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# The development of a vermiculite based 3D printable fire-resistant concrete and an assessment of its fresh state and mechanical properties

**Kyle B. Stebbing and Algurnon S. Van Rooyen**

Stellenbosch University, Department of Civil Engineering, South Africa

**Abstract**

3D Printed Concrete (3DPC) is in the preliminary stages of implementation and is considered a large part of the $4^{th}$ industrial revolution (4IR). Currently there is sparse 3DPC research focusing on insulation properties such as sound and heat. This research focuses on the development of a lightweight vermiculite-based fire-resistant 3DPC. Vermiculite is a mica-like mineral consisting of shiny porous flakes. The flaky structure results in vermiculite having high lubricating characteristics, allowing it to be used as a fire-resistant material. Various grades of vermiculite were tested resulting in the use of micron grade vermiculite for the 3DPC trial mixes. Fresh state tests were performed with the best mix having a mini-slump of 170mm and a dynamic shear reduction factor of 0.72. Mechanical tests resulted in a compressive strength of 4.88 MPa and a tensile strength of 0.56 MPa tested at 7 days.

**Keywords:** 3DPC, Expanded Vermiculite, 4IR, Fire-resistant

## 1. INTRODUCTION

The world is on the precipice of the $4^{th}$ industrial revolution (4IR). The goal of 4IR is to create adaptive networks through the digitization of industrial processes. Implementing 4IR technology creates an environment in which mechanized automation operates and shares information without the need for human interaction, improving efficiency [1]. Although the construction industry would greatly benefit, it is still slow to implement these technologies. One of these 4IR technologies showing great potential is 3D printed concrete (3DPC) which offers benefits such as a substantial reduction in construction time, waste minimisation, lowered cost of elements compared to traditional construction methods and environmentally friendlier when recycled construction materials are used [2]. 3DPC technology holds the advantage over traditional concrete by not requiring formwork during construction. However, it requires complex rheological parameters making it notoriously difficult to develop.

Rheological parameters are paramount to successfully 3DPC and requires that thixotropy, constructability, printability and workability be considered. 3DPC mixes need to optimally balance these requirements to achieve success [3]. Thixotropy is the most important parameter and materials exhibiting thixotropy have the consistency of dough at rest and flows readily when energy is applied through perturbation, thus having a distinct difference between dynamic and static yield stress. For 3D printing, the bottom layer requires a sufficiently developed static yield stress to maintain its shape and carry the weight of subsequent layers printed on top. The compressive stress in the bottom layer is directly proportional to the number and height of successive layers. When the compressive stress in the bottom layer exceeds the uniaxial yield stress, the layer will deform which leads to collapse [4]. Yield stress also directly relates to the pumping of concrete as pressure is applied to the concrete to facilitate flow. Only once the static yield stress is exceeded does the material start to flow, thereafter the dynamic yield stress is maintained. A low dynamic stress places less strain on the motor of the pump, while also reducing the chance of water separating from the mix. A high static yield stress results in a stiff concrete with better buildability, as it requires more energy to flow. By determining the dynamic shear reduction factor, the ratio of the average yield stress over the peak yield stress, an indication of the thixotropic nature of the concrete can be achieved. In general, the higher the factor the more thixotropic the material. Due to these factors, concrete with an optimal balance between high static and low dynamic yield stresses is required for effective 3DPC [3]. An indication of the printability of concrete can be found using the mini-slump test where researchers have reported successful 3D printable mixes in the slump range of 150 to 190 mm, dependent on the mix constituents [8].

Researchers developing 3DPC found that replacing large aggregates with finer material such as sands, clays, and lightweight aggregates (LWA) provided better rheological properties but required higher cement volumes, increasing cost. The use of finer material lead to excessive shrinkage and cracking, requiring shrinkage reducing admixtures and fibre reinforcement to be incorporated and the higher cost of cement is offset through the use of supplemental cementitious materials (SCM's) such as fly ash, silica fume limestone filler, and blast furnace slag are used [2].

To design a fire-resistant concrete the use of non-combustible thermal insulators is key. The primary focus is to obtain higher volumes of air-solid interfaces, low heat transfer and low radiation at high temperatures, which can be achieved by using porous materials [5]. LWA are porous in nature, therefore, their use could improve the thermomechanical behaviour of cementitious materials [6]. This research focuses on expanded vermiculite (EV) which is a mica-like mineral that consists of shiny porous flakes and is produced by heating raw vermiculite to 1000°C. The flaky structure results in high lubricating characteristics, allowing it to be used as a fire-resistant material and a lightweight filler for heat insulation [7]. EV's notable properties are a low thermal conductivity (0.04 – 0.14 W/m K), good sound absorption coefficient (0.7 – 0.8 at 1kHz), high melting point (1240 – 1430°C), non-toxic and chemically inert [7]. A study conducted on the high-temperature performance of self-compacting mortars (ScM) containing EV concluded that introducing EV showed a reduced loss of compressive strength at higher temperatures compared to the control ScM [5]. The use of EV in concrete increases porosity and sound insulation while decreasing the density, thermal conductivity, and mechanical strength [7]. However, the primary challenge of using EV in the mix is an increased water absorption rate due to the porous nature of EV, which affects the workability, open time and shrinkage of the concrete.

EV sees study as an addition to various concretes and shows a versatility in potential uses, however, there is no research coupling EV with 3DPC. Stellenbosch University (SU) developed a 3DPC mix that conforms to the rheological requirements necessary for 3DPC [3]. This paper aims to extend the existing body of knowledge by developing a lightweight 3D printable EV concrete, based on the SU 3DPC mix, with potential practical applications in the fire safety sector. In this investigation, the absorption rates of the different grades of EV are tested. Mini-slump flow tests are conducted on mixes with unsaturated-, saturated-, and silane treated EV, to evaluate slump loss and find a solution to the absorption issue. Rheology, and early age mechanical tests (tensile- and compressive strength) are performed on the most viable mixes.

## 2. EXPERIMENTAL DESIGN

### 2.1 Experimental Programme Rationale

Materials used to fire-rate buildings are expensive. EV is currently used in various forms to protect against fire due to its low thermal conductivity. EV has been used in bricks, however, there is no research coupling EV with 3DPC. Developing a 3DPC containing EV could save costs used on protecting buildings from fire post-construction, by pre-fabricating or in-situ printing fire-rated EV concrete.

### 2.2 Mix Design

#### 2.2.1 Materials

EV mixes were designed using CEM II 52.5N Portland cement with relative density of 3.14 as binder and fly ash (FA) and silica fume (SF), both with a relative density of 2.2 as cement extenders. For filler, silica sand with relative density 2.64 and maximum particle size of 0.3 mm and graded EV is used. Potable tap water is used for mixing and the superplasticiser (SP) CHRYSO®Fluid Premia 310 is used.

#### 2.2.2 Mix design procedure

In this investigation the EV concrete mix designs are done based on conservation of mass and setting the total volume as 1000 litres. Table 1 gives the concrete mix compositions used in this investigation. Mix 1 enables a comparison between saturated and unsaturated EV, mix 2 determines the effectiveness of silane treated EV, mix 3 is the standard SU 3DPC mix [2] and mix 4 and 5 are based on mix 3.

Table 1: EV concrete mix compositions in kg per 1000 L

| Mix | w/c | Cement | FA | SF | Sand | EV | Water | SP |
|---|---|---|---|---|---|---|---|---|
| **1** | 0.7 | 357 | - | - | 771 | 27.7 | 250 | - |
| **2** | 0.5 | 412 | - | - | 165 | 82.4 | 206 | - |
| **3** | 0.45 | 579 | 165 | 83 | 1167 | - | 261 | 12.24 |
| **4** | 0.57 | 546 | 165 | 83 | 584 | 142 | 313 | 25.6 |
| **5** | 0.625 | 474 | 165 | 83 | 467 | 170 | 296 | 25.6 |

### 2.3 Experimental Testing

#### 2.3.1 Sieve test

Sieve tests were performed on four different grades of EV according to BS EN 993 standards. The tests are performed using sieves of various sizes, a catching bowl and a vibratory machine. The sieves aperture sizes are 0.075 mm, 0.15 mm, 0.3 mm, 0.6 mm, 1.18 mm, 2.36 mm, and 4.75 mm. The sieves are weighed and stacked from smallest to largest aperture in the vibratory machine, 100g of vermiculite is placed on the 4.75 mm sieve and the lid is placed on top. The vibratory machine is turned on for 10 minutes to allow enough time for the vermiculite to be accurately sieved. The sieves are weighed to determine the weight of the vermiculite retained.

### 2.3.2 EV absorption rates

Absorption rate tests of the fine-, superfine-, and micron grade EV were performed by submersing 400g of EV in water for 24hrs and measuring the weight gain. The increase in mass of the sample was recorded at intervals during the 24hr period. It was determined that the EV's absorptive properties would limit its use in 3DPC. To solve the absorption problem, trial mixes were performed with unsaturated, saturated and treated EV. The saturated EV was soaked in water and the treated EV was coated with a hydrophobic chemical. Relative densities were calculated from the mix designs as the pycnometer test proved unsuitable and gave inaccurate results.

### 2.3.2 Fresh state tests

The fresh state tests performed were mini-slump and rheology. The mini-slump is determined by placing a cone on a hydraulic turntable and filling the cone with the mix. The cone is removed and the handle is turned, raising and dropping the turntable. This is repeated 15 times after which the average diameter of the concrete is measured, indicating the flowability of the mix. This test is performed over 30 minutes at 15 minute intervals. Thereafter the rheology is tested using the German ICAR Rheometer. The rheometer is set up by inserting the vane and calibrating the rheometer. The bucket is filled with 20L of concrete, the rheometer is inserted and stress growth tests are performed. The test is run for 60s after which a rest period of 10s is observed and the test is then repeated. This process is repeated for rest periods of 30s, 60s, 90s, 120s, 10 mins, 20 mins and 30 mins. The rheometer test outputs applied torque readings which are used to determine the yield stress given as Equation 2.1.

$$T = \frac{\pi D^3}{2}\left(\frac{H}{D} + \frac{1}{3}\right)\tau \qquad \text{Equation 2.1}$$

Where T is the applied torque (Nm), D is the vane diameter (m), H is the vane height (m) and $\tau$ is the yield stress (Pa).

### 2.3.3 Mechanical tests

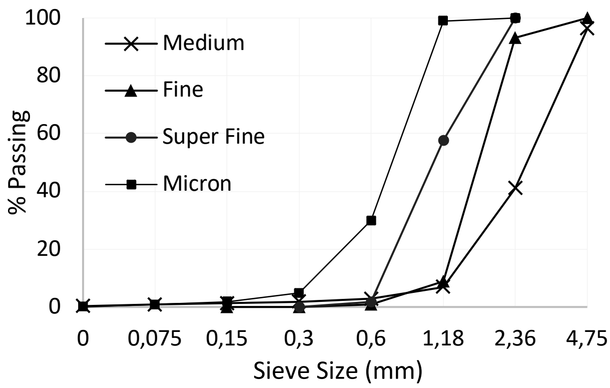
To test the mechanical properties of the mix, the concrete must be in a hardened state. This is achieved by casting the concrete into moulds and allowing it to cure for 24 hours. Once hardened it can be removed and cured for the desired duration. For this investigation 7-day strength was tested, the result of which is roughly 70% of the final strength. To determine the mechanical properties the concrete's compressive and tensile strength was tested using a Zwick/Roell Z250 materials testing machine. The machine applies force to the samples until failure and gives the maximum force they are able to withstand. The compressive stress test requires cubes to be crushed and the tensile strength test requires cylinders to be split. This testing process was done for 3 - 100x100x100mm cubes and 3 cylinders of each mix to obtain an average. The densities of the concrete mixes were calculated by measuring the weight of the samples, dividing it by the volume of the samples and taking the average.

## 3. RESULTS

### 3.1 EV Test Results

The sieve test results of the different grades of EV is presented in Figure 1. The 3D printer nozzle has a 4 mm aperture; therefore, the large grade cannot be used as it has a particle size of 8mm. The medium grade has a smooth S-curve grading which is close to the ideal Fuller curve [3], however, it would need to be sieved to remove the 5% of particles larger than 4.75 mm. The medium grade was removed from consideration as sieving would not be feasible for large scale use. As can be seen, the micron grade has the smoothest grading.

Figure 1: Sieve Analysis of EV Gradings




To achieve an improved S-curve grading for the filler, silica sand was combined with the micron vermiculite. The silica sand grading is shown in Figure 2 and the combined filler grading along with the ideal Fuller grading is shown in Figure 3.

Figure 2: Silica Sand Grading

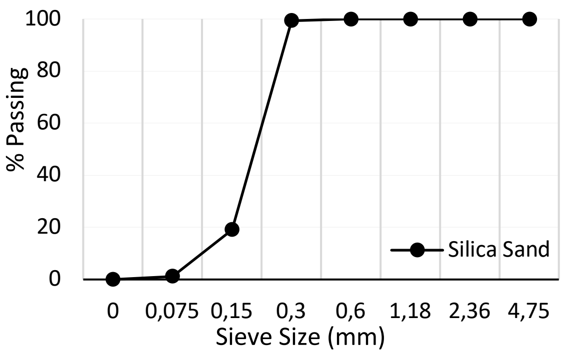
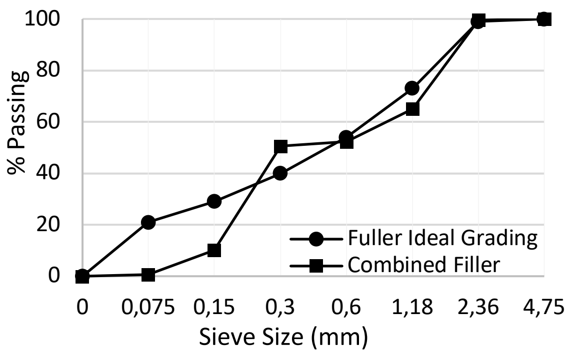
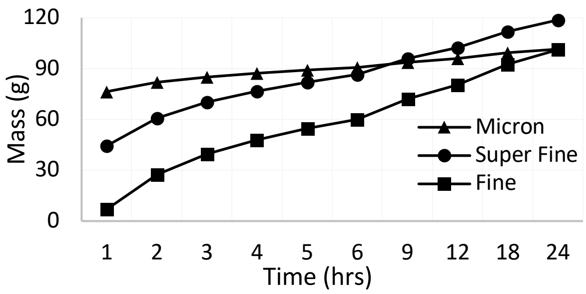



Figure 3: Combined Filler & Fuller




The relative density was determined to be 0.145, 1,11 and 0.644 for the unsaturated, saturated and treated vermiculite. Figure 4 shows the rate of absorption of the micron, superfine, and fine grade of EV. The micron grade absorbs the least amount of water, however, in its initial hour of submersion it soaks up the most water. This is likely due to the particles being smaller thereby allowing the EV to have more surface area in contact with the water. The smaller particles also mean less pores, therefore the micron EV soaks up the least water. Based on these results along with the sieve analysis it was decided that the micron EV shows the most promise for incorporation into a 3DPC mix.

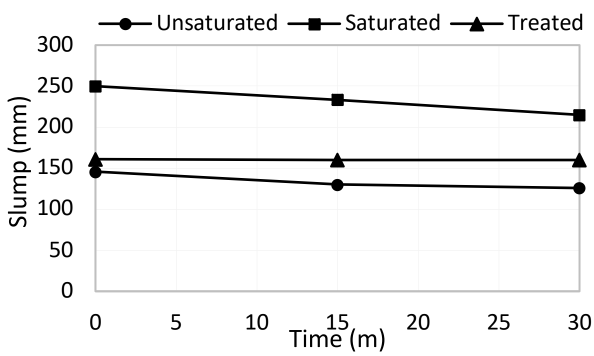
Figure 4: Comparative EV Absorption




### 3.2 Fresh State Test Results

The results from the mini-slump tests, on the unsaturated-, saturated-, and silane treated EV mixes are shown in Figure 5. The additional water of the saturated EV affects workability of the mix. The mini-slump results show a rapid decrease in slump which is not ideal for 3DPC. It should be noted that as the EV becomes saturated it sinks, however, this process is not uniform and as such it is not possible to achieve partially saturated EV in this manner. The process to achieve fully saturated EV takes between 36 and 48 hrs. For these reasons as well as large scale reproducibility it was decided that this method of treatment is ineffective. The unsaturated EV has better mini-slump results, however, there is still a notable decrease in slump over time. Additionally, unsaturated EV soaks up the water, thereby not allowing the cement to hydrate effectively, as well as affecting the workability of the mix. Mix 2 was developed to determine the capabilities of treated EV. As can be seen the mini-slump remains constant which means the mix will not dry out, resulting in a mix that retains workability, consistency and cohesion over time.

Figure 5: Mini-Slump of EV Treatments




Mini-slump and rheology tests were performed on mix 4 and 5. They had a slump of 170 mm and 145 mm respectively. Mix 4 has better mini-slump results. Table 2 shows the stress growth test results. Both mixes demonstrate sufficient difference between their peak and minimum yield stresses to be good candidates for 3D printing. The average dynamic yield stress is taken between the rest periods of 30s and 120s and the peak yield stress is taken at

0s. Therefore the dynamic shear reduction factor is 0.72 and 0.65 for mix 4 and 5 respectively. Mix 4 would be considered to be more thixotropic. Mix 4 has a lower difference between the peak and minimum yield stress, most likely due to the higher slump.

Table 2: Peak and Minimum Yield Stresses Between Rest Periods

| Rest Period (s) | 0 | 10 | 30 | 60 | 90 | 120 |
|---|---|---|---|---|---|---|
| **Mix 4** | | | | | | |
| **Peak Yield Stress (Pa)** | 1218 | 973 | 931 | 881 | 818 | 794 |
| **Min Yield Stress (Pa)** | 877 | 782 | 694 | 639 | 589 | 534 |
| **Mix 5** | | | | | | |
| **Peak Yield Stress (Pa)** | 2589 | 1788 | 1739 | 1695 | 1640 | 1575 |
| **Min Yield Stress (Pa)** | 1663 | 1542 | 1444 | 1384 | 1320 | 1268 |

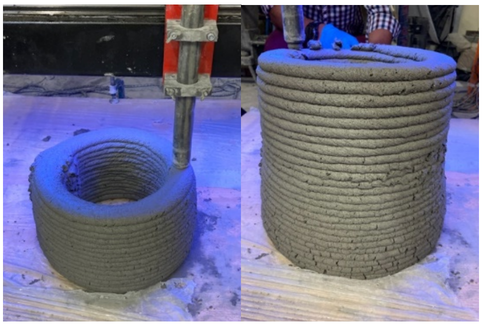
### 3.3 Mechanical Test Results

The mechanical test results of Mix 4 and Mix 5, conducted at an early age of 7 days, is presented in Table 3. Mix 5 has a compressive and tensile stress half of that which mix 4 exhibits, while only containing 10% more EV. Upon demoulding a few samples of Mix 5 crumbled confirming that the mix lacks cohesion and is inferior to mix 4. Mix 4 and 5 have a density of 1466 kg/m³ and 1318 kg/m³ respectively. Mix 4 was printed to test its 3DP capabilities, shown in Figure 6. Significant buildability was demonstrated with 27 - 10mm layers being printed before the process was halted due to bulging of the lower layers. This could be improved by a slight reduction in slump. Furthermore, fibres and a viscosity modifier should be incorporated to improve the cohesion of the mix.

Table 3: Average compressive- ($\sigma_c$) and average tensile stress ($\sigma_T$) in MPa of Mixes 4 and 5

| Description. | Mix 4 $\sigma_c$ | Mix 4 $\sigma_T$ | Mix 5 $\sigma_c$ | Mix 5 $\sigma_T$ |
|---|---|---|---|---|
| **Average** | 4.88 | 0.56 | 2.36 | 0.27 |
| **Std** | 0.217 | 0.067 | 0.165 | 0.053 |

Figure 6: Mix 4 3D printed.



## 4. CONCLUSION

In this investigation the development of a 3D printable lightweight EV-based fire-resistant concrete is reported. Sieve analyses and absorption rate tests of the eligible grades of EV were conducted. Mini-slump tests were performed to evaluate the printability and slump retention of unsaturated-, saturated-, and silane treated EV concrete mixes. Finally, early age mechanical tests were done on the EV mixes developed and the best mix was printed. The following conclusions can be drawn:

- Sieve analyses results revealed that micron EV in combination with silica sand closely resembles the Fuller-Thompson curve, which is the ideal grading curve for 3DPC.
- Absorption tests show that micron EV had the steadiest absorption rate and that treated micron EV is best for incorporation in EV based 3DPC.
- Rheology and mini-slump tests showed both final mixes having thixotropic properties. However, the 50% EV mix showed superior fresh state properties over the 60% EV mix.
- A significant difference between the dynamic and static yield stress was noted for both viable mixes, indicating a high probability of printability.
- Mechanical test results showed that the 50% EV mix was far superior to the 60% EV mix, while only being 150kg/m³ denser.
- The 50% EV mix was printed and achieved 27 – 10mm layers.

In conclusion, silane treated micron EV showed the best results for including EV in 3DPC. The 50% EV mix showed improved fresh state and mechanical properties over the 60% EV mix and has great potential for 3D printable EV concrete. For mix improvement it is recommended that fibres and a viscosity modifier be incorporated to improve the cohesion and buildability.

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