

MATERIALS AND METHODS FOR DIGITAL CONSTRUCTION

by

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## ABSTRACT

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The benefits of digital manufacturing and 3D printing technology when applied in the construction industry have proven to demonstrate considerable advantages over conventional production methods including increase in efficiency, reduced labor, ability to realize complex architecture and design optimization to effectively utilize materials characteristics and decrease production waste. For industry today, the 3D printing has emerged to address many of the needs to improve the material performance, enhance properties, and deliver complex structures with efficiency. In the case of concrete, the extension of the extrusion method to 3D (and even 4D) printings is the technological challenge that requires the modification of fresh properties of concrete and asphalt which was further explored in this research. This research reports on the extrusion of cement based compositions using a custom made 2D printer. By incorporating additional material modifiers such as polymers, fibers, and nanoparticles the fresh properties of extruded mixtures can be precisely controlled. Such mixtures can be precisely optimized to not only for the digital of 2D/3D printing and non-traditional responses such as auxetic behavior which can be tuned to perform in harsh conditions such as extreme-temperature exposures. Future research is needed to implement the developed materials and concepts in critical

components of infrastructure which, in addition to mechanical loadings, undergo thermal expansion and exposed to harsh temperature environments.

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## RESEARCH OVERVIEW

Modern construction industry needs paradigm shift towards digital manufacturing, where advanced technologies such as 3D printing and material extrusion are tuned to effectively mass manufacture the construction materials with improved performance. This work reports on the effects of polymer-based and nanosilica viscosity modifiers in portland cement-based inks to improve the fresh properties and mechanical performance, and investigated the suitability of developed compositions for digital manufacturing.

The objectives of this study were to characterize the reactive powder materials used in the materials of construction design, such as portland cement, limestone, and clay to conduct trial prints of compositions with clay and concrete mixtures using 2D/3D printing equipment to determine the key variables for the extrusion, to test obtained ceramic and cement-based composites, to conduct testing of optimized hybrid asphalt mixtures based on large volumes of limestone and cement reactive powders under low-temperature fracture, and to evaluate the response of 3D printed auxetic composites.

Based on the experiments, it was demonstrated that water-soluble polymers (such as PVA) significantly improve the workability, extrudability, and mechanical properties of cement based materials. The optimized cement based inks were developed for extrusion and 3D printing providing potentially cost-effective and sustainable solutions for future digital construction.

The research findings can have important implications for construction industry, paving the way for the adoption of digital manufacturing technologies in the production of advanced construction materials. By leveraging the benefits of the advanced technologies, new materials

can be created that are stronger, more durable, and more sustainable, while reducing costs and minimizing construction waste.

Overall, this work contributes to the growing body of knowledge on digital manufacturing in the field of construction industry and provides insights into the potential of polymer nanomaterial modification on improving the performance of construction materials.

# 1. INTRODUCTION

## 1.1 Background and problem statement

In the era of sustainability and advanced technology, the performance of structural materials is continuously improving due to engineering maximizing the properties and enhance the value. Engineers have been able to manipulate the composition and structure of materials at the nanoscale to achieve the enhanced mechanical, thermal, and electrical properties. In particular, improvements in strength, modulus of elasticity, and thermal expansion of materials have enabled new applications and technologies.

Modern materials can be engineered to possess new properties and perform in non-traditional ways that are not characteristic to their inherent bulk structure. These can be called meta-materials whose macro-structure can be designed so that the elastic modulus and Poisson ratio are manipulated due to optimization of internal geometrical structures. Examples of these materials are auxetic polymers and ceramic materials, which are not yet found in biology (Ren et al.2018).

Within the industry today, the 3D printing of structural elements has emerged into the field of ceramics, concrete, asphalt and steel digital manufacturing. In the latter methods, the “ink” or cementitious (Jianchao et. al, 2017, Rehman et. al, 2021). The extrusion method is the technology that allows for more modification concrete performance and production methods and hence further explored in the reported research. The potential of 3D printing in the construction industry has demonstrated that the extrusion technique is a viable method for producing structures with specialized properties and designs. Recent advancements in the development of materials for 3D printing of concrete have been the focus for many researches. A study by Jianchao et al. (2017) focuses on the development of a cement-based ink for 3D printing of

concrete. The research investigates the effects of various factors, such as the ink composition, nozzle diameter, and printing speed, on the printing quality and mechanical properties of concrete. The study demonstrated the potential of 3D printing as a tool for producing complex geometries and innovative designs that are difficult to achieve using traditional construction methods (Yang et al, 2021). The use of specialized concrete and asphalt mixtures can also lead to improvements in the mechanical properties and durability of the printed structures, making the se more suitable for the use in a real-world applications. A review conducted by Rehman et al. (2021) encompasses the current state of knowledge about the mix designs rheology, mechanical properties, microstructural analysis, and durability of 3D printed concrete. The authors identified the main factors affecting the properties of 3D-printed concrete, such as materials printing parameters, and curing conditions. These studies proved that the development of materials for 3D printing is an active area of research and highlight the importance and potentials for material selection and optimization as required for successful implementation of 3D printing in the construction industry.

Yet portland cement concrete is not the only material used in construction or transportation, because various applications can require different approaches and alternate solutions such as asphalt to sustain pavements at varying loading and temperature conditions. The incorporation of polymer and reactive powders such as portland cement can enhance and convert conventional asphalt 'black tar' into a new product, which is strong, waterproof, self-healing and durable.

## **1.2 Hypothesis**

The use of chemical admixtures, polymers and nano-silica as rheology modifiers can affect the fresh properties of portland cement concrete and asphalt mixtures resulting in optimized performance which can be cost effective solutions for construction material extrusion and 3D printing technologies.

## **1.3 Objectives**

- Characterize the reactive powder materials used in the materials design, such as portland cement, fly ash, limestone, clay;
- Conduct the extrusion tests with clay, cement based ink mixtures using 2D and 3D printing equipment to determine the variables affecting the extrusion of composites;
- Test ceramic and cement based composites manufactured with 2D and 3D printing application;
- Conduct testing on optimized asphalt mixtures at low temperatures mechanical response and fracture, and toughness;
- Develop a test procedure for evaluation of extrudability of clay and cement based inks.

## **1.4 State of the art review on performance of asphalt concrete**

Concrete is one of the most used construction materials that humanity has created. This material is a strong and durable that can withstand heavy loads and extreme weather conditions while also being versatile and so applicable for a wide range of construction projects, from residential buildings and bridges to roads and dams.

Since the development of Superpave® in the late 1980's, winter weather performance of asphalt concrete has been one of the main areas of research (Asphalt Institute 2001, Fromm et al, 1971). At low temperatures, most asphalt binders act as an elastic non-Newtonian material where the ratio of shear stress to shear strain is not proportional. At lower temperatures the material deforms under load but then returns to its original shape once it is unloaded. Typically, at lower temperatures, a low stiffness is generally desired because this allows the asphalt material to resist low-temperature cracking (Kandhal et al, 1988). If the material is stressed beyond the material strength capacity, the asphalt can fracture as other brittle elastic solids and this results in thermal cracking (Gaw 1977).

Low-temperature thermal cracking is a considerable issue to asphalt pavement designers.

Thermal cracking is especially important to evaluate in climates with cold temperatures because these result in non-load associated distress. By definition, thermal cracks are intermittent transverse cracks that are formed when the asphalt material contracts due to low temperatures. If the tensile stresses within the layer exceed the tensile strength of the material then the asphalt layer cracks. These thermal cracks can form from a single-cycle of low temperatures and can develop from repeated freezing and thawing cycles.

When an asphalt material cracks, water and aggressive chemicals can penetrate the crack and start to deteriorate the asphalt pavement. Prevention of water penetration is critical for the service life and sustainability. Once deterioration is significant, potholes repair materials are required to fill the crack and prevent future deterioration. The repair material must bond to the surrounding pavement and prevent water from penetrating into the boundary between the patch and the existing material (Roberts et al, 1996). After the pothole repair material is placed, it is then important to ensure the low-temperature performance.

When performing the material evaluation under low-temperature conditions, proper selection of proper asphalt binder is the best way to assure the adequate resistance to thermal cracking (Anangos et al 1972). Researchers have recommended that the use of patching materials with low asphalt binder stiffness helps to reduce the effects of thermal cracking. Commonly excessively aged asphalt binders have poor performance at lower temperatures because of the age hardening developed due to excessive oxidation. Therefore, repair mixtures should be designed with soft asphalt binders with extended aging resistance to minimize effects of low-temperature thermal cracking.

Possible solution to design sustainable pavement includes Cold Mix Asphalt (CMA) based on bitumen emulsion which can be used to prepare the pothole repair material. An asphalt emulsion is a mixture of asphalt, water, and an emulsifying agent. In CMA, when the mixture is placed, the water typically evaporates from the mixture, thus leaving the asphalt binder to hold the aggregates together. A research project at UWM investigated CSS-1hL asphalt emulsion with a 62.6% residue content. Where 62.6% of the emulsion was asphalt binder and the remaining 37.4% was water (with a small percentage of the emulsifying agent). The "CSS" is a cationic

emulsion that is slow setting. The "1" refers to a lower viscosity emulsion. The "h" means that the emulsion residue is harder and the "L" means that the emulsion is heat up.

The objective of research was to use reactive powder materials such as fly ash and portland cement CMA mixtures. It was envisioned that these materials can improve the performance of the repair material and can also provide a partial replacement and reduce the overall emulsion quantity. Latex was used to enhance the performance of fly ash (Class C) and portland cement CMA mixtures. [ fly ash and Portland cement were used at a 30% emulsion residue replacement (by mass). Table 1 lists 5 mixture types and specific mixture [proportions. Figure 1 demonstrates the shape of the specimens] as well as finished specimen surfaces.

**TAB LE 1-1: The composition of CMA mixture proportioning (grams)**

Mixture	Sample ID	Mixture proportioning (grams)					
		Emulsion	Latex	Coarse aggregate	Sand	Fly ash	Portland cement
1	Control	250	0	315	135	0	0
2	Fly ash 30%	175	0	315	135	75	0
3	Fly ash 30% + Latex	175	50	315	135	75	0
4	Portland cement 30%	175	0	315	135	0	75
5	Portland C 30% + Latex	175	50	315	135	0	75



**FIGURE 1-1: Specimens and specimen surfaces based on (from left to right):**

**control, fly Ash 30%, fly Ash 30% + latex, portland C 30%, and portland C 30% + latex**

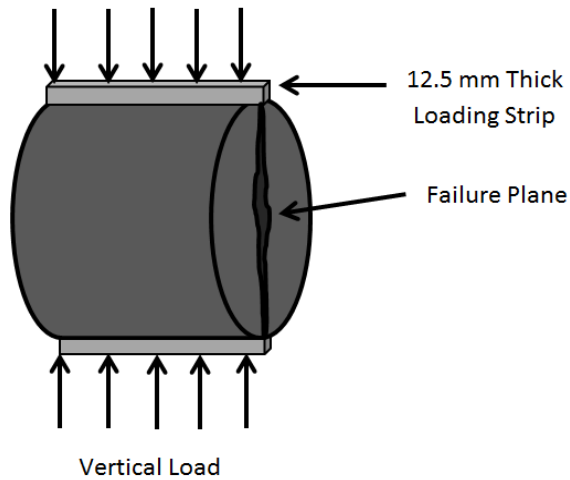
Latex was added to further soften the asphalt binder matrix (which already contained some latex) which could induce a more elastic response at lower temperatures and prevent low-temperature thermal cracking. Fly ash was added to enhance the performance and to also reduce the overall emulsion volumes. Lastly, portland cement was used as an innovative way to develop a self-healing pothole repair material. The overall idea of the proposed patch material was to have a material that can provide an excellent bond to existing pavement material, but can also self-heal if cracks are developed. It is envisioned that the unhydrated portland cement grains within the mixture matrix can hydrate and harden if water penetrates the crack and thus seals the crack and prevents further cracks from developing.

In this study, the low-temperature thermal cracking of Cold Mix Asphalt mixtures was evaluated by using the Indirect Tension Testing (IDT) machine. The Humboldt Indirect Tensile Machine (IDT) was used to evaluate indirect tension properties in accordance to ASTM D6931-12. This machine uses a single compressive load that acts parallel to the vertical plane of the specimen

(Figure 1.2). As the vertical compressive load pushes down on the specimen (at a rate of 50 mm/min.), the horizontal tensile forces begin to develop. The specimen then fails by splitting in half along the vertical plane that the load acts on (Figure 1.3). The required thickness of the loading strip for a 101.6 mm diameter asphalt specimen is 12.7 mm and this setup was used for this study. This specific thickness provides a uniform loading condition which produces a nearly uniform stress distribution. Before testing, the specimens were conditioned at  $-18^{\circ}\text{C}$  for 24 h.



**Figure 1-2: Humboldt indirect tensile (IDT) machine**



**FIGURE 1-3: Indirect tension test Setup with induced plane failure**

The IDT provides two important properties that are very useful in the analysis of CMA mixtures: ultimate tensile stress and tensile strain at failure. Mixtures that develop high ultimate strengths can typically resist the effect of vehicle loads better than softer or weaker materials. Mixtures that develop high tensile strains can typically be classified as a more elastic material since these materials can deform without necessarily cracking or fracturing. It is important to achieve both high ultimate tensile strengths as well as high tensile strains at failure.

Equations for tensile stress and tensile strain are used as following:

$$\sigma_x = \frac{2P}{\pi dt} \quad \text{Eq. 1}$$

$$\sigma_y = \frac{6P}{\pi dt} \quad \text{Eq. 2}$$

$$\text{where: } \varepsilon_f = 0.52x_t \quad \text{Eq. 3}$$

$\sigma_x$  = horizontal tensile stress at center of specimen, (MPa);

$\sigma_y$  = vertical tensile stress at center of specimen, (MPa);

P = applied load, (N);

d = diameter of specimen, (mm);

t = thickness of specimen, (mm).

$\varepsilon_f$  = tensile strain at failure (mm/mm);

$x_t$  = horizontal deformation across the specimen (in.).

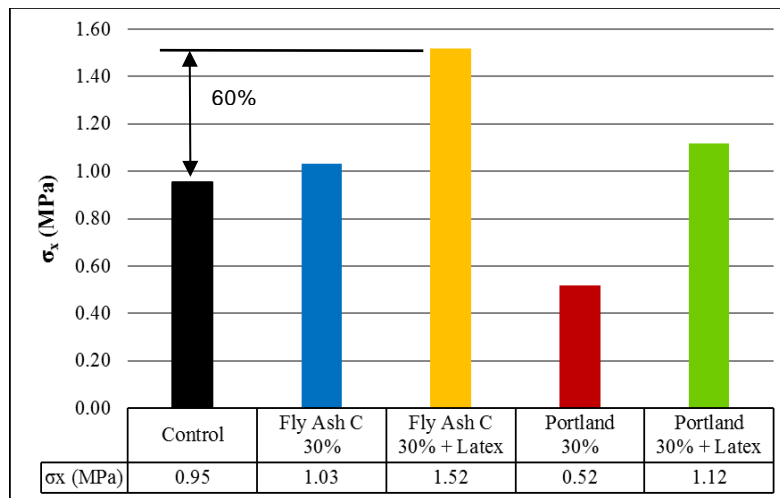
The indirect tension testing was used to evaluate the low temperature properties of developed asphalt patching materials. The results of the Indirect Tensile Test (IDT) of the CMA samples with reactive powders are reported in Table 1.2. This table also lists the diameters and

thicknesses of the specimens. These results demonstrate that most of the specimens developed higher strength when compared to the control mixture. The Fly Ash 30% + Latex material had failed under the highest load of 9.74 kN which is much higher than what was observed for the control mixture. The Portland C 30% mixture had the lowest load at failure of only 3.65 kN. When comparing the horizontal deformation at failure, the Portland C 30% + Latex mixture developed the highest failure deformation at 3.84 mm which is an indication of excellent elastic behavior at the low temperatures. The Portland C 30% mixture, however, had the lowest deformation at failure with only 1.70 mm and this is an indication of a more brittle material response observed at low temperature.

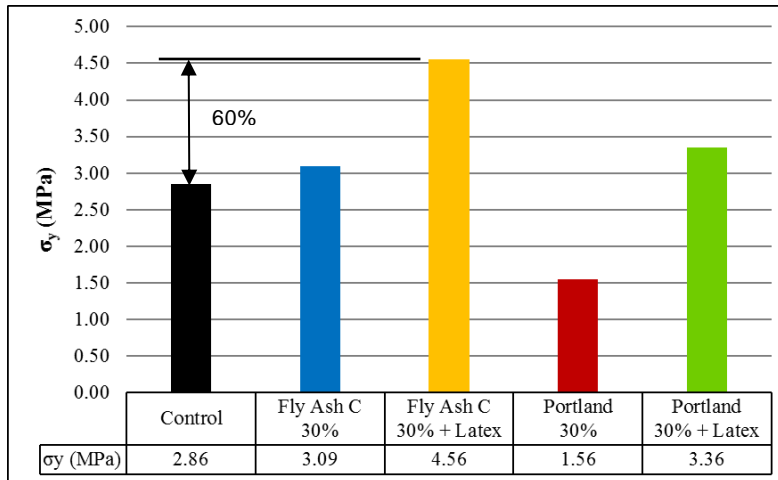
**TAB LE 1-2: The IDT ultimate load and horizontal deformation at failure**

Sample ID	P (kN)	$\delta_f$ (mm)
Control	5.4	2.62
Fly Ash 30%	6.5	3.61
Fly Ash 30% + Latex	9.7	2.06
Portland C 30%	3.7	1.70
Portland C 30 % + Latex	7.7	3.84

Figure 1.4 and Figure 1.5 report the horizontal tensile stress ( $\sigma_x$ ) and the vertical tensile stress ( $\sigma_y$ ) of the CMA samples respectively. These results give a visual correlation to the maximum load results represented in Table 1.2. From these results it can be concluded that the maximum vertical and horizontal stresses at failure for most of the CMA samples were higher than the control samples. The Fly Ash 30% + Latex mixture had the highest maximum horizontal stress of 1.52 MPa and a maximum vertical stress of 4.56 MPa. The Portland C 30% + Latex also developed a significant maximum horizontal stress at failure of 1.12 MPa and a maximum vertical stress of 3.36 MPa at failure. The Portland C 30% material had the lowest maximum horizontal stress of 0.52 MPa and the lowest maximum vertical stress of 1.56 MPa. This demonstrates that the addition of latex provides considerably improved mechanical performance.

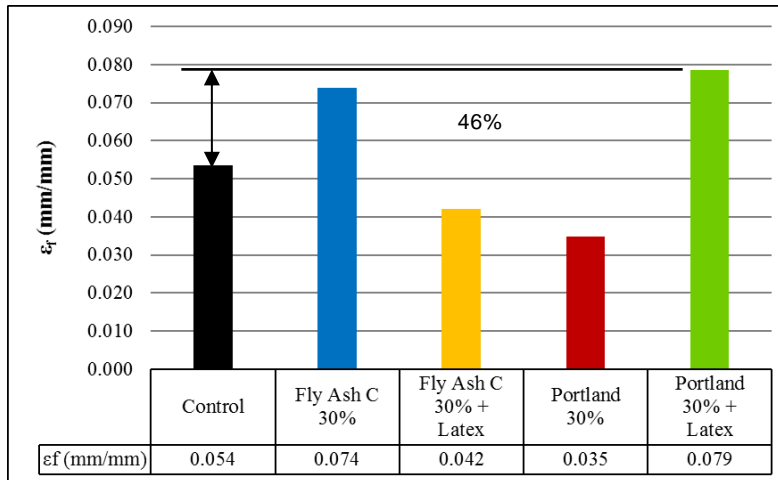


**FIGURE 1-4: The comparison of the horizontal stress at the center of the specimen**



**FIGURE 1-5: The comparison of the vertical stress at the center of the specimen**

Figure 1.6 reports the tensile strain at failure for the CMA specimens. These results give a visual representation of the maximum flow (displacement) results reported in Table 1.2. The Portland C 30% + Latex material experienced the highest strain at failure of 0.079 mm/mm which is an interesting result since typical portland cement concrete mixtures are generally more brittle when tested in tension. This composition had demonstrated significant deformation capacity at 30% emulsion residue replacement (by mass) at the low temperature and this parameter is critical for low-temperature performance. This mixture is especially interesting because it can be positioned as having a potential for self-healing. When cracks begin to develop, there is an expectation that unhydrated cement can react with water and dissolved elements, fill the cracks, hydrate, and harden to seal cracks. Further testing should be performed to evaluate the self-healing potential of this composition.



**FIGURE 1-6: The horizontal tensile strain of developed repair composition at failure**

This research produced very interesting and significant results for low-temperature thermal cracking resistance of the CMA pothole repair material. The addition of fly ash Class C at 30% replacement of emulsion residue provided higher ultimate strengths when compared to the control mixture. When latex was used to modify the mixtures with fly ash, the ultimate strength was even higher demonstrating that the latex aided in strength gain. Compositions with portland cement at 30% replacement of emulsion had a lower ultimate strength when compared to the control mixture. However, when latex was added to the mixtures with portland cement, the ultimate strength was higher than the control mixture. The mixtures with both portland cement and latex also had demonstrated the highest deformation at failure out of all the mixtures which is an interesting and promising result. It was concluded that further testing should be focused on the evaluation of mixtures with higher dosages of portland cement, fly ash, and potentially latex in respect to potential self healing behavior. Overall, whit application route makes this material suitable for 3D printer.

### **1.5 Potential solution, 3D printing**

Currently, 3D printing technology has made it possible to produce complex shapes and structures using concrete as an ink material. The 3D printed concrete structures have several advantages, including faster construction times, lower labor costs, and reduced production waste. The 3D printing also allows for greater design flexibility, enabling the architects and engineers to create more intricate and innovative structures. Additionally, 3D printing can reduce the environmental impact of construction by using less material and producing less waste. The use of 3D printed concrete is still a relatively new area, but it is an exciting development in the construction industry with strong potential to transform the way buildings are designed and constructed.

In addition to concrete, road infrastructure utilizes asphalt and a range of bitumen-based materials used from the beginning in Sumerian civilizations in Mesopotamia. Asphalt concrete is composed of asphalt cement and aggregates. The asphalt cement primarily functions as a binder, to hold the aggregate when placed onto the surfaces. The binder is composed of bitumen which is a petroleum derivative which can be very brittle at low temperatures, while at intermediate service temperatures, asphalt concrete is considered a viscoelastic material since it demonstrates both elastic and viscous properties. At higher temperatures, the bitumen can be very fluid, allowing for better incorporation with the aggregates. Due to these variations in material behavior at different temperatures, asphalt cement and concrete are aptly considered to fall into the category of thermoplastics.

Asphalt cement has been used for construction for a long time and has a rich history. Its use dates back to around 6,000 B.C. in Sumeria, where it was utilized as a shipbuilding material. In Egypt around 2600 B.C., it was used as a material for waterproofing, mummification, and

building structures. The material was also used as mortar for buildings and paving blocks, caulking for ships, and for numerous waterproofing applications in different parts of the world. The first known natural asphalt pavement in the United States was laid in 1876 on Pennsylvania Avenue in Washington, D.C. Prior to the mid-1850s, asphalt was obtained from natural pools in various locations worldwide, including Trinidad Lake. However, with the discovery and refining of petroleum in Pennsylvania, asphalt became widely known. By 1907, most asphalt was obtained directly from the distillation process of petroleum refineries rather than from natural deposits. Today, the refined petroleum is the source of almost all asphalt materials.

Asphalt concrete is made up of two distinct components: aggregates and asphalt cement. The mixture comprises around 95% of aggregates, while asphalt cement accounts for roughly 5% of the total mass. Asphalt cement, which is also called binder, is composed of various petroleum hydrocarbons that have different chemical structures. The primary elements in asphalt include carbon and hydrogen, while it also has sulfur, oxygen, nitrogen, nickel, and vanadium. Asphalt binder is a durable and robust material that has exceptional waterproofing and adhesive properties.

Creating a robust and cost-effective infrastructure while minimizing the need for future repairs is a complex undertaking that requires innovative technological advancements. Researchers have investigated the use of mineral fillers like portland cement (PC) and industrial by-products such as fly ash to enhance the performance of asphalt pavements (Ali et al., 1996; Churchill et al., 1999; Asi et al., 2005; Tapkin, 2008; Faheem & Bahia, 2010). In these studies, cement and fly ash were considered as fillers with the expectation of similar performance to mineral fillers. Sobolev et al. (2013) discovered that incorporating fly ash in asphalt mixtures (ASHphalt) improves asphalt performance to a level comparable to polymer modification. This

improvement was attributed to fly ash's unique spherical shape, favorable size distribution, and chemical composition.

Overall, the use of cement, lime, and fly ash in bitumen materials has many advantages as it improves the performance and reduces the environmental impact and costs associated with the production and application of asphalt (Tapkin, 2008). Reactive powder fillers like these provide many benefits in asphalt, including enhanced mixing, placing and compaction, stability, water damage resistance, rutting resistance, flexibility, and freeze-thaw damage resistance.

The use of reactive powders can help to solve the problem of low temperature cracking and promote the phenomenon of 'self-healing' that is from the incorporation of cements (Cloutier 2019).

## **1.6 Asphalt binder and concrete**

Performance of asphalt concrete which is commonly used as a paving material for roads, highways, and other transportation infrastructure. The performance of asphalt mixtures can be evaluated based on several key factors, such as Stability that refers to the ability of the asphalt mixture to resist the deformation and maintain the shape under traffic loads. Stable asphalt mixtures are less prone to rutting and other forms of permanent deformation.

Its durability refers to the ability of the asphalt mixture to resist damage and deterioration due to environmental factors such as temperature changes, moisture, ingress and UV exposure. Smoothness refers to the surface quality of the asphalt pavement, which affects driving comfort and vehicle maintenance costs.

The most important factor for a project is its cost-effectiveness, which defines the cost of asphalt mixtures and the long-term performance. Overall, the performance of asphalt mixtures

depends on a combination of factors such as the type and quality of the asphalt binder, aggregate properties, mixture design production, construction practices, and maintenance. The performance of asphalt mixtures at low temperatures is an important consideration, especially in regions where temperatures can drop below freezing. Low adequate temperature performance is challenging to achieve as it related to the stiffness and brittleness of the asphalt binder, which can lead to cracking and other forms of distress in the pavement. Another challenge is to attain an adequate mechanical performance within a wider range of severe temperatures.

In summary, asphalt pavements that are not designed for low-temperature conditions can experience cracking and other forms of damage, which reduce the service life and increase the maintenance costs. For this reason, it is important to evaluate the low-temperature performance of asphalt mixtures and ensure that they can withstand the conditions of field exposure.

The low-temperature performance of asphalt mixtures is a key parameter for ensuring the safety, durability, and cost-effectiveness of pavement structures. Testing and evaluation of asphalt mixtures at low temperatures can help identifying the materials and design practices that can improve the performance under these conditions.

In cold weather climates, thermal cracking is a crucial parameter to consider since such cracks are directly related to low temperature exposures. Reducing the stiffness of asphalt mixtures and control of thermal expansion properties can mitigate the effects of thermal cracking, and so can be important for low-temperature applications. It is known that stiffer asphalt mixtures typically perform poorly in lower temperatures, while softer asphalt tends to perform better. Aged asphalt binders also tend to exhibit poor performance in lower temperatures, as the binder undergoes excessive age-hardening due to extensive oxidation.

The Semi-Circular Bending Test (SCB) is a widely used test method to evaluate the low-temperature performance of asphalt mixtures. The test involves subjecting a notched semi-circular asphalt specimen to a 3-point bending load at a low temperature. The notch at the bottom of the specimen is used to initiate a crack, and the load-displacement response of the specimen is recorded. The Fracture Energy (Gf) and Stiffness (S) of the asphalt mixture can be calculated based on the load-displacement response of the specimen. The Fracture Energy (Gf) represents the energy required to propagate a crack in the asphalt mixture. The Stiffness (S) of the asphalt mixture is a measure of its resistance to deformation under a given load. The lower the stiffness of the asphalt mixture, the better its low-temperature performance, as it can better accommodate thermal stresses without cracking. The governing equations for these parameters are as follows

$$G_f = W_f / A_{lig} \quad \text{Eq. 4}$$

$$W_f = \int P du = W + W_{tail} \quad \text{Eq. 5}$$

$$A_{lig} = (r - a) * t \quad \text{Eq. 6}$$

$$W_f = \int P du = W + W_{tail}, \text{work of fracture (J)}$$

P = applied load (N);

u = load line displacement (m);

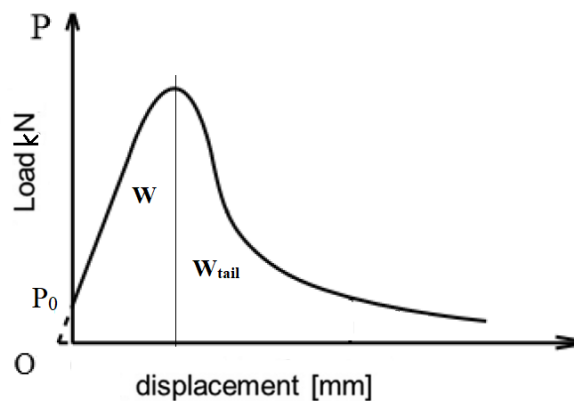
$A_{lig}$  = ligament area (m<sup>2</sup>);

r = specimen radius (m);

a = notch length (m);

$t$  = specimen thickness (m).

To evaluate the resistance of asphalt mixtures to low-temperature thermal cracking, the Semi-Circular Bending Test (SCB) was utilized. The SCB is a 3-point bending test conducted on semi-circular specimens with a notch at the bottom, at lower temperatures, to assess Fracture Energy ( $G_f$ ) and Stiffness ( $S$ ). Fracture Energy,  $G_f$  ( $J/m^2$ ), represents the energy required to generate a unit surface area of a crack. This is calculated by dividing the work of fracture (area under the load vs. load line displacement curve) by the ligament area (ligament length and specimen thickness).



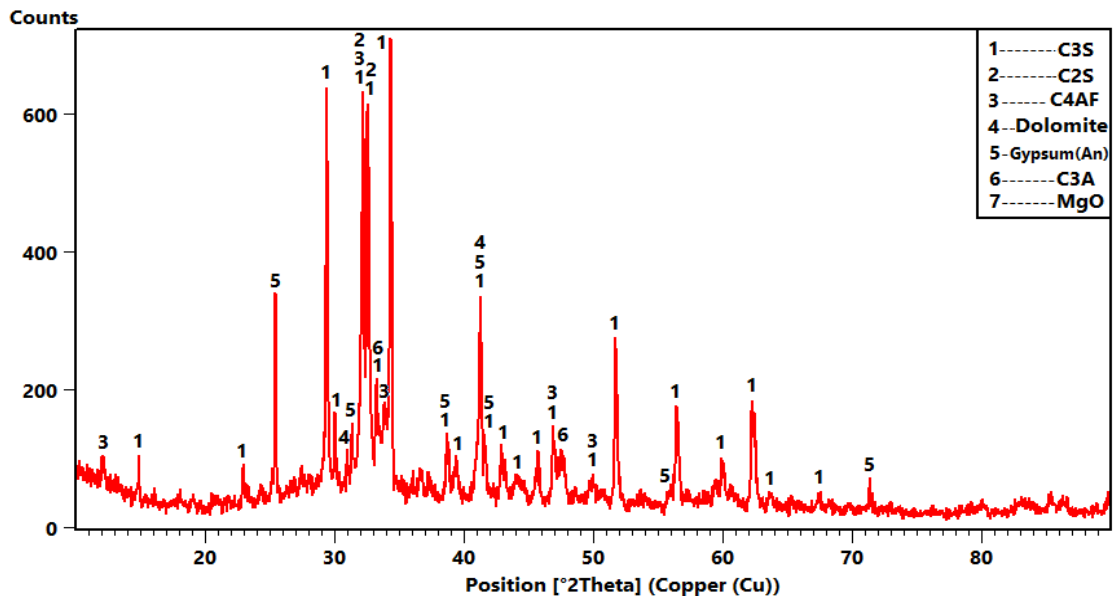
**Figure 1.7: Stiffness ( $S$ ) determination from low-temperature testing**

At low temperatures, asphalt exhibits brittle behavior, lacking the defined ductility response, and is prone to sudden failure under applied loads. Therefore, for fracture toughness calculations based on obtained data, the area of the load-CMOD up to the maximal load designated as  $W$  was used. Also, a pre-load of  $P_0 = 80$  N has been applied prior to fracture testing, so the area up to the preloading was not used for fracture toughness calculations. These adjustments enabled the comparisons of compositions with polymer and filler modifications.

## 2. MATERIALS CHARACTERIZATION

### 2.1 Composition characterization of reactive powders

For mineral verification, a Bruker X-ray diffractometer was used to probe the portland cement and determine the crystallinity of powder before application in the experiments. To prepare the samples for XRD, the powders were homogenized carefully placed onto the shallow well of the XRD holder, and once there was a small amount of material, the metal spatula was used to flatten the mound and this packed the sample into a dense configuration. The surface of the material needed to be flat and dense to ensure that the X-ray absorption is not affecting the intensity of low angle peaks. Once the sample was prepared on the holder, the sample was placed into the main compartment of the XRD machine and magnetically locked into place for testing.



**Figure 2-1:** The XRD pattern of portland cement powder

Using X-Ray Fluorescence (XRF) as per ASTM C114, the chemical oxide composition of the investigated powders was tested to understand the potential interaction with other components of composition and asphalt mastics and asphalt binders.

The chemical oxide composition is summarized in Table 2-1, and it can be observed that some chemical oxides such as Na<sub>2</sub>O, TiO<sub>2</sub>, and P<sub>2</sub>O<sub>5</sub> are present in minor quantities (< 1%).

**Table 2-1: Chemical Composition of Reactive Powders**

<b>Sample ID</b>	<b>Na<sub>2</sub>O (%)</b>	<b>MgO (%)</b>	<b>Al<sub>2</sub>O<sub>3</sub> (%)</b>	<b>SiO<sub>2</sub> (%)</b>	<b>P<sub>2</sub>O<sub>5</sub> (%)</b>	<b>K<sub>2</sub>O (%)</b>	<b>CaO (%)</b>	<b>TiO<sub>2</sub> (%)</b>	<b>Fe<sub>2</sub>O<sub>3</sub> (%)</b>
<b>LS</b>	0.04	5.64	0.81	4.63	0.03	0.14	54.15	0.02	0.49
<b>PC</b>	0.11	3.69	5.02	22.55	0.08	0.99	69.39	0.38	2.68

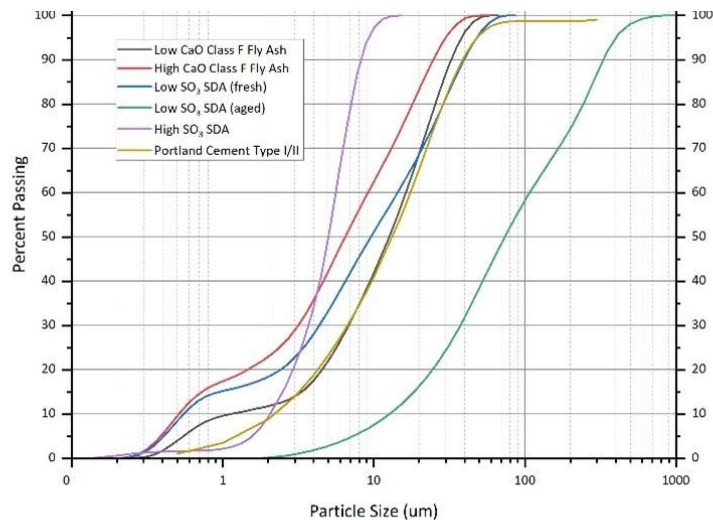
## **2.2 Particle size distribution (PSD)**

The particle size distribution (PSD) of powders was evaluated based on ASTM D4464-10 using laser light scattering. This test method can measure the equivalent spherical diameter for particle sizes in the range of 1 to 300 μm. To conduct the laser diffraction test, a sample of the material was dispersed in isopropyl alcohol solution and circulated through the path of a laser light beam.

When the light beam hits a particle, it is scattered. This scattered light is then collected by a photodetector and converted to an electrical signal, and analyzed with the assumption that all the

particles were spherical. From the PSD curves, the  $D_{10}$ ,  $D_{50}$ , and  $D_{90}$  values are determined. The diameters  $D_{10}$ ,  $D_{50}$ , and  $D_{90}$  correspond to the portion of the material that is 10%, 50%, and 90% finer of corresponding size, respectively. Figure 2-2 presents the particle size distribution curves for all investigated powders. It can be noted that PC reactive powder had a larger average particle size compared to manufactured (not collected from baghouse) LS filler. The PC reactive powder particle size ranges between 2- 98  $\mu\text{m}$ , whereas the LS filler particle size ranges between 1 -59  $\mu\text{m}$ . Here, the LS filler, used as a reference material in this study, is still close to portland cement in terms of production technology (ball milling) and shape.

It can be observed that the LS filler material is characterized by smaller particles since the  $D_{10}$  value is only 3.84  $\mu\text{m}$  when compared to cement reactive powder having a  $D_{10}$  value of 6.3  $\mu\text{m}$ . The values for  $D_{50}$  of LS and PC materials are reported in Figure 2-2. It can be noted that LS filler has a  $D_{50}$  value of 13.13  $\mu\text{m}$ , while this value for PC reactive powder is 17.74  $\mu\text{m}$ .



**Figure 2-2: Particle size distribution of investigated powders**

For 3D printing study, the cementitious materials included Type I portland cement (NPC II), metakaolin (MK) powder, nano-silica in sol-gel form, polyvinyl alcohol (MW 85,000-124,000) from Sigma Aldrich and a high range water-reducing admixture (HRWRA) known as superplasticizer.

The PC Type I/II from Holcim, had the composition as providing by manufacturer that meets ASTM C150.

### **2.3 Research methodology**

The research goals were achieved through the completion of the subsequent tasks.

#### **Task 1: Literature review**

- Conduct a review on asphalt mastic and mixture characteristics which can lead to the development of compositions for 3D printing.
- Report on current research efforts on asphalt mastics with traditional fillers and investigate the limited research on asphalt concrete with portland cement.
- Report on research and application of cement based inks for 3D printing.

#### **Task 2: Experimental design and testing**

1. Evaluate the chemical and physical properties of the reactive powders.
2. Evaluate the components for asphalt and concrete research such as reactive powders, Portland cement, asphalt binder types, and WMA additives to use in asphalt and Portland cement composition.
3. Perform 2D prints of trial cement ink composites.
4. Perform 3D prints of clay and cement based auxetic structures.
5. Investigate the low-temperature performance of reactive powder-based asphalt.

### **Task 3: Make conclusions on application of 3D printing in construction**

#### **2.4 Preparation of cement paste and mortar**

3D printing compositions were tested at varying water-to-cement ratios depending on the dosage of included metakaolin, superplasticizers and PVA pavements in the mix. The metakaolin was added up to 20% by weight, as a replacement of cement, whereas the PVA solution with 5% concentration by weight replaced. Mixes were prepared using a standard 3-speed Hobart commercial mixer using the following protocol:

1. Add the 80% of the water, PVA polymer solution, nano-silica (if used) and SP into the bowl;
2. Add metakaolin to water and mix for 60 seconds using low speed;
3. Add 80% of cement into the bowl and start mixing at a low speed for 60 seconds;
4. Allow the mix to rest for 15 seconds, and add rest of water and any dry remaining powder materials into bowl;
5. Continue mixing for 45 seconds at low speed; then switch to medium speed and mix for an additional 60 seconds.

The compositions investigated for 2D and 3D printing are reported in table 2-3,

**Table 2-3: Experimental matrix of cement pastes**

	Mix ID	Water/Cementitious material	Metakaolin	PVA	Superplasticizer	Nano-silica	Sand/Cementitious material
3D ink	3D ink	0.45	20% cementitious material	2.5% cementitious material	0.5 % cementitious material	0	0
2D ink	32-0	0.32	0	0	0	1	0
	35-0	0.35	0	0	0	1	0
	35-0.5	0.35	0	0	0	1	0.5
	36-0.5	0.36	0	0	0	1	0.5
	37-1	0.37	0	0	0	1	1
	38-1.5	0.38	0	0	0	1	1.5
	39-1.5	0.39-1.5	0	0	0	1	1.5
	0-40	NS0-40	0	0	0	0	1.5
	0-39	NS0-39	0	0	0	0	1.5
	0-38	NS0-38	0	0	0	0	1.5
	0-37	NS0-37	0	0	0	0	1.5
	0-36	NS0-36	0	0	0	0	1.5
	2-40	NS2-40	0	0	0	2	1.5
	2-40	NS2-42	0	0	0	2	1.5
	1-41	NS-41	0	0	0	1	1.5

## 2.5 Asphalt concrete preparation

The asphalt testing protocol includes various testing such as aggregate coating, workability, aging resistance, moisture damage resistance, fatigue-cracking resistance, and low-temperature thermal-cracking resistance. This research reports on low-temperature performance of polymer modified portland cement-based hybrid asphalts. Table 2-4 provides the details of the experimental testing plan for investigated asphalt mixtures. At least two samples were tested for all the tests, and average values were calculated and reported.

**Table 2-4: Experimental matrix for asphalt concrete tests**

Test	Measured Indicator	Type of binders	Filler	Filler Dosage	Replicates per Test
Low temperature performance cracking	Fracture energy	<ul style="list-style-type: none"> <li>• WMD modification</li> <li>• Polymer modification low-temperature grade</li> </ul>	<ul style="list-style-type: none"> <li>• Reference</li> <li>• PC</li> <li>• LS</li> </ul>	40% replacement of binder by volume	$\geq 2$

The composition of investigated asphalt concrete are reported in table 2-5.

**Table 2-5 Asphalt mix proportions**

Mix ID	Sample type	Polymer modified	Replacement Bitumen by volume
NP0	Optimized polymer admixture	Yes	0
NP-LS40	Polymer mix with 40% of LS replacement of bitumen	Yes	40% of Limestone
NP-PC40	Polymer mix with 40% of PC replacement of bitumen	Yes	40% of portland cement
NR0	Reference asphalt with no polymer	No	0
NR-LS40	Ref mix with 40% of LS replacement of bitumen	No	40% of Limestone
NR-PC40	Ref mix with 40% of PC replacement of bitumen	No	40% of portland cement
SP0	Reference asphalt with no polymer	Yes	0
SP-PC40	Ref mix with 40% of PC replacement of bitumen	Yes	40% of portland cement

## 2.6 Asphalt mixture preparation

For this research there were two different types of mix designs: control mixtures without any fillers, mixtures containing LS filler as 40% replacement of binder by volume, and mixtures containing PC reactive powder as 40% replacement of binder by volume. The control mixtures used a total binder content of 6.1%. The mixtures containing LS filler and PC reactive powder had 40% (by volume) bitumen replacement which means that the total added binder content was reduced to 3.5%. The aggregate quantities were constant throughout all the mixtures to allow for a more even comparison between the three different mixture types. All mixtures were modified with Evotherm additive to create a warm-mix asphalt (WMA). The total mass of the mixtures as well as the added binder mass were calculated using the equations below:

$$\text{Total Asphalt Mixture Mass} = \frac{\text{Aggregate Mass}}{1 - P_b} \quad \text{Eq. 2.1}$$

$$\text{Added Binder Mass } (P_b) = \left[ \frac{\text{Aggregate Mass}}{1 - P_b} \right] - \text{Aggregate Mass} \quad \text{Eq. 2.2}$$

Where:

*Aggregate Mass* = Total mass of aggregates (4.7 kg or 1.5 kg);

$P_b$  = added binder content in %.

The total mass of the mixture and the amount of binder content for a batch are dependent on the particular test for which the mixtures were utilized. To determine the bulk specific gravity ( $G_{mb}$ ) using ASTM D6857/D6857M-11 procedure, the mass of all the aggregates required for compacting was 4.7 kg. In contrast, the batch used to determine the maximum specific gravity ( $G_{mm}$ ) according to ASTM D6752/D6752M-11 only necessitated 1.5 kg of aggregates.

The asphalt mixing process was carried out following the guidelines of AASHTO T312-12. First, all materials were weighed, and the aggregates were thoroughly mixed before being heated to a specific temperature in an oven. For mixtures that included fillers, the LS and PC were added to the mixed aggregates before heating. The mixing occurred at 120 C, and compaction was done at 115 C, except for polymer modified PG64-10-based mixtures which required higher temperatures of 150 C for mixing and 145 C for compaction due to high viscosity of bitumen. The compaction temperature was kept lower than the mixing temperature to replicate the temperature drop that happens during the transportation in real-world applications. The required amount of asphalt binder was also warmed to the mixing temperature. Once all materials reached the mixing temperature, the aggregates were placed in a hot mixing bucket, and the appropriate amount of asphalt binder was added to achieve the desired batch weight. The mixing bucket was then put in the Humboldt asphalt mixer and mixed for 3 minutes at 60 RPMs. After mixing, it was observed that all the aggregates were evenly coated. It is worth mentioning that although fillers were used as a substitute for asphalt binder, they were processed as aggregates in the mixing process.

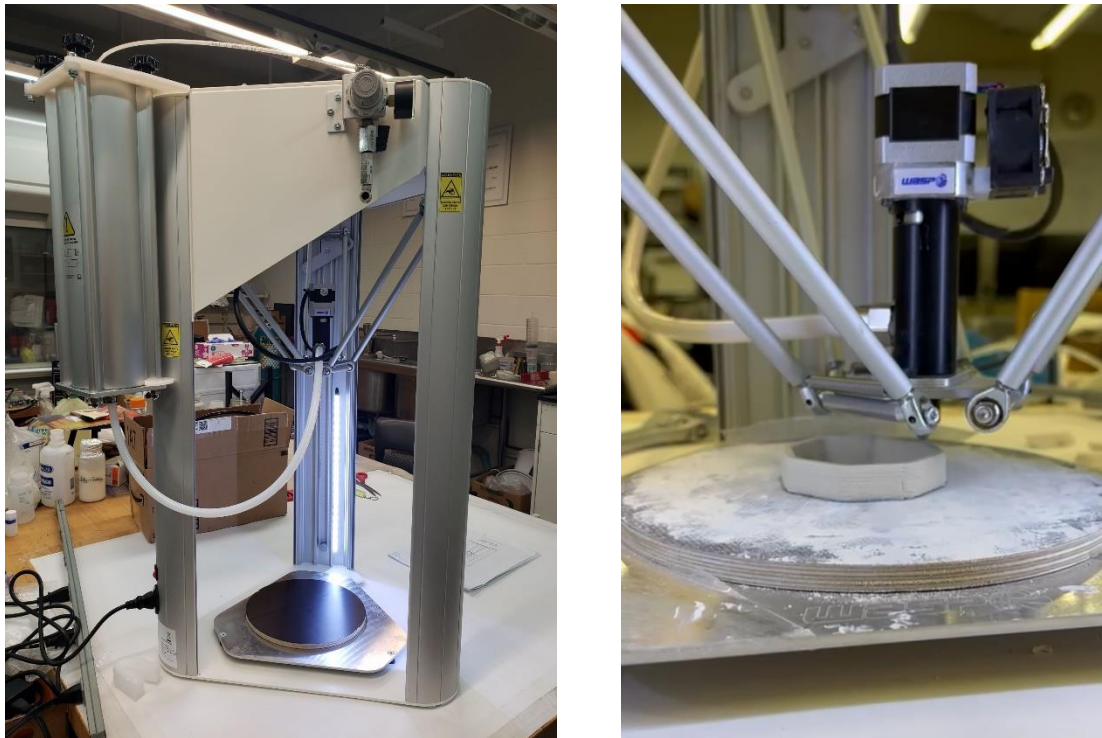
For preparation of mastics, the following is the proposed procedure for blending the powders with asphalt binder:

1. Preheat powder in oven at  $135 \pm 5^{\circ}\text{C}$ ;
2. Heat asphalt at  $135 \pm 5^{\circ}\text{C}$ ;
3. Place empty quarter of a gallon paint can on top of insulating plywood on the scale to prevent heat from reaching the platen;
4. Zero the scale;

5. Pour target mass of asphalt into the can (recommended 500 grams in a quarter of a gallon can);
6. Measure Evotherm additive using a dropper (skip this step for HMA);
7. Determine the mass of filler required based on the mass of asphalt according to the target filler concentration by mass;
8. Put the can of asphalt in the heat mantel and adjust temperature to  $135 \pm 5^{\circ}\text{C}$ ;
9. Heat asphalt in the mantel for 10 minutes;
10. Insert the mechanical stirrer in a such way that it is located at the bottom third of the can depth. (Use dispersing stirrer to prevent filler agglomeration);
11. Start mechanical stirrer at 1300 revolutions per minute;
12. Put an aluminum foil over the can and make a hole to allow space for adding filler into the can and make sure to prevent dust going into air;
13. Add Evotherm additive to asphalt binder (skip this step for HMA);
14. Add filler in small increments while stirring, targeting mixing time of 30 minutes;
15. After all the filler is added, continue stirring for five minutes; This makes the total stirring time to be 30 minutes;
16. After blending, the mix will be poured into smaller ointment tins. (50 grams each in 8 oz ointment tins);
17. Cover the container tins and store at room temperature for future testing.

## 2.7 3D printing procedure

A Delta WASP 2040 Clay 3D printer was used to extrude clay and cement pastes into the desired geometry defined by a g-code from an STL file generated by CURA slicing software. The paste is deposited onto a circular flat base plate by a rotating screw mechanism, where the 'ink' is pressure fed by a pneumatic system hooked to a vertical storage tank beside the printer. Different nozzle sizes are available for the system, but for the reported experiments 2mm diameter nozzle was deemed to be best fit for printing. The printer uses a pneumatic system with 4 psi of pressure to push the ink into a tube feeding and into the rotating screw extruding module. The speed of the extruder is dictated by the g-code generated by the slicing software CURA.

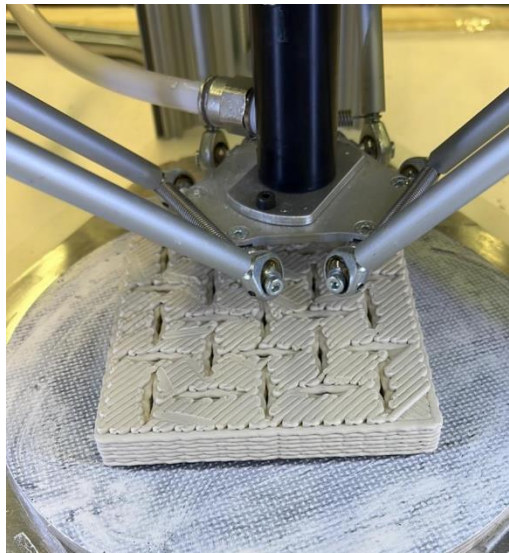


**Figure 2-3 WASP printer, and extruding clay (right)**

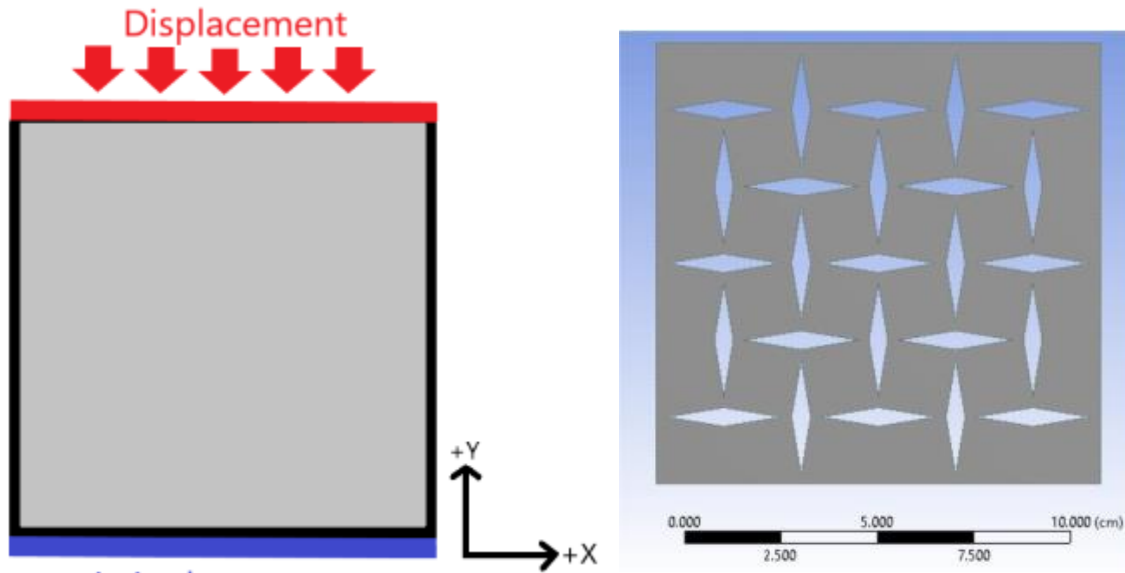
## 2.8 Extrusion of clay into auxetic geometry

The clay ink was prepared to the proportions of 75% porcelain clay and, 25% water. Using the following procedure:

1. Lay the mixture on a clean surface;
2. Add water (25% water for 75% clay mixture) and mix until total absorption. If the material feels too dry add some water (water can be added up to 30% depend on the nozzle size, the smaller nozzle size needs mor fluidity and more water);
3. Subdivide the mixture in balls to be loaded inside the internal of the aluminum tank (if the tank is big enough to pour the material easily, its not needed to subdivide to small portions);
4. Increase the pressure up to 4 psi and start printing.



**Figure 2-4 Printing of auxetic geometry**



**Figure 2-5 Schematic of auxetic shapes**

In order to understand the effect of geometry and printing parameters, clay was used to print the auxetic geometry and then tested to measure the Poisson's ratio, figure 2-5. Using an Instron testing frame, the auxetic geometry was loaded under the Y compressive force direction until failure. An extensometer was attached to a bracket that was glued onto a portion of the piece. This was to ensure the accurate measurement of the lateral changes in the x-direction and record the differences.

### 3. EXPERIMENTAL RESULTS AND DISCUSSION

#### 3.1 Low temperature testing of asphalt concrete

Asphalt binder and asphalt concrete are designed to resist low temperature cracking as verified with SCB test. For the SCB test, the specimens were saw cut to disks with a thickness of  $25.4 \pm 2.0$  mm. A centrally located notch measuring  $1.5 \pm 0.3$  mm by  $10.0 \pm 0.5$  mm was cut at the base of each specimen as demonstrated by the Figure 3-1.

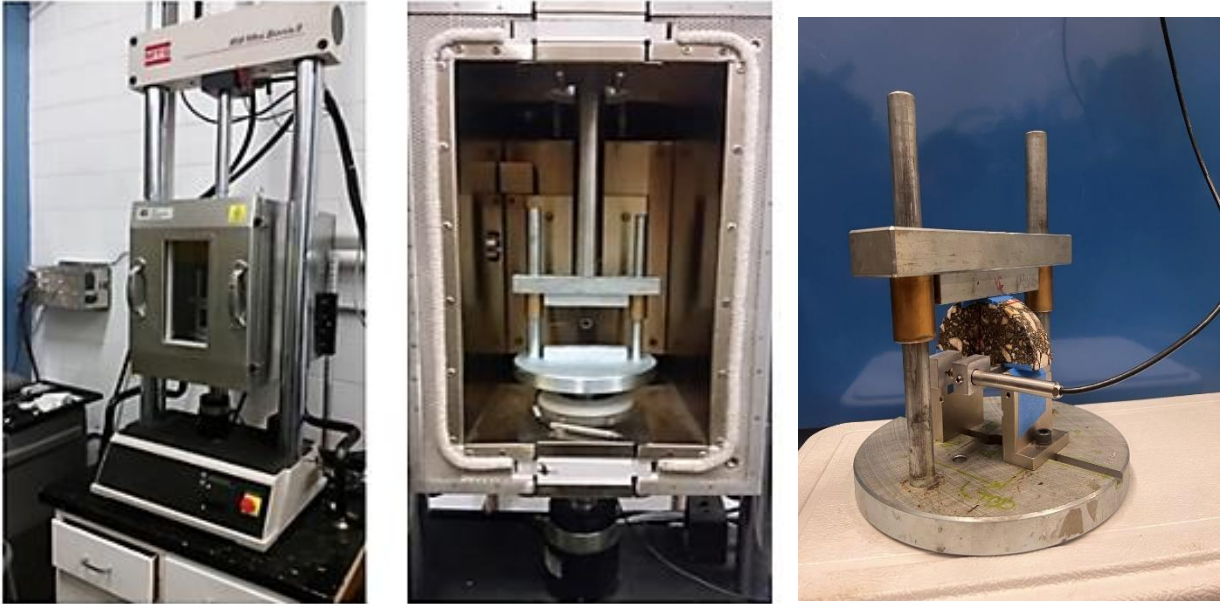


**Figure 3-1:** Probe used to monitor the temperature of the samples and test specimen in the MTS frame.

For N-series of compositions the temperature for the test was set at  $-18 \pm 1^\circ\text{C}$ , and for the S-series, test temperature was  $0^\circ\text{C}$ , the loading rate was  $0.03 \text{ mm/min}$ . The specimens were conditioned for  $4 \pm 0.5$  hours at  $-23 \pm 1^\circ\text{C}$  before testing, and duplicates were tested. To enable a direct comparison of all investigated specimens, the same loading condition were used during the experiment.

The MTS 858 Mini Bionix II loading frame is a type of testing equipment commonly used to perform the mechanical testing on materials. It is designed to apply different types of loads to a sample, such as compression, tension, and fatigue loading. The MTS 651 Environmental Chamber is an accessory that is used to control the temperature and humidity of the testing environment. The chamber is connected to a temperature controller that ensures the temperature inside the chamber is accurate and stable throughout the testing process. The MTS data acquisition software was used to collect and analyze data from the testing equipment. It allowed to monitor the performance of the material being tested in real-time, and to generate graphs and reports based on the collected data.

The SCB test was conducted an updated 3-point testing frame. The test was concluded when the load fell below  $0.5 \text{ kN}$ . This machine was used to assess both the vertical load and the vertical load-line displacement. The LVDT was attached to the specimen to record crack mouth opening displacement (CMOD).

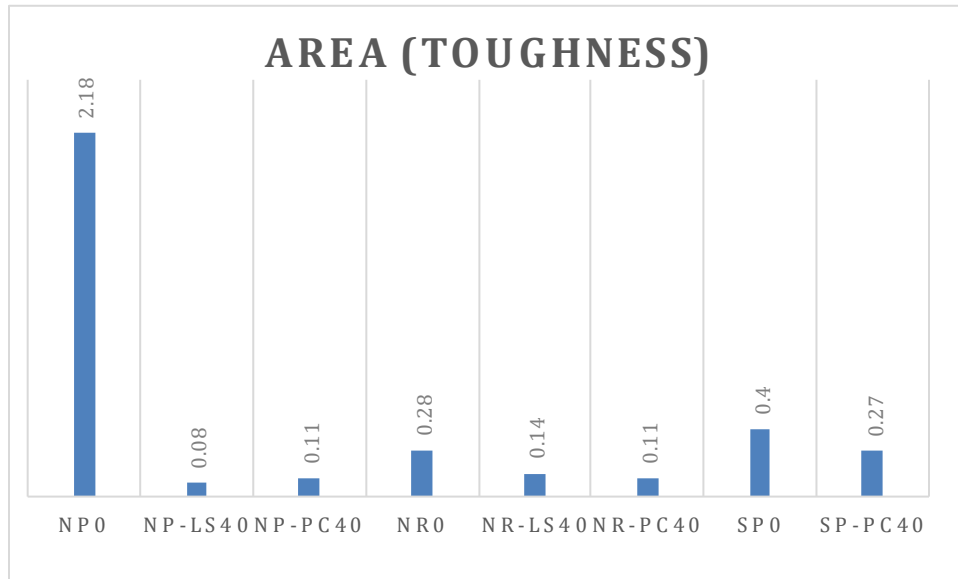


**Figure 3-2: MTS environmental chamber with testing frame**

The experimental results for low-temperature performance of hybrid asphalt concrete are reported in table 3-1.

**Table 3-1: Low-Temperature Results:**

<b>Sample ID</b>	<b>MAX LOAD (kN)</b>	<b>MAX DISPLACEMENT T (mm)</b>	<b>STIFFNESS (kJ/mm)</b>
NP0-1	2.89	1.40	3.45
NP0-2	3.78	0.65	2.18
NP-LS40-1	2.16	0.11	0.07
NP-LS40-2	2.06	0.07	0.09
NP-PC40-1	1.43	0.10	0.12
NP-PC40-2	1.56	0.12	0.11
NR0-1	3.24	0.11	0.28
NR0-2	3.38	0.12	0.29
NR-LS40-1	3.55	0.08	0.15
NR-LS40-2	2.95	0.07	0.14
NR-PC40-1	2.04	0.07	0.11
NR-PC40-2	1.84	0.10	0.11
SP0-1	2.52	0.50	0.43
SP0-2	1.70	0.50	0.38
SP-PC40-1	1.40	0.50	0.27
SP-PC40-2	1.35	0.50	0.26



**Figure 3-3:** Plot of toughness in samples

Based on experimental results, the NP0 had the most maximal load and, overall, polymer modified samples had a better displacement and reduced maximal load. Polymer admixtures can help to reduce cracking in concrete at low temperatures by increasing its flexural strength and toughness. This can help prevent the damage from thermal and mechanical stresses. But when bitumen is replaced with large volume of powder material such as PC or LS, a reduction at displacement at failure and formation of material take place.

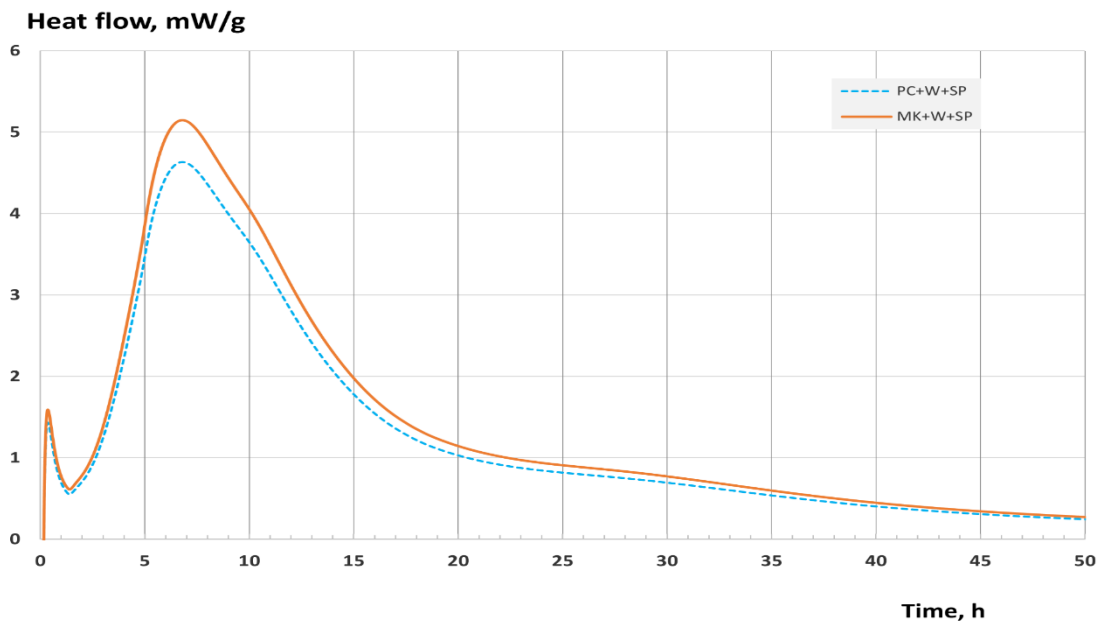
### **3.2 Effect of mixing order on cement paste with metakaolin and cement**

Cement paste is an essential component of concrete, which is the most widely used construction material. Cement paste is typically made from a mixture of portland cement and water, and is capable to hydrate forming a solid and durable material. However, the properties of cement paste can be improved by adding supplementary cementitious materials, such as Metakaolin (MK). Metakaolin is a pozzolanic material that reacts with calcium hydroxide in cement paste to form additional cementitious compounds. This process improves the workability of cement paste, the strength, durability, and making it an attractive solution for construction applications. In this experiment, the effect of mixing order on cement paste with metakaolin and cement was investigated.

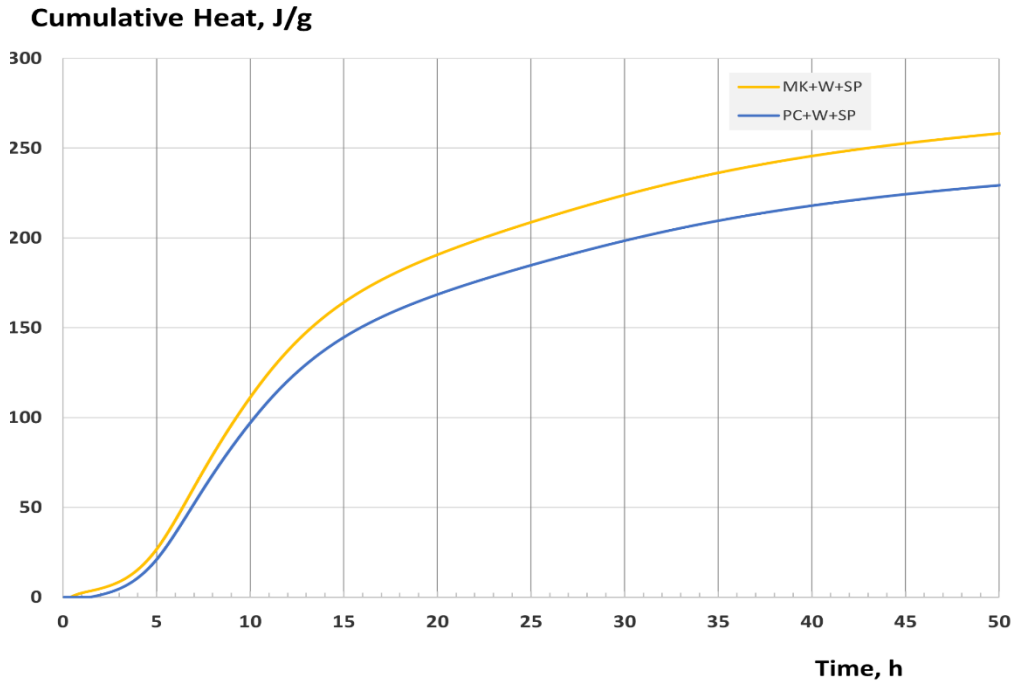
Two different mixing methods were evaluated to prepare cement-based ink for 3D printing. The materials used in this experiment were portland cement (Type I/II) and metakaolin. The water-to-cementitious materials ratio (w/cm) was used at 0.45 for both experiments. The first mixing procedure included portland cement and water (water plus polymer solution) plus superplasticizer first and then the additional cementitious material (MK). The second procedure included mixing of the MK and water plus superplasticizer first then added the portland cement. Both compositions were evaluated for the heat of hydration and compressive strength testing at the age of 1, 7 day, and 28 days. This experiment was intended to enable the selection of the best mixing procedure.

The heat of hydration (HoH) experiment was conducted using an isothermal calorimeter, to understand the effects of mixing procedure of metakaolin powder on the hydration of polymer modified cement paste. Figure 3.1 reports on the main hydration peak of  $C_3S$  which increased along with the total cumulative heat when metakaolin was incorporated into the mix. This is an

indication of accelerated hydration, affecting the strength development which is important for digital construction; The acceleration of  $C_3S$  hydration can be explained by a better dispersion of metakaolin. When second mixing procedure was used the total hydration heat after 50 hours of hydration was 13% higher.

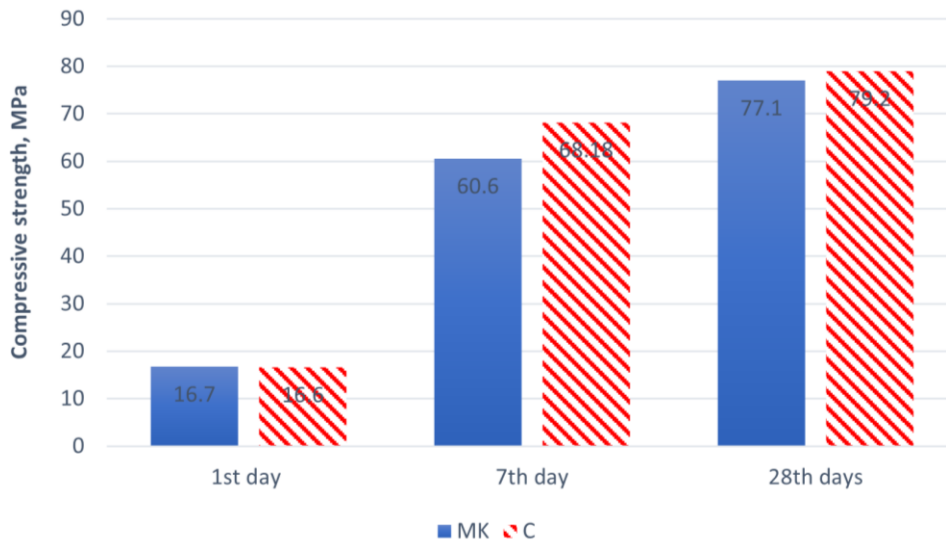


**Figure 3-4:** Heat evolution of cement pastes with MK



**Figure 3-5:** Total Cumulative Heat of cement paste inks

The early strength of cement pastes was measured using a compressive test machine from ELE International. The 2-in specimens were loaded at a rate of 1.4 kN/sec until failure.



**Figure 3-6:** Compressive strength of cement paste with MK

### **3.3 2D printing of cement mortar inks**

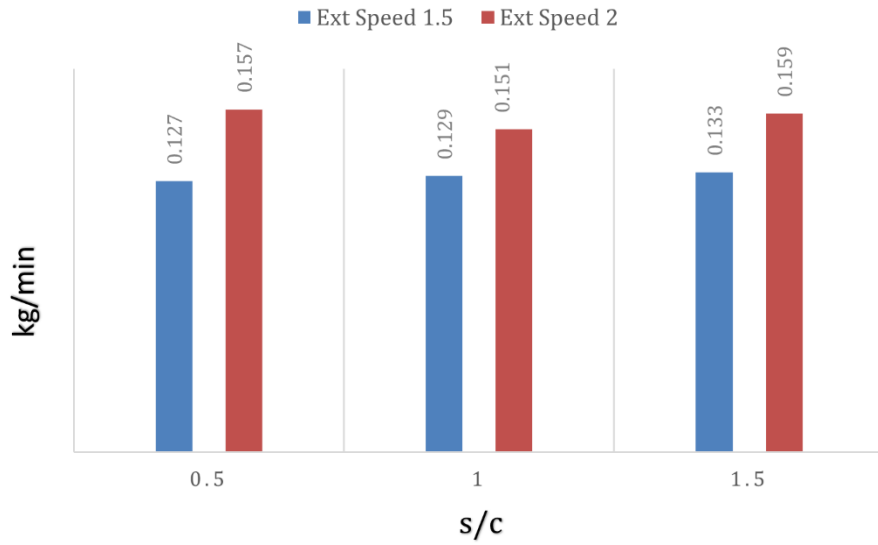
The 2D printing experiment was focused on the evaluation of printability of cement-based materials using a custom 2D printer frame. The goal of the experiment was to find the best printing parameters including printing speed, different nozzle sizes, and different ink compositions.

The parameters which are of importance for 2D/3D printing are fresh density, flowability, and extruding speed since each of these parameters can directly affect the printing process.

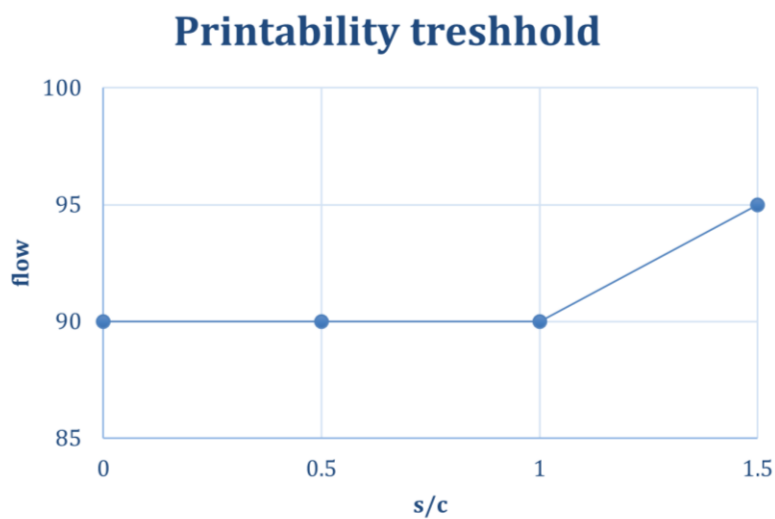
The extruding speed is the speed at which the cement-based material is extruded through the nozzle and deposited onto a moving platform which is also synchronized to provide a continuous cast. This parameter is important for printing because it affects the accuracy and consistency of the printed object and its ability to adhere to the previous layer. Different extruding and platform speeds were tested to determine the best parameters for the setup.

Fresh density is the density of the cement-based material tested immediately after it is mixed (and before it sets). This parameter is important for printing because it enables volume-weight calculation which helps to design the material and correlated to the extrusion through the nozzle. The ink mixes can be tested at varying fresh densities and the differences between the compositions can help to determine suitable mixtures for printing.

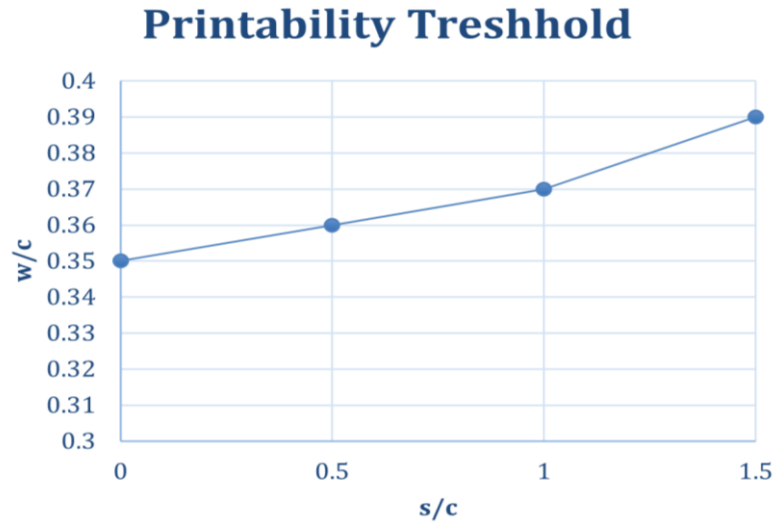
The graph below indicates that all the printable mixes with different S/C (sand to cementitious material) have almost the same extrusion rate over density parameters.



**Figure 3-7:** The effect of sand to cement ratio on extrusion (kg/min)



**Figure 3-8:** The effect of sand to cement ratio on printability of mortars



**Figure 3-9:** Effect of S/C and W/C on production of printable ink compositions.

The flowability of mortar is a measure of how easily the cement-based material flows under dynamic load. This parameter is important for printing because it can affect the extrusion process, the process of space-filling between the layers, and smoothness quality of the final printed object. A flow table was used to test the flowability of the different ink compositions with varying fresh densities.

**Table 3-2:** Printability with different flows

Sample	s/c	w/c	Flow (%)	Printability	Fresh density (kg/lit)
0.35-0.5	0.5	0.35	85	no	2.04
0.36-0.5	0.5	0.36	90	yes	2.02
0.37-1	1	0.37	90	yes	2.09
0.38-1.5	1.5	0.38	88	no	2.17
0.39-1.5	1.5	0.39	95	yes	2.14

It was proved that the flow is the most important parameter for an ink printability. It was determined that as long as the mix has a minimum flow of 90 to 95% it could be printable.

Overall, the experiment to find the optimal printing parameters for cement-based ink materials was calculated with the objective to produce high-quality printable objects. By testing different compositions with varying proportions fresh densities, flowability, and extruding speeds, the best combination of parameters can be determined.

When cement paste hardens under ambient conditions (temperature  $23 \pm 2$  °C and RH 50%), it undergoes a process called drying shrinkage, which can cause it to crack and potential failure over time. Drying shrinkage occurs because the water from the cement matrix evaporates, causing the volume of the paste to decrease. This decrease in volume can create the internal stresses that lead to cracking, figure 3-10. In real applications, the drying shrinkage is accompanied with chemical autogenous shrinkage.



**Figure 3-10:** Cracks of initial cement prints after 4 hours of lab exposure

The best approach to prevent drying shrinkage and cracking is to use aggregates/sand to the inks. Sand is a granular material that can help cement particle and paste to form the structure of the cast inks within the spaces between the sand grains, reducing the overall volume of the mixture and limiting the amount of drying shrinkage that occurs. By increasing the sand to cement ratio in the mix (to at least 1.5), the drying shrinkage can be controlled resulting in improved the overall strength and durability of 2D/3D printed cement-based material.

Here, sand is effective in preventing shrinkage due to its granular nature. Sand particles are larger than cement particles, and when mixed together due to interlocking and a formation a solid framework that resists the shrinkage is realized. This 3D framework helps to distribute the stresses that occur during material deposition and also during drying limiting the volumetric deformations, stresses, and formation of internal cracks.

It is important to note that the optimal sand to cement ratio can vary depending on the specific application and the properties of the materials being used. By testing different ratios and adjusting other parameters such as volume of cement paste as needed, the mixture can be optimized based on the specific needs improving the printability and durability of the cement-based material.

When adding sand to a cement-based mix, several factors can influence the optimal nozzle size and other printing parameters. Sand particles are typically much larger than cement particles, which can affect the flow properties of the mixture. If the sand particles are too large relative to the nozzle size, clogging of the nozzle or cause uneven extrusion. This can result in defects in the printed structure and may require a larger nozzle size to accommodate the larger particles.

It is well known that cement-based materials shrinking under low humidity conditions (< 70%), which can lead to cracking and other defects in the printed structure. By adding sand to the mix, you can reduce shrinkage and improve the overall strength and durability of the material.

Adding sand to the mix results in a larger particle size distribution and changes the rheology of the mixture. As a result, nozzle size is needed to be adjusted to ensure the mixture flow and even deposition. By testing different nozzle sizes and adjusting other parameters including the compositions, such as using viscosity modifiers such as PVA or nano silica as needed, 2D/3D printing process can be optimized for specific mixtures and applications.

### **3.4 Effect of nano-silica on printability**

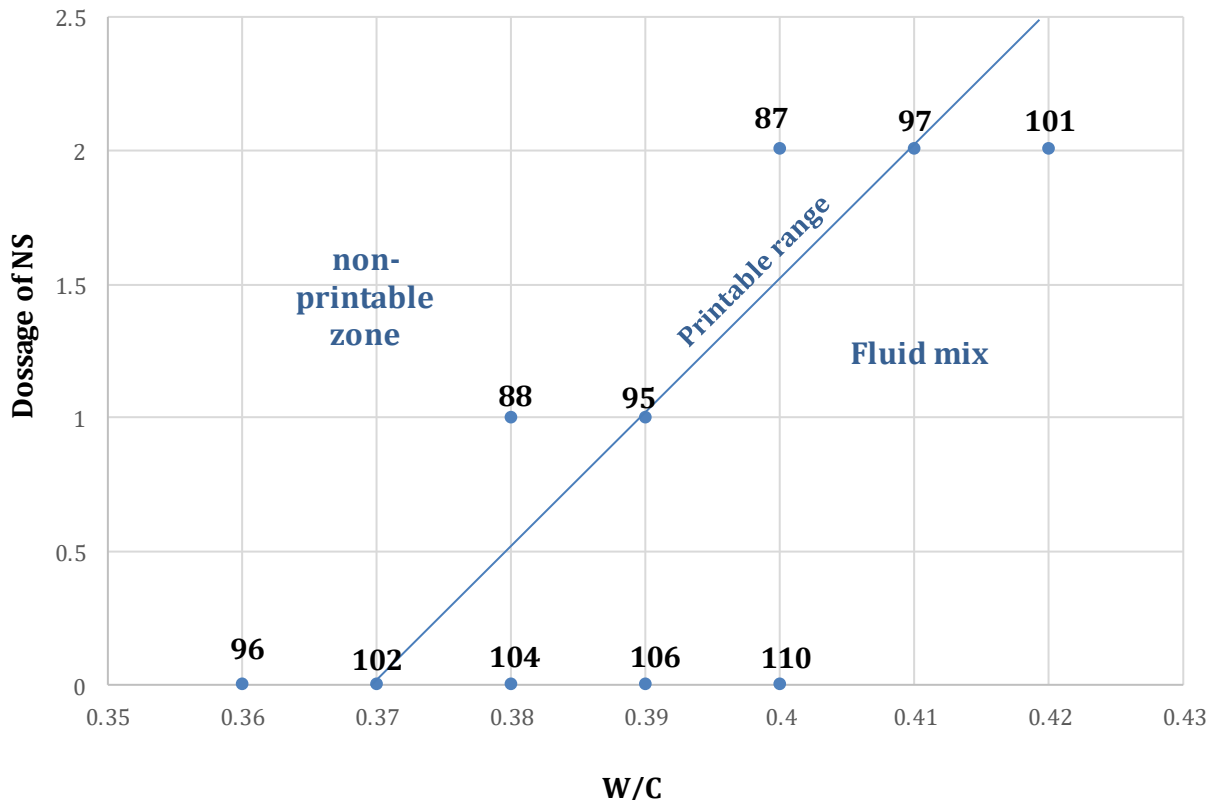
Nano-silica is a form of colloidal amorphous silica dioxide that has particles with a diameter of less than 100 nanometers. When added to cement paste, nano-silica can improve fresh properties, strength, durability, and other performance characteristics.

The main effect of nano-silica on the cement pastes is related to filling in the voids between the cement particles, which can make the paste and matrix denser and reduce the porosity of the cast. This, in turn, can increase the strength and durability of the cement-based compositions.

In addition, nano-silica can also interact with the calcium hydroxide (CH) gel forming during the hydration process of cement, leading to the formation of smaller and more uniform C-S-H particles. This can result in a more dense and less porous microstructure, enhancing the mechanical properties of the cement-based materials.

As for the flow of the mix, adding nano-silica can increase the viscosity and reduce its workability (and flowability). The specific effect of nano-silica on the flow of the mix is viscosity modification and adding the cohesiveness which depends on various factors such as the type of cement used, the dosage of nano-silica, and the water to cement ratio.

The figure 3-11 indicates the effect of different percentages of NS (0, 1, and 2 %) on w/c required for extrudability; also, on the graph each point specifies the flow of the mix. Based on the results of the experiments, the best compositions with NS which are printable is marked by the blue line, indicating the ink mixes which are printable. Therefore, all the compositions at the left side of the line, are not printable because of the low flowability. However, the mixes at the right side are printable, however they are not the best mixes for printing, because of high fluidity and lack of stability, these inks do not possess sufficient adhesive strength to maintain their structural (shape) integrity of the printed layers.



**Figure 3-11:** Effect of Nano-Silica on W/C and printability

#### **4. CONCLUSIONS**

It was found that nano-silica can improve the fresh properties, strength, durability, and other performance characteristics of cement paste by filling in voids, and creating a more dense microstructure due to pozzolanic effects. However, adding nano-silica can also increase the viscosity and reduce workability, which affects the flow of the mix. However, the use of nano-silica in 2D/3D printing cement-based ink is necessary to have a better cohesiveness, the optimal composition for printing with nano-silica is determined by the balance between extrudability and stability, as mixes with low or high flowability are not suitable for printing.

Additionally, I found that polymer modified asphalts had a better performance than the reference samples. The warm mix asphalt compositions with portland cement or limestone replacement had lower toughness. Polymer modified asphalts show better performance due to the addition of polymers to the asphalt mix. Polymers improve the mechanical properties of the asphalt mix, including its stiffness, strength, and durability. Polymer can also improve the resistance to cracking and deformation, which is particularly important in areas with heavy traffic or extreme temperature fluctuations. Additionally, polymer modification can improve the adhesion between the asphalt binder and the aggregate particles, which leads to better overall performance of the asphalt mix.

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