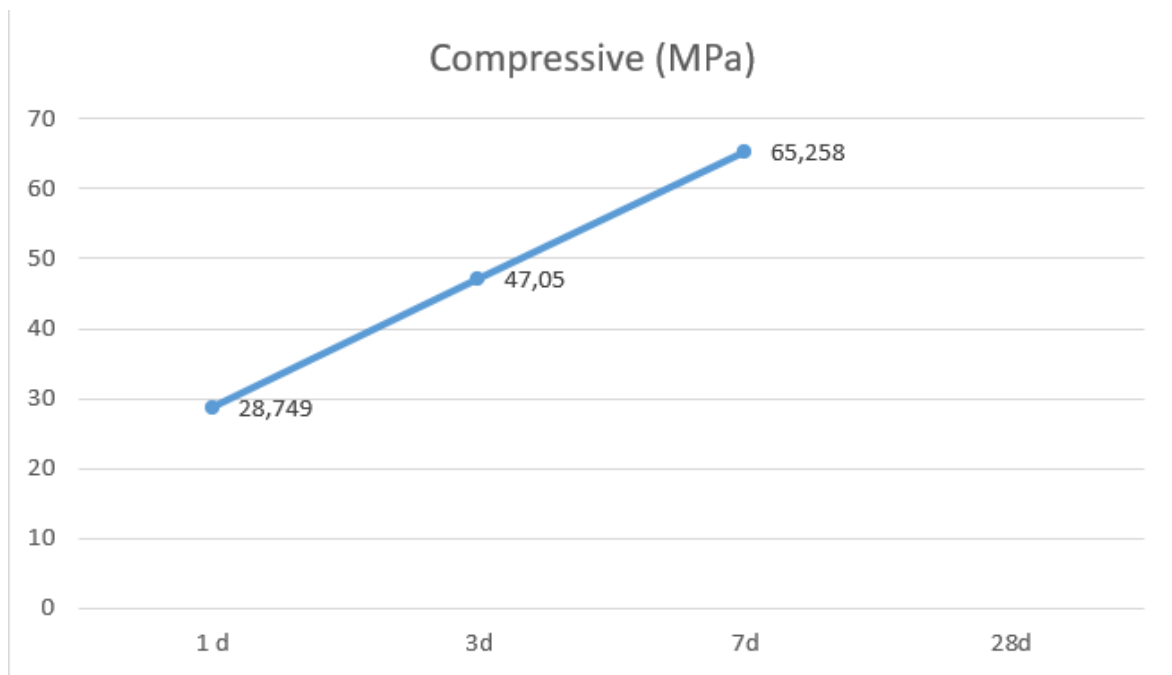


Figure 36: Compression strength of geopolymer C after 1, 3, and 7 days of curing

The compressive strength of geopolymer C was 28.749 MPa following one day of drying. This value increased to 47.05 MPa after three days. Finally, after 7 days of curing, this value changed and reached 65.258 MPa. These findings demonstrate that the material becomes increasingly more resilient to compressive stress as it cures since they significantly increase compressive strength with rising curing time.

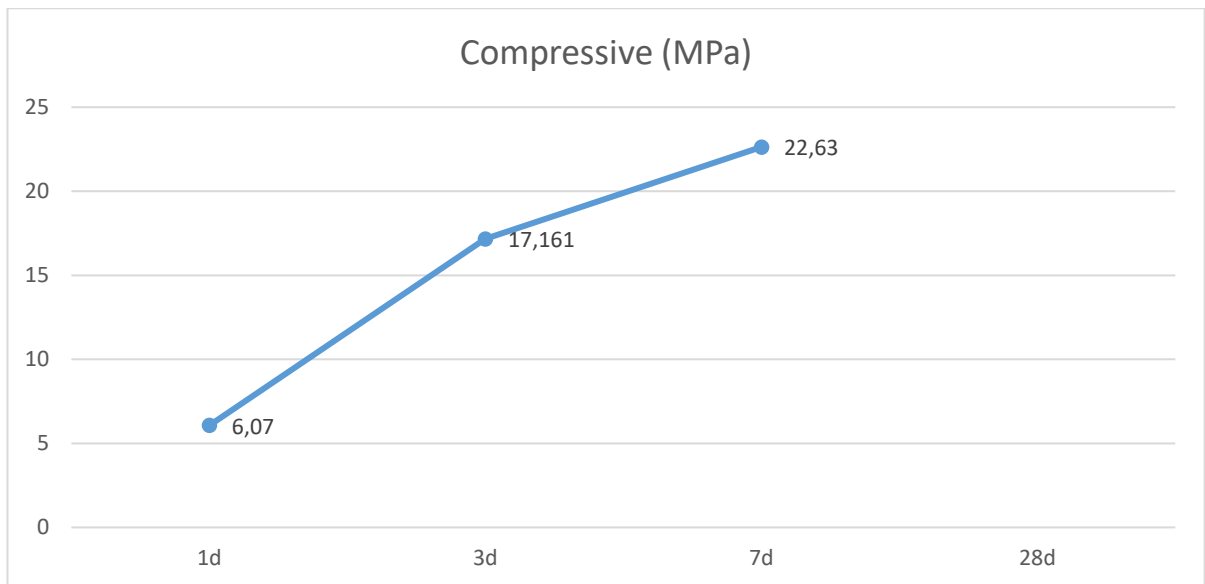


Figure 37: Compression strength of geopolymer D after 1, 3, and 7 days of curing

After one day of curing, the studies revealed an initial compressive strength of 6.07 MPa. However, this value increased significantly to 17.161 MPa on day 3, demonstrating a noticeable improvement. Furthermore, after 7 days of curing, the compressive strength reached a value of 22.63 MPa, which indicates that the geopolymer has achieved a relatively high degree of mechanical characteristics.

With cast specimens, every specimen is fractured in the same way. The compression-exposed sides on all four were equally cracked, which is a successful failure. Figure 38 depicts a few tested samples.



a)



b)

Figure 38: Some of the samples after compression testing: a) geopolymer A b) geopolymer B

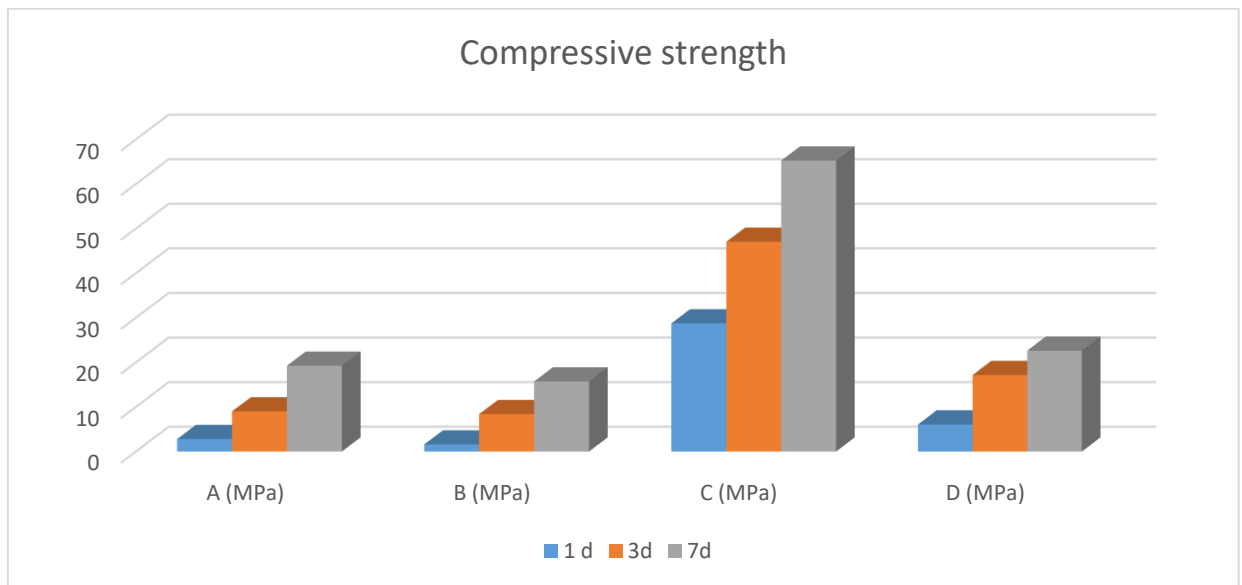


Figure 39: Comparison of compressive strength

The compressive strength outcomes for four different geopolymers were evaluated at different curing times (Figure 39). Geopolymer A exhibited a low initial compressive strength of 2.797 MPa after one day of curing, which significantly increased to 9.001 MPa after three days and finally reached 19.242 MPa after seven days of drying. Geopolymer B provided an initial compressive strength of 1.591 MPa after one day of curing, which increased significantly to 8,397

MPa after three days and reached 15.723 MPa after seven days. Geopolymer C exhibited the highest compressive strength values at all curing times. Finally, geopolymer D exhibited an initial strength of 6.07 MPa, which significantly increased to 17.161 MPa after three days and reached 22.63 MPa after seven days.

Comparing the compressive strength values for each day of curing, it is evident that geopolymer C exhibited the highest values at all curing times, followed by geopolymer D, geopolymer A, and geopolymer B. Geopolymer C showed the highest initial strength of 28.749 MPa, which increased to 65.258 MPa after seven days of curing, indicating its excellent mechanical properties. Although geopolymer A and geopolymer B also showed improvements in strength over time, their initial compressive strength values were relatively low compared to geopolymer C and D.

The results indicate that geopolymer C exhibited the best mechanical properties among the tested geopolymers, with the highest results at all curing times. Geopolymer D also showed promising results, with significant improvements in strength over time.

4.3 Flexural strength results

The same procedure was done for flexural strength tests for casted samples. The results for 1, 3, and 7 days for geopolymers are illustrated in Figure 40-43 and summarized in Table 14-17.

The examination of the flexural strength of a geopolymer material involves testing its resistance to bending under a specific load. The results of such tests provide insights into the behavior of the material over time as it undergoes curing.

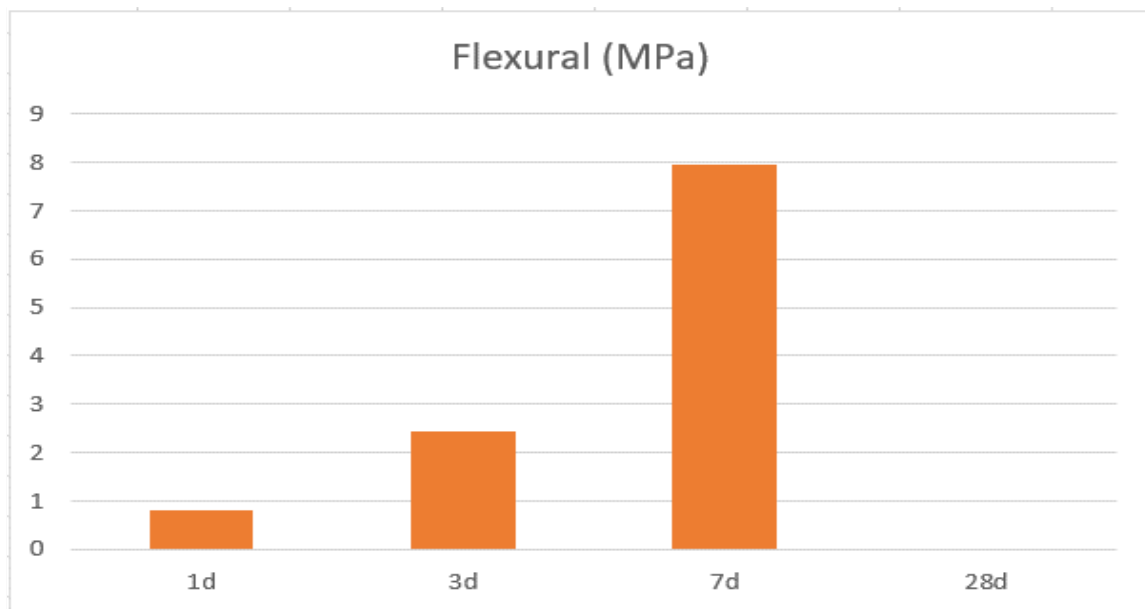


Figure 40: Flexural Strength of geopolymer A after 1, 3, and 7 days of curing

The results indicated that the flexural strength increased significantly with curing time. Specifically, after 1 day of curing, the flexural strength was measured at 0.817 MPa, which increased to 2.435 MPa on the 3rd day and further increased to 7.953 MPa on the 7th day.

Table 14: Outcomes of flexural strength of geopolymer A

Age	Flexural (MPa)
1d	0,817
3d	2,435
7d	7,953

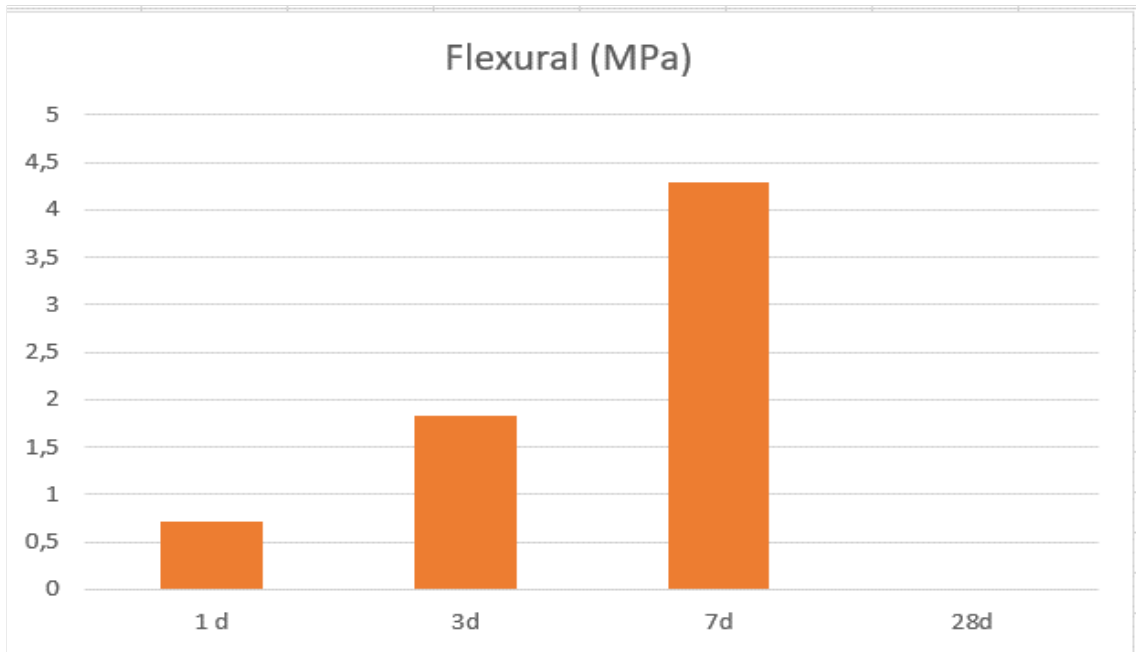


Figure 41: Flexural Strength of geopolymer B after 1, 3, and 7 days of curing

In the geopolymer B, the flexural strength increased from 0.707 MPa at 1 day to 1.83 MPa at 3 days. Further increases were observed at 7 days, with a flexural strength of 4.293 MPa recorded.

Table 15: Outcomes of flexural strength of geopolymer B

Age	Flexural (MPa)
1d	0,707
3d	1,83
7d	4,293

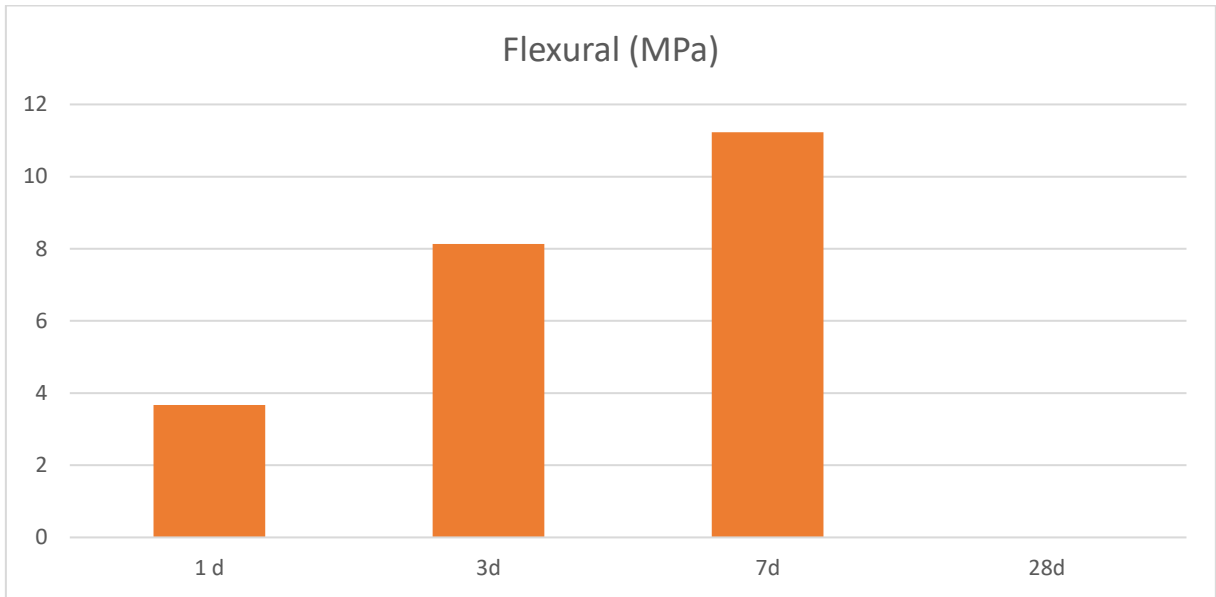


Figure 42: Flexural Strength of geopolymer C after 1, 3, and 7 days of curing

After 1 day of curing, the flexural strength was measured as 3.67 MPa. This value increased to 8.135 MPa and further reached 11.02 MPa after 7 days of drying. These results demonstrate the importance of proper curing procedures in the development of the material's mechanical properties.

Table 16: Outcomes of flexural strength of geopolymer C

Age	Flexural (MPa)
1d	3,67
3d	8,135
7d	11,02

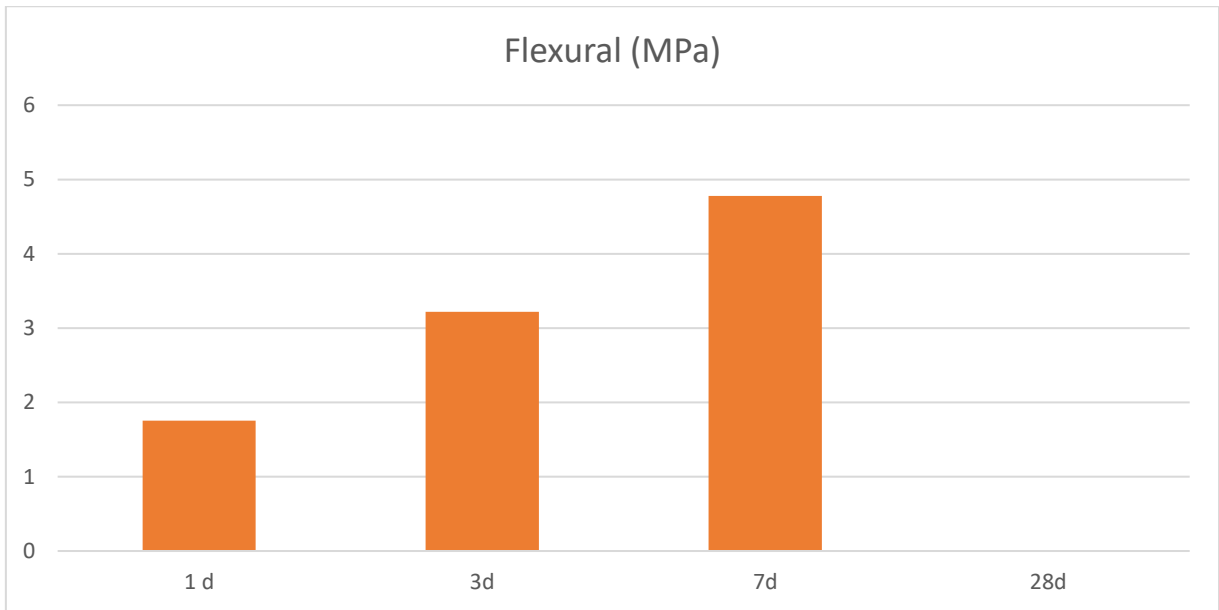


Figure 43: Flexural Strength of geopolymer D after 1, 3, and 7 days of curing

The findings of the geopolymer D showed that the material's flexural strength increased significantly from 1.753 MPa on the first day of curing to 4.78 MPa on the seventh day.

Table 17: Outcomes of flexural strength of geopolymer D

Age	Flexural (MPa)
1d	1,753
3d	3,218
7d	4,78

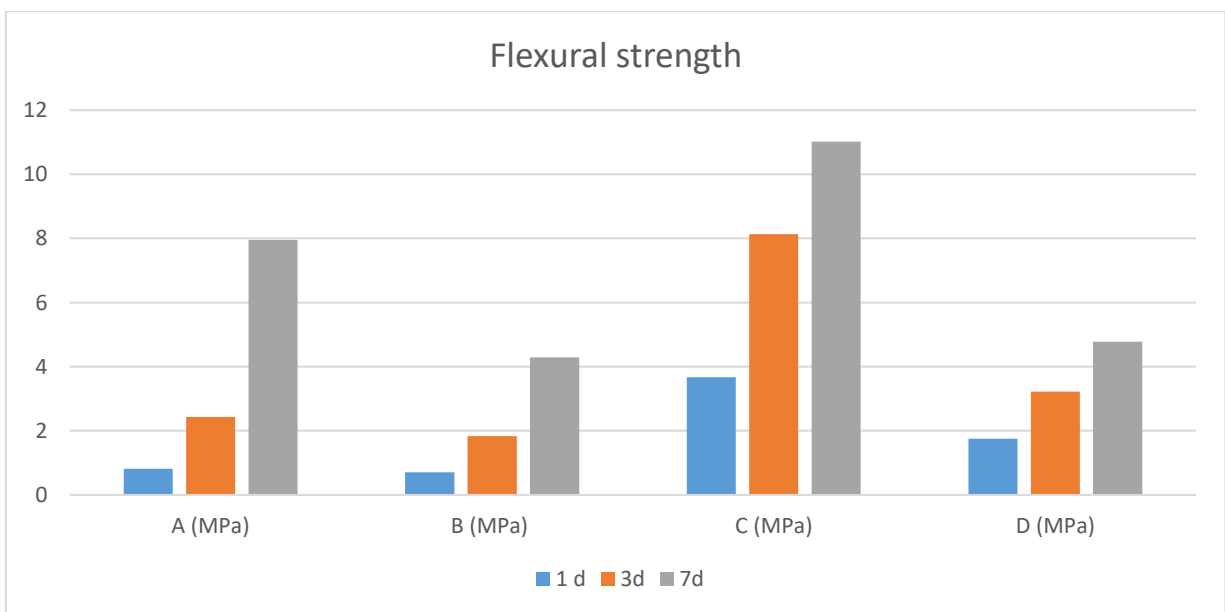


Figure 44: Comparison of Flexural strength

To compare the mechanical characteristics of the four different geopolymer samples, designated A, B, C, and D, the results of the flexural strength tests were examined (Figure 39). The results showed that as curing time rose, each geopolymer's flexural strength grew dramatically.

For Geopolymer A, the value increased from 0.817 MPa on the first day of curing to 2.435 MPa on the third day and further to 7.953 MPa on the seventh day. Geopolymer B exhibited an increase in flexural strength from 0.707 MPa on the first day to 1.83 MPa on the third day and finally to 4.293 MPa on the seventh day. Geopolymer C showed a similar trend, with the flexural strength increasing from 3.67 MPa on the first day to 8.135 MPa on the third day and further to 11.02 MPa on the seventh day. Geopolymer D exhibited an increase in flexural strength from 1.753 MPa on the first day to 4.78 MPa on the seventh day.

Comparing the results, it is evident that Geopolymer C provided the highest values among all the geopolymer samples, followed closely by Geopolymer D. The results of Geopolymer A and B showed a relatively lower increase in flexural strength compared to Geopolymers C and D.

The findings demonstrate the importance of proper curing procedures in the creation of geopolymer mechanical properties. The gradual development of this material structure and the formation of bonds between its constituent particles are responsible for the observed trend of increasing flexural strength over time. Therefore, extended curing times can lead to the development of higher flexural strength values in geopolymer materials.

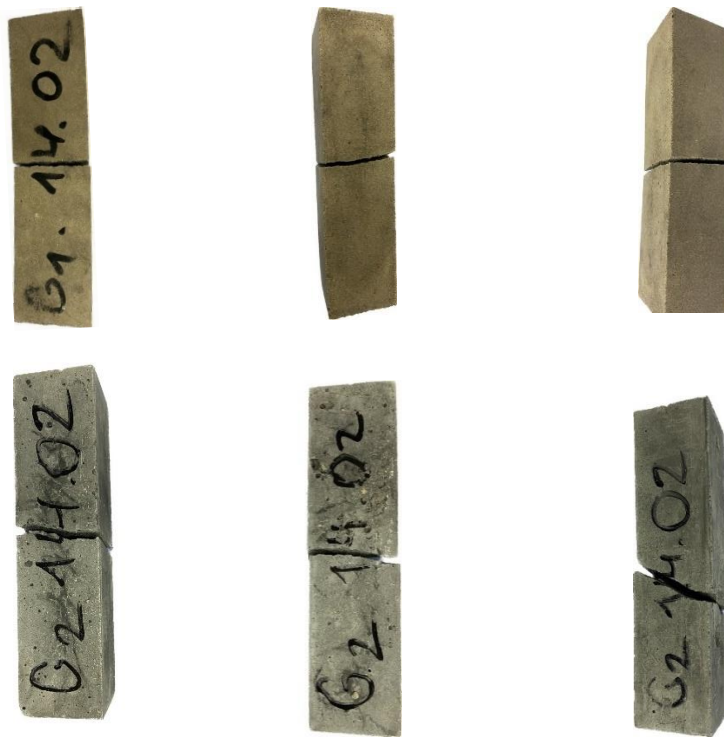


Figure 45: Some of the samples after flexural testing: a) geopolymer A b) geopolymer B

Figure 45 illustrates samples of flexural specimens as seen from the top and side. The middle of every specimen with a horizontal layer was evident.

4.4 Setting time results

In this study, geopolymer samples denoted as A and B were evaluated for their setting time. However, the experimental observation revealed that the material did not dry even after 6 hours of monitoring, and the testing device could not be employed to evaluate the samples' setting time. This result suggests that geopolymer samples A and B may possess unique properties, such as high porosity and low permeability, which can hinder the drying process and delay the setting time.

To further evaluate the characteristics of samples A and B, alternative testing methods, including SEM or XRD, could be employed. These techniques can provide insights into the microstructure of the samples, which can help explain the observed behavior. Overall, understanding the behavior of geopolymer mechanical properties is crucial for their successful implementation in various applications.

The setting time of geopolymers C and D were determined to have yielded satisfactory outcomes, as shown in Figure 46. Specifically, the initial setting time of geopolymer C was found to be 180 minutes, with the final setting time being evaluated at 420 minutes. On the other hand, geopolymer D exhibited an initial setting time of 210 minutes, with the final setting time being evaluated at 450 minutes. These findings indicate that both geopolymers C and D possess sufficient setting times, which are essential properties for their intended applications. The data

obtained through this analysis may also be useful in optimizing the formulation and production of these geopolymer materials for future use.

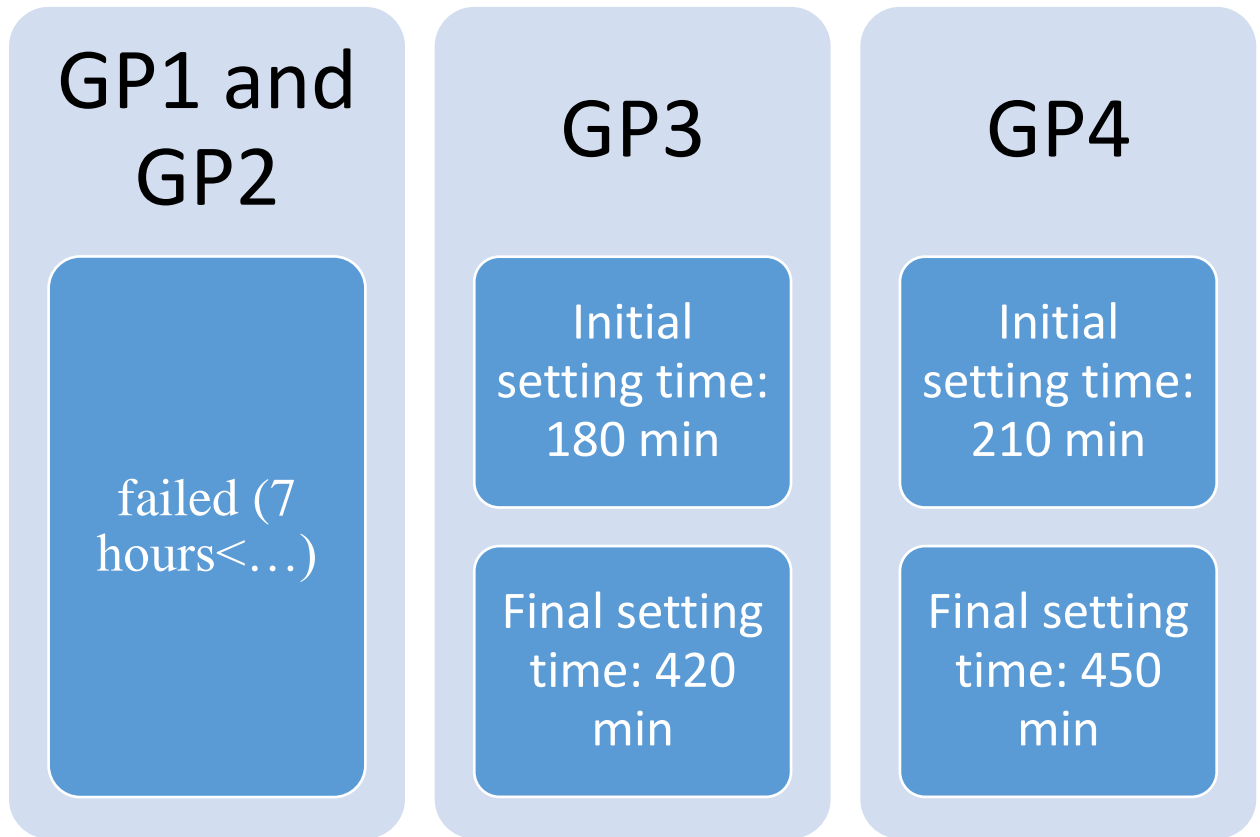


Figure 46: Setting time results

The observed setting time values suggest that geopolymer D has a relatively long setting time, which may be advantageous in certain applications where longer setting times are desired. However, it is worth to note that the setting time can be influenced by various factors, including the composition of the geopolymer mix, curing temperature, and curing time.

4.5 Results of Microwave Heating

Sun et al. [127] conducted a study to evaluate the effectiveness of microwave heating as a means of improving the mechanical properties of materials. Their findings suggest that this method is only applicable to geopolymer samples that contain fly ash, as it may decrease the properties of those that do not. To test this hypothesis, several geopolymer samples were subjected to microwave heating for different durations (20, 30, and 40 seconds) using a power output of 800W. A geopolymer sample that had been cured for 1 day and had a compressive strength of 28,749 MPa was selected as a reference point. However, the results obtained after the microwave heating indicated that the compressive strength had decreased, as can be observed in Table 18.

Table 18: Results of microwave heating after 1 day of curing (GP3)

800 W	20 s	22,632 Mpa
	30 s	9,231 Mpa
	40 s	11,96 Mpa

However, this research work evaluated the effects of microwave heating on GP4, which contains fly ash, and its impact on compressive strength. The results indicated a substantial improvement of 24% in compressive strength when the sample was subjected to 800 W of power for 10 seconds. A reference point was established using a geopolymer sample that had been cured for 1 day and had a compressive strength of 5.932 MPa. Following microwave heating, the compressive strength of the sample increased to 7.621 MPa.

The outcomes of the study are presented in Table 19, which shows the compressive strength values of all the heated samples. The enhancement in compressive strength seen in the GP4 sample points to microwave heating as a feasible strategy for raising the strength of fly ash-containing geopolymer samples. The findings of this study contribute to the development of sustainable and efficient methods for enhancing the strength properties of geopolymers. Further research is recommended to investigate the effects of different power and time parameters on the properties of geopolymer samples subjected to microwave heating.

Table 19: Results of microwave heating after 1 day of curing (GP4)

600 W	10 s	6.633 MPa
	20 s	5.358 MPa
	30 s	6.014 MPa
700 W	10 s	4.668 MPa
	20 s	6.294 MPa
800 W	10 s	7.621 MPa
	20 s	5.179 MPa
	30 s	4.884 MPa

4.6 Expected experiments with the 3D printer

For this investigation, a cavity pump on the 3D-printing machine will be operated to extrude the material. The geopolymer will be extruded using the built-in pump and the rectangular nozzle. The toolpath design will also impact the fresh properties of the various mixtures, which has been demonstrated to be a crucial component in successful 3D-printing toolpath design. The resulting geopolymer paste will be applied layer by layer using a laboratory 3D printer with an assembly volume of 100x190x180 cm (WxDxH) as illustrated in Figure 47 and Figure 48.



Figure 47: Concrete 3D printer

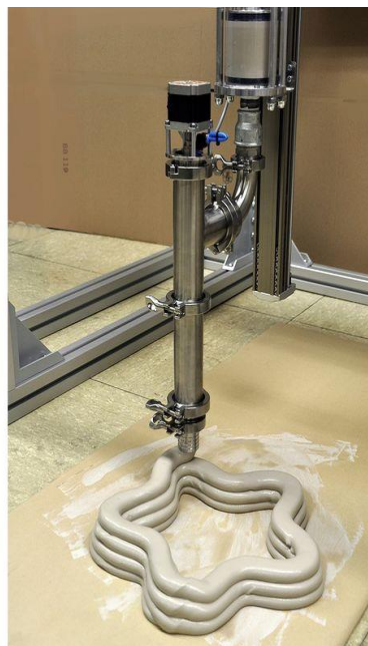


Figure 48: Mixing print head

Data preparation for printing, collection, and output are all necessary steps in the 3D printing research process. Rhino® and Grasshopper® are used to calculate and generate toolpaths for this study. The 3D modeling tools of Rhino are connected with the graphical algorithm editor Grasshopper, which enables the generation of toolpaths and parametric designs using geometrical tools. Because of their parametric options, these technologies are primarily utilized for architectural projects. Then the toolpaths are translated using the Grasshopper® plugin. Process variables, such as printing speed and nozzle height, will be kept constant to ensure that the microwaving period formed the only dependent factor affecting the quality of the produced objects in this investigation.

The evaluation of the material's extrudability and buildability is a critical aspect of this investigation, as detailed in Section 3.3. To this end, a sample cylinder will be printed, as depicted in Figure 49, to determine the material's suitability for additive manufacturing applications. Specifically, GP 3, one of the four types of geopolymers synthesized, will be utilized in the printing process, as it has yielded the most promising results among the tested formulations.

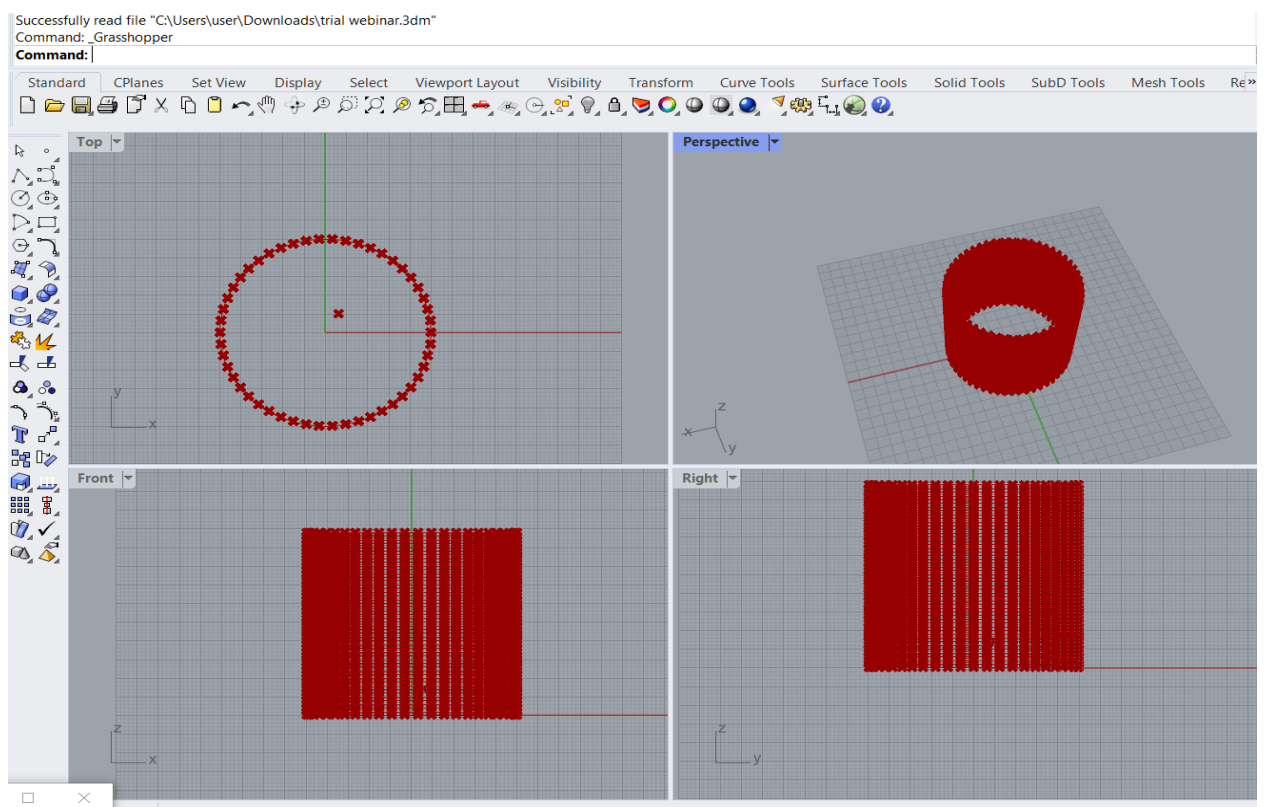


Figure 49: Creating a model in Rhino 7

Moreover, Figure 50 illustrates the G-code required for the printing of the proposed cylinder model. G-code plays an indispensable role in the additive manufacturing process, as it guides the printer's actions to create the desired geometry. The implementation of Mix 3 in

conjunction with the appropriate G-code parameters will enable the production of a high-quality cylinder prototype, which will be subjected to rigorous testing to evaluate its extrudability and buildability properties.

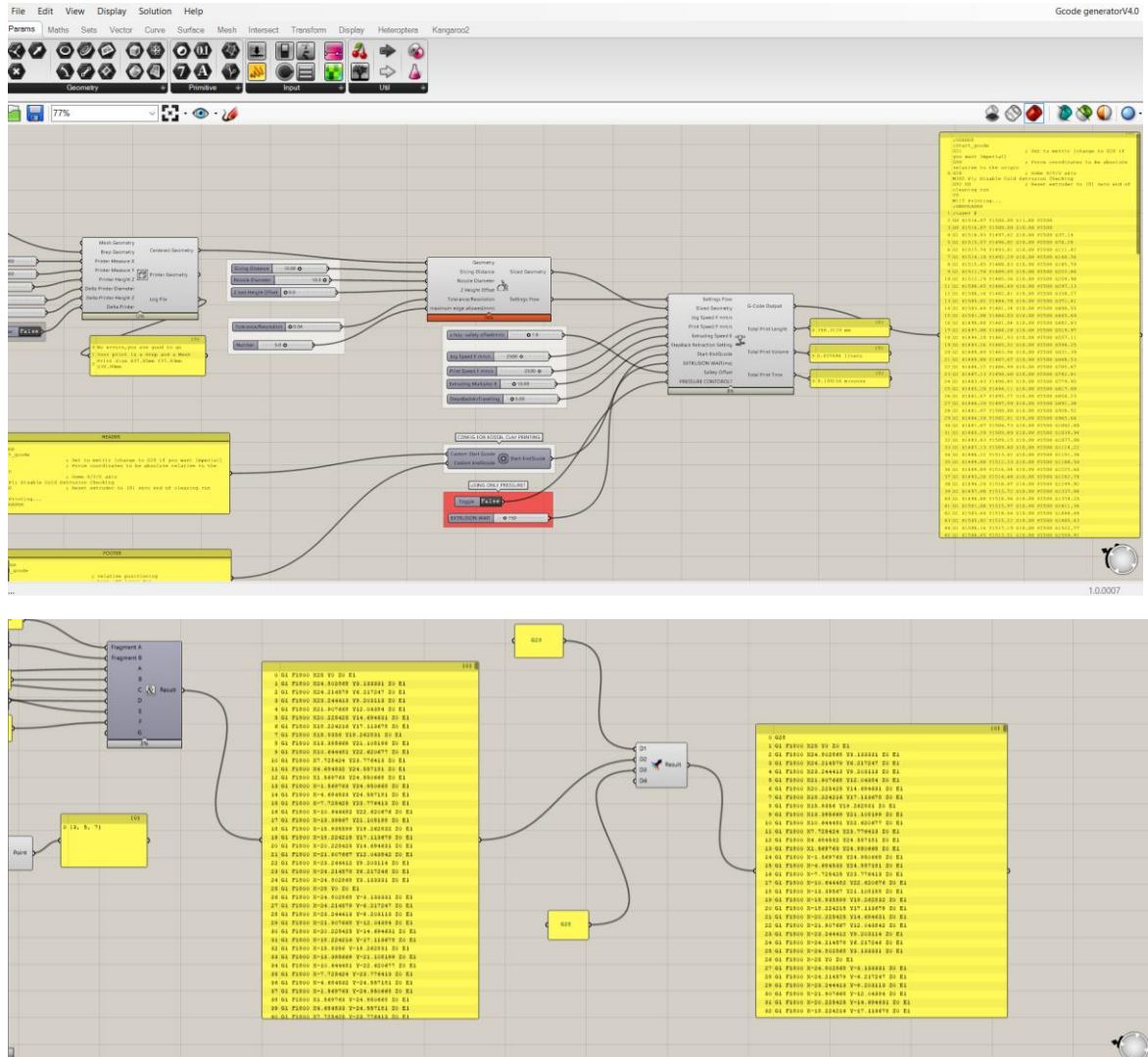


Figure 50: Generating G-code for cylinder in Grasshopper

The utilization of GP3 in the printing process is anticipated to produce highly favorable outcomes, thereby supporting the material's suitability for various additive manufacturing applications. By conducting a comprehensive analysis of the printed cylinder's characteristics, the study aims to contribute significantly to the existing knowledge on the potential applications of geopolymers in additive manufacturing.

4.7 Discussion

The evaluation of the properties of geopolymer is critical for understanding their potential applications and optimizing their performance. The use of rigorous testing methods and analytical techniques can provide valuable insights into the behavior of these materials over time, helping to unlock their full potential.

The study evaluated the mechanical properties of four types of geopolymer, which were cured for 1, 3, and 7 days at ambient temperature. Analysis of the compressive and flexural strengths of the geopolymer specimens revealed a remarkable enhancement in the mechanical characteristics of the material over time. The data indicate that longer curing times are necessary to expand the mechanical properties of geopolymer, which is important for its effective use in the construction industry and other sectors.

The geopolymer containing metakaolin (Metaver) and Fly Ash showed an initial compressive strength of 2.797 MPa, which significantly increased to 19.242 MPa after 7 days of curing. Similarly, the geopolymer containing metakaolin (Kokshetau) and Fly Ash exhibited an initial compressive strength of 6.07 MPa, which increased to 22.63 MPa after 7 days of curing. The flexural strength of geopolymer samples also increased significantly over time, with the highest value recorded after 7 days of curing.

Interestingly, geopolymer samples denoted as A and B did not dry even after 6 hours of monitoring, and the testing device could not be employed to evaluate their setting time. This result suggests that these samples may possess unique properties, such as high porosity and low permeability, which can hinder the drying process and delay the setting time. To learn more about the behavior of these materials, additional testing techniques like SEM or XRD might be beneficial. Ultimately, the study established the geopolymer D setting time, which was found to be 210 minutes for the initial setting and 450 minutes for the final setting.

Comparing the results, it is obvious that geopolymer C showed the highest values of mechanical strength among all geopolymer samples, closely followed by geopolymer D. The results of geopolymers A and B showed relatively lower results compared to geopolymers C and D. This can be explained by the composition of geopolymers, and it is assumed that the quality of the metakaolin that was used for geopolymers A and B is low, since for the preparation of geopolymers C and D, all components remained the same except metakaolin.

The effect of mixing order on the synthesis of geopolymer was examined in a recent study by [140]. After three days of curing, the compressive strength of the resulting concrete was measured using three different types of mixers, one of which was FA-based. For the FA-based geopolymer concrete created in their mixer, the authors reported a compressive strength result of 17.85 MPa. In comparison, this research work also examined the compressive strength of geopolymer concrete after 3 days of curing, using an FA-based geopolymer designated as D. The compressive strength result obtained in this study was 17.93 MPa, which is slightly higher than the result reported by [140].

These results raise the possibility that the mixing order may have a small impact on the compressive strength of geopolymer concrete, but more analysis is required to substantiate this

hypothesis. It is crucial to choose the mixing technique when constructing geopolymer concrete carefully since the compressive strength of the material may be affected by the use of various mixers. A successful deployment of the geopolymers in diverse applications may be aided by the study's significant insights into the mechanical characteristics and setting times of various types of geopolymers. The findings emphasize the value of longer curing times for improving the mechanical characteristics of geopolymer, which can expand its potential uses in building and other industries. Further testing techniques may offer more information about the behavior of geopolymers and may result in the creation of fresh, enhanced formulations.

CHAPTER 5 – Conclusion and Future Work

5.1 Conclusion

The research presented leads to the conclusion that AM is a technology with great potential for the construction sector. The use of AM for concrete structures has numerous benefits, including freedom in design, mechanized construction, and eco-friendliness. Unfortunately, due to technological shortcomings, particularly in mechanical control and suitable printing materials, the application of AM in this field is currently constrained.

The research study focused on developing an optimal extrusion design for 3D-printed geopolymer materials using locally sourced raw materials. The research aimed to identify the components and parameters necessary for successful extrusion design, determine the ideal proportion and mixture of geopolymer for the extrusion process, investigate the use of microwave curing to accelerate the formation of the geopolymer structure, and validate the outcomes of the study by testing the main characteristics of the geopolymers.

Extended curing times greatly improve the mechanical properties of geopolymer, which is essential for its effective use in construction and other industries, according to the research study. The research also identified that insufficient research has been conducted on the properties of geopolymers, optimal extrusion design for 3D-printed geopolymer materials using locally sourced raw materials, specific standards for geopolymer, and the need for research specifically focused on mechanical control and appropriate printing materials for the extrusion-based method of AM.

5.2 Contribution to Knowledge

The research study focuses on investigating an optimal extrusion design for 3D-printed geopolymer materials utilizing locally sourced raw materials. The proposed methodology includes exploring the components and parameters of the entire structure necessary for a successful extrusion design, determining the ideal proportions and mixture of geopolymer for the extrusion process, and assessing the microwave heating methods of geopolymers' potential for industrial-scale production.

The research study's proposed methodology and findings can also have significant economic and architectural advantages for the construction industry. By developing an optimal extrusion design for 3D-printed geopolymer materials, your research will enable the creation of structures with complicated designs while reducing labor costs and formwork. Furthermore, by providing guidelines for the production of geopolymers, your study can encourage the industrial production of geopolymers, which can be an economically affordable 3D construction printing system using locally sourced raw materials.

In summary, this research study's contribution to knowledge lies in its proposed methodology and findings, which can have significant environmental, economic, and architectural advantages for the construction industry.

5.3 Future work

Future research in the areas below can be done to increase understanding in those areas:

1. Investigation of the long-term performance of 3D-printed geopolymer structures: While the mechanical characteristics of geopolymers have been examined in this study, the long-term performance and durability of these structures remain unclear. Further research can be conducted to investigate the durability of geopolymer structures under different environmental conditions, including exposure to water, temperature, and chemicals.

2. Optimization of the extrusion process parameters: The extrusion process parameters, including the extruder speed, nozzle size, and layer thickness, can impact the quality and accuracy of 3D-printed geopolymer structures. Future research can optimize these parameters to achieve higher accuracy, faster printing speeds, and better surface quality.

3. Scale-up of AM methods: The 3D printing process for geopolymer materials was evaluated in this study at a laboratory scale. Research in the future can concentrate on expanding the procedure for industrial-scale manufacturing and investigating the viability of utilizing 3D-printed geopolymer structures in actual construction projects.

4. Comparative analysis of geopolymer materials with other construction materials: It has been demonstrated that geopolymer materials have several advantages over conventional building materials, particularly affordability, and environmental friendliness. Future research can conduct a comparative analysis of geopolymer materials with other construction materials, including concrete and cement, to ascertain geopolymer's viability as a sustainable substitute.

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