# Blast and Fire Resistant Material

# B A M

## EXCELLENCE/0421/0137

## DELIVERABLE D3.2

## SMART COMPOSITE GEOPOLYMERIC CONCRETE (SCGC)



Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

## Project Information

| | |
|---|---|
| Project Title | Blast and Fire Resistant Material |
| Project Acronym | BAM |
| Programme | EXCELLENCE HUBS |
| Grant Number | EXCELLENCE/0421/0137 |
| Project Duration | 01.04.2022 – 31.03.2024 (24 months) + 3 months extension |

## Document information

| | |
|---|---|
| Deliverable Number | D3.2 |
| Deliverable Title | Smart Composite Geopolymeric Concrete (SCGC) |
| Version | 1.0 |
| Dissemination Level | Public |
| Work package | WP3 |
| Authors | Ioanna Giannopoulou (FRC), Andreas Lampropoulos (UBrighton), Ponsian Robert (FRC) |
| Contributing partners | FRC, RECS, UCY |
| Delivery Date | 08.05.2024 |
| Planned Delivery Month | M15 |
| Keywords | Geopolymers, Fibres, Curing, Fire, Blast, Impact, Properties |



Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

## Version History

| No | Description | Author | Organization | Date |
|---|---|---|---|---|
| **1** | First draft | Dr Ioanna Giannopoulou<br>Dr Andreas Lampropoulos | FRC<br>UBrighton | 04.12.2023 |
| **2** | Second draft with the addition of Partners' contribution | Dr Ioanna Giannopoulou<br>Mr Ponsian Robert<br>Dr Ourania Tsioulou | FRC<br>FRC<br>UBrighton | 30.01.2024 |
| **3** | Review of the document | Dr Demetris Nicolaides<br>Dr Thomaida Polydorou<br>Dr Andreas Lampropoulos | FRC<br>UCY<br>UBrighton | 29.04.2024 |
| **4** | Final version with integration of the reviewers' feedback | Dr Ioanna Giannopoulou | FRC | 08.05.2024 |



Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

# Table of Contents





Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

## EXECUTIVE SUMMARY

One of the key objectives of the research project "***Blast and Fire Resistant Materials (BAM)***" was the design and development of an innovative, sustainable, and low-cost composite material, which will offer simultaneously the appropriate resistance against blast, impact and fire, according to the relevant standards (WP3, Task 3.2). This material, ***Smart Composite Geopolymeric Concrete*** (***SCGC***), is designed for use in both existing and new constructions and buildings. Its purpose is to dissipate the energy of blast/impact loads during explosions, by enhancing their ductility and toughness. Additionally, SCGC aims to protect buildings from fires that may follow such events or occur independently. The production of SCGC was investigated by two processes, namely conventional casting and advanced 3D printing.

The Host Organisation (FRC) has successfully designed and developed a high performance geopolymer material based on an industrial by-product, the ground granulated blast-furnace slag (GGBFS). The optimized material exhibited compressive strength higher than 130 MPa and flexural strength of 8.6 MPa. The reinforcement with steel, PPE or basaltic fibers did not significantly improve the material's mechanical strengths. The optimized SCGC was also subjected to thermal treatment at high temperatures up to 1050 °C for two hours, where it lost a significant portion of its mechanical performance and structural integrity. However, the high compressive and flexural strength of the developed SCGC have been considered as a positive indication for the material's ability to exhibit blast and impact resistance, and thereby to be used for the protection of buildings against blast, impact and fire.

Moreover, the Foreign Organisation of the project, i.e., the University of Brighton (UoB) has successfully investigated the design and development of another fiber reinforced geopolymer concrete (FRGC) with strain hardening characteristics (WP3, Task 3.2). The geopolymer matrix of this material was based on a ternary binder of Fly Ash (FA), Ground Granulated Blast-Furnace Slag (GGBS), and Silica Fume (SF) mixtures with potassium silicate alkaline activator. The mechanical properties of FRGC, the impacts of fibre type, volume percentage, and fibre aspect ratio were investigated; tests for the compressive, tensile, and flexural strengths were carried out to determine the mechanical properties of FRGC. Scanning electron microscopy (SEM) was also employed to evaluate the microstructure of geopolymer mixes under investigation.

Deliverable "***D3.2- Smart Composite Geopolymeric Concrete (SCGC)***" presents the efforts performed to design, develop and optimize the new SCGC material with blast, impact and fire resistance, as well as the new FRGC material with same targeted characteristics.



Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

The properties of the new SCGC were validated in WP4, Task 4.2, including its fire resistance according to the standard ISO 834 time-temperature curve and the results are presented in Deliverable "***D4.2-Validation of Materials in the Laboratory***". In addition, the flowsheets followed to produce SCGC by casting and 3D printing processes are also developed and presented in Deliverable "***D4.1-Flowsheets of Materials Production***". Finally, the experimental results and findings of ***D3.2*** fed the Deliverables "***D5.1-Technoeconomic Evaluation***" and "***D5.2-Cost Benefit Analysis (CBA)***".



Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

## LIST OF FIGURES





Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

## LIST OF TABLES





Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

# 1. Introduction

WP3 and specifically Task 3.2 of the research project "***Blast and Fire Resistant Materials (BAM)***" deals with the design and development of an innovative, sustainable, and low-cost Smart Composite Geopolymeric Concrete (SCGC) with dual functionality: to resist against fire and blast/impact loads. In addition, the development of a second, innovative, Fiber Reinforced Geopolymer Concrete (FRGC) has been investigated in a parallel study implemented also in the frame of WP3, Task 3.2.

Nowadays, a blast and impact resistant concrete is synonymous to the Ultra High Strength Concrete (UHSC), usually reinforced with fibers, which has the advantage of very high compressive and flexural strength (>150 MPa and >20 MPa, respectively). However, these materials have the disadvantage of very high cost, while they also suffer from intense explosive spalling phenomena and loss of their structural integrity when exposed to high temperatures that are developed during a fire incident occurring after blast and impact events. The possibility to protect the UHSC material against fire with a commercially available, superficial fire-resistant material is an expensive and not efficient solution, as such materials are usually destroyed during fire and have very low mechanical strength (<3 MPa) to withstand any blast and impact loads.

This gap was aimed to be filled by the new SCGC and FRGC materials designed and developed in the research project BAM, which are focused on offering an efficient solution to any structure for simultaneous blast/impact and fire resistance. The casting production method was followed for both the new materials, while the production of SCGC was also investigated by using the high-end technology of 3D printing.



Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

# 2. Design and Development of the Smart Composite Geopolymeric Concrete (SCGC)

## 2.1 Optimization of materials by casting production process

The Smart Composite Geopolymeric Concrete (SCGC), which is an innovative material for the protection of new and existing buildings and structures against blast, impact and fire was designed and developed by FRC (in collaboration with other project partners), using the casting production process. This material was based on an industrial by-product, specifically on ground granulated blast furnace slag (GGBFS), thus decreasing the total production cost and enhancing sustainability. The used GGBFS contained about 37 %wt. of $SiO_2$, 9 %wt. $Al_2O_3$, 45 %wt. of CaO and 7 %wt. of MgO and it is imported in Cyprus as raw material for the needs of cement industry. The optimization of SCGC was carried out by investigating the effect of the main geopolymerization process parameters (including solid-to-liquid (S/L) ratio, composition of the alkaline activator, addition of solid aggregates and soluble silicates (silica fume)) and the type and content of fibers used for reinforcing, on the compressive and flexural strengths of the materials. The target values set for the SCGC were a compressive strength of at least 150 MPa and a flexural strength of at least 20 MPa. According to the literature, these values would ensure the material has the required blast and impact resistance.

For the preparation of the SCGC material samples, sodium hydroxide and silicate solutions were first mixed for 2 minutes using a magnetic stirrer, to form the alkaline activator. The activator was then mixed with the geopolymer precursor (GGBFS) in a Hobart mixer for 5 minutes to form a homogeneous and viscous paste. Subsequently, the silica sand used as aggregate, the silica fume and the fibers were added in the paste and mixed gently with a spatula. Especially for the fibres' addition, a sieve (with aperture marginally smaller than the fibres' length) was used to allow through a mild vibration a homogeneous distribution within the geopolymeric paste. After that, the fresh geopolymeric paste was cast into steel cubic moulds of dimensions 50 mm x 50 mm x 50 mm, covered with a plastic film and cured for 7 days at 30 °C, under atmospheric pressure and non-controlled humidity conditions. After curing, specimens were demoulded and left for hardening for another 7 days at ambient conditions, before any measurement and test to be carried out. The prepared SCGC materials were evaluated in terms of compressive and flexural strengths and the optimized among them were assessed for thermal stability and mechanical performance at high temperatures and for fire resistance according to the standard ISO 834 fire-curve.

The GGBFS used for the development of the SCGC was basic slag, with increased basicity $k_b$ = 1.13. Basicity ($k_b$) is a very important property for slags, indicating the weight ratio of basic



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION
*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

oxides (CaO and MgO) to acidic oxides ($SiO_2$ and $Al_2O_3$) contained in the slag. If the basicity of a slag is greater than one, the slag is referred to as "basic"; in the opposite case, it is referred to as acidic. In general, the increase of slag basicity increases its cementitious properties. The used slag is also considered suitable for alkali activation, since its reactivity expressed with the "simple basicity" index $CaO/SiO_2$ (1.21) is ranging between 0.5 – 2, while its modulus of activity $Al_2O_3$ / $SiO_2$ (0.24) is between 0.1 and 0.6 [(1)].

The development and optimization of the SCGC was performed through 10 extensive experimental series, in which the effect of various process parameters on materials compressive and flexural strengths was investigated. Specifically, the following process parameters were investigated:

- the solid-to-liquid (S/L, g/mL) ratio in the geopolymer binder.
- the volumetric ratio of activator's solutions, sodium silicate to sodium hydroxide (SS : SH, v/v).
- the molarity (M) of sodium hydroxide solution ([NaOH], mol/L).
- the addition of aggregates in the form of silica sand (SS addition, %wt. of the precursor).
- the addition of easily dissolved silicates, in the form of silica fume (SF, %wt. of the precursor).
- the introduction of silica fume in the geopolymer system, as solid in the precursor or after dissolution in the activator.
- the addition of different types of fibers, i.e., steel, PPE and basalt.
- the addition of fibers with simultaneous addition of silica fume.

The optimization of the SCGC revealed the following observations:

a) The solid-to-liquid ratio, S/L, was proved to be a very important factor affecting the rheology of the formed geopolymeric paste and thus, its workability and setting time. Even small changes of this factor led to significant variations in the workability of the geopolymeric paste. In addition, the activator's solutions ratio, SS : SH (v/v), which is related to the concentration of soluble silicates in the geopolymeric system and therefore is an indicator of the extent of the geopolymeric network, was also proved to be a very important factor that affects the viscosity of the formed paste. As it is obvious in Figure 1, the increase of the S/L and SS:SH ratios led to less workable geopolymeric pastes.

[1] F. Winnefeld et al., Influence of slag composition on the hydration of alkali-activated slags, Journal of Sustainable Cement Based Materials, 2015, 4 (2), pp. 85-100.



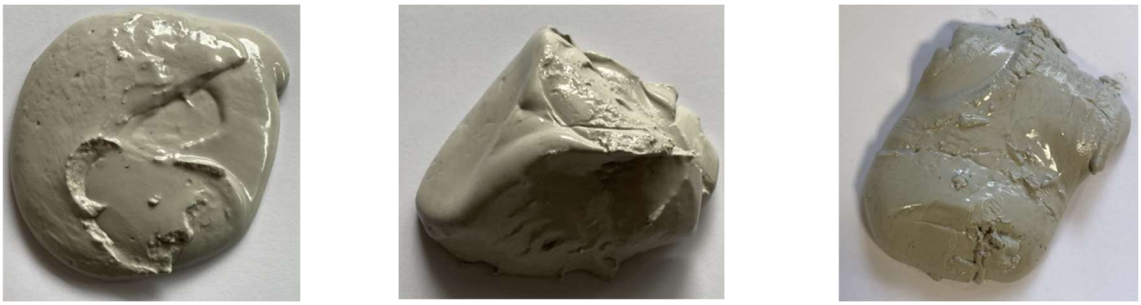
Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

S/L = 3 g/mL
SS / SH = 1.5 : 1

S/L = 3.2 g/mL
SS / SH = 1.5 : 1

S/L = 3.5 g/mL
SS / SH = 1.5 : 1



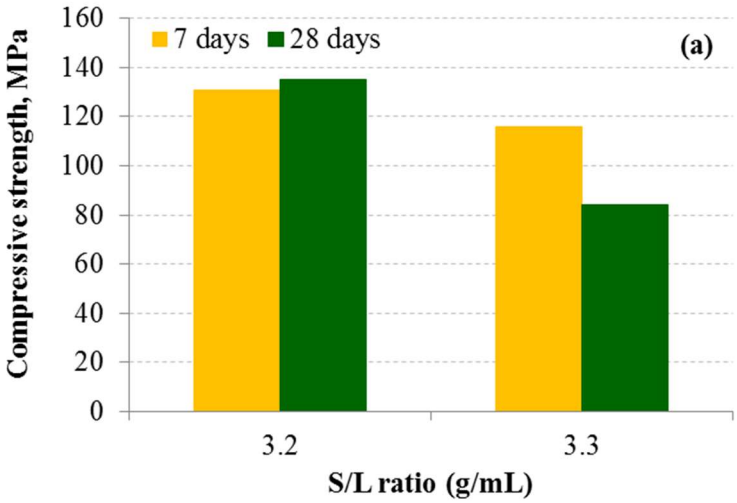
S/L = 3 g/mL
SS / SH = 2.5 : 1

S/L = 3.2 g/mL
SS / SH = 2.5 : 1

S/L = 3.3 g/mL
SS / SH = 2.5 : 1

**Figure 1.** Effect of S/L and SS:SH ratios on the viscosity and workability of the geopolymeric paste.

b) The viscous geopolymeric pastes formed as the S/L and SS:SH ratios were increased made the materials moulding difficult, thus creating discontinuities in the mass of the specimen that reduced the materials' mechanical strengths (Figure 2).






Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

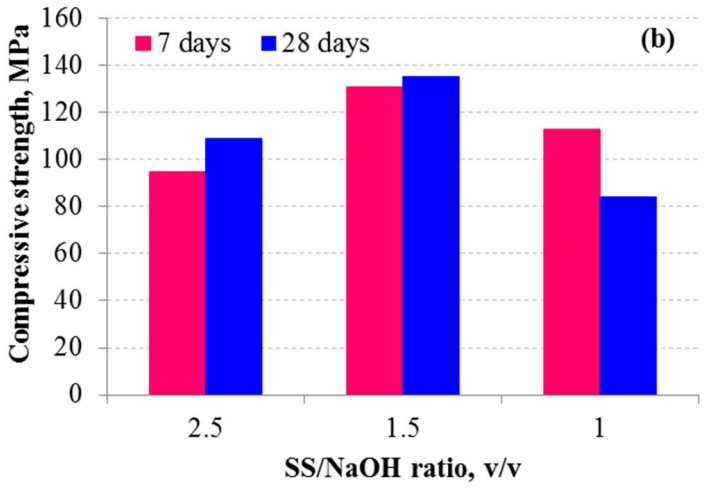



**Figure 2.** Effect of (a) S/L ratio and (b) SS/SH ratio on the compressive strength of SCGC materials (curing conditions: T = 30 °C, t = 7 days).

c) The addition of silica sand (15-85% wt.), as well as of silica fume (0.5-15% wt.), in the geopolymer precursor did not improve the compressive strength of the materials. Same result had also the addition of silica fume in the alkaline activator. The silica sand was added in the geopolymer precursor after standing in distilled water for 24 hours and careful draining (saturation with water). However, this addition did not improve the compressive strength of SCGC, despite the fact that different quantities of silica sand were explored.

d) The effect of fibers on compressive strength was investigated with various additions ranging from 0.6 to 11% by weight, using three different types of fibres: steel fibers, PPE fibres, and basalt fibres. Each type of fibre had an optimal volume, which varied among the different fibres. High compressive strength was achieved at the optimized fibre content, although it was generally equal to or lower than the compressive strength achieved by the optimized GGBFS-based geopolymeric binder. Specifically, a compressive strength of 132 MPa was achieved with the addition of 1.6% by weight of steel fibres. A compressive strength of 104 MPa was reached with the addition of 0.7% by weight of PPE fibres, and 102 MPa was achieved with the addition of 1.6% by weight of basalt fibres.

e) The addition of steel fibres did not improve the flexural strength of SCGC. More precisely, the optimum addition of steel fibres resulted in a material with almost the same compressive and flexural strengths as the geopolymer binder, before the addition of any fibres.



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

In Table 1, the synthesis conditions of the optimized SCGC material are given, while Table 2 summarizes its basic properties.

**Table 1**: Synthesis and curing conditions of the optimized SCGC material.

| **Synthesis Conditions** | |
|---|---|
| ***Parameter*** | ***Value*** |
| Mass of GGBFS (g) | 800 |
| Mass of Silica sand | 0 |
| Mass of Silica fume (g) | 0 |
| Volume of 7 M NaOH (mL) | 100 |
| Molarity of NaOH solution (M) | 7 |
| Volume of Na-silicate solution (mL) | 150 |
| Fibers (% wt.) | 0 |
| ***Ratio*** | ***Value*** |
| S/L ratio (g/mL) | 3.2 |
| SS to SH volumetric ratio (v/v) | 1.5 |
| **Curing Conditions** | |
| ***Parameter*** | ***Value*** |
| Temperature (°C) | 30 |
| Time (days) | 7 |

**Table 2**: Properties of the optimized SCGC material.

| ***Property of SCGC*** | ***Value*** |
|---|---|
| Density (g/cm$^3$) | 2.12 |
| Compressive strength (MPa) | 135.47 |
| Flexural strength (MPa) | 8.60 |
| Water absorption (% wt.) | < 0.05 |



Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

## 2.2 Thermal stability and mechanical performance of SCGC at high temperatures

The optimum SCGC material was exposed to temperatures of 600, 800 and 1050 °C for 2 hours, using the electric furnace shown in Figure 3. The SCGC specimens used for this testing were cured at 30 °C for 7 days and then left for hardening at ambient conditions for at least one month. These tests have been carried out as a preliminary assessment of the material's fire resistance.

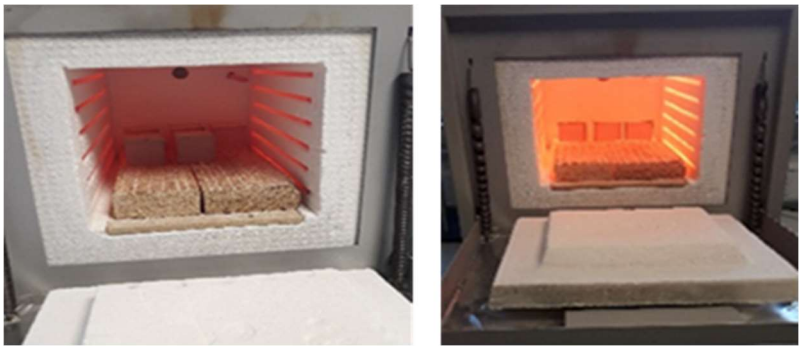


**Figure 3**. The electric furnace used for the fire resistance assessment testing of SCGC.

In each test, three cubic samples with dimensions 50 x 50 x 50 $cm^3$ were placed in the furnace and heated with a constant heating rate of 4.4 °C / min, until to reach the desired temperature (600, 800 or 1050 °C), where they left for 2 hours. The samples were then removed from the furnace and allowed for cooling down to room temperature in open air conditions, before the performance of any measurement or testing. The samples exposed to the elevated temperatures were assessed in terms of their compressive strength, density, mass loss and linear shrinkage. Moreover, their structural integrity was macroscopically investigated.

The properties of SCGC after their exposure to high temperatures for 2 hours are presented in Table 3.

**Table 3**: Properties of the Na-FRG and K-FRG materials after exposure to high temperatures.

| *Material ID* | *Temperature (°C)* | *Residual Compressive Strength (MPa)* | *Density ($g/cm^3$)* | *Mass loss (% wt.)* | *Linear Shrinkage (%)* |
|---|---|---|---|---|---|
| *SCGC* | 30 | 124.3 | 2.1 | | |
| | 600 | 75.7 | 1.8 | 18.8 | 1.9 |
| | 800 | 8.8 | 2.1 | 18.6 | 7.3 |
| | 1050 | 7.3 | 2.2 | 18.6 | 8.7 |

An intensive cracking appeared on the surface of all specimens after their exposure at all the investigated temperatures. Due to this cracking, the compressive strength of SCGC was sharply decreased after its thermal treatment (Table 3).



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

## 2.3 Production of the SCGS by the 3D printing method

The DELTA WASP 3MT 4.0 LMD (Liquid deposition Modelling) 3D Printing machine with manual extruder, including 3D printing and modelling software, was used for the purposes of 3D printing process in the frame of the BAM project. The equipment was established and operating at Frederick Research Center since March 2021 (Figure 4).

**Experimental work:**

The research team performed several successful trial runs on the 3D printer to produce the materials designed in the project. Initial experimental trials were conducted to familiarise with the equipment and the printing process. The most important process parameters controlling the process feasibility, i.e. good material deposition, and, thus, the quality of the build-up structure are the following:

- Nozzle diameter
- Layer height
- Extrusion rate
- Printing speed
- Internal infill type
- Infill percentage


**Figure 4**: DELTA WASP 3MT 4.0 LMD 3D Printing machine.



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

The essential steps for the material preparation with 3D printing are elaborated in the following:

- **3D Model Design and Slicing of the Model**
  - Preparation of the 3D model (30 cm x 30 cm x 5 cm) using computer aided design (CAD).
  - Save the 3D model in Standard Triangulation Language (STL) format.
  - Slicing of the 30 cm x 30 cm x 5 cm model into layers of equal height.
  - Conversion of the sliced into G. Code format, using SIMPLIFY 3D software.
- **Preparation of Printable Geopolymeric Slurry**
  - Preparation of the alkaline solution by dissolving NaOH pellets of analytical grade (99.9% purity) in deionized water to obtain a sodium hydroxide solution (NaOH) of 7M (mol/L) concentration; the prepared solution was left for at least 24 hours to stabilize.
  - After 24 hours, a predefined volume of the 7M NaOH solution was mixed with the relevant volume of sodium silicate solution ($Na_2SiO_3 \cdot xH_2O$) of MR > 3.4 in a plastic beaker using a magnetic stirrer for 1-2 minutes, to obtain a homogeneous alkaline activating solution.
  - The alkaline activator was then added into the geopolymer precursor consisting of a predetermined quantity of well mixed fly ash and blast furnace slag powder, and the resulting mixture was mixed for 4-5 minutes until a homogeneous geopolymeric paste was obtained.
  - Measuring the viscosity of the geopolymeric paste before the beginning of printing and ensuring sufficient rheology during printing (Table 4).

**Table 4**: Viscosity and rheology of mixture prior and during printing.

| S/L (g/ml) | Viscosity (Pa.s) | Shear (Pa) | Speed (rpm) |
|---|---|---|---|
| 3.8 | 8.59 | 4.50 | 500 |
| 3.6 | 7.46 | 3.98 | 500 |



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

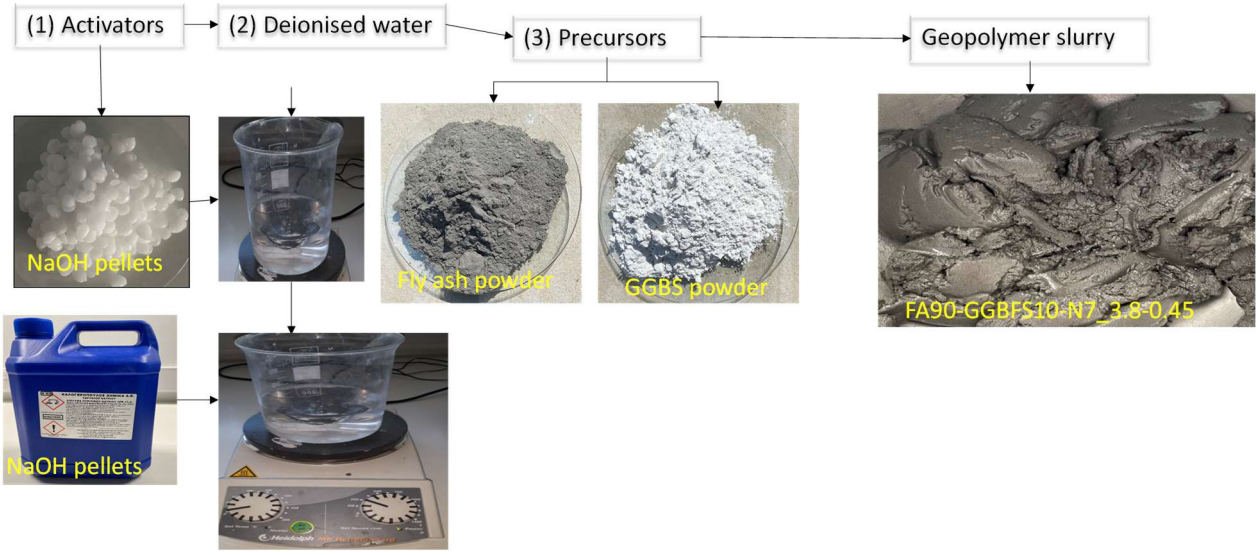



**Figure 5**. Preparation procedure of geopolymer slurry.

- **<u>3D Printing Process Execution</u>**

The parameters adopted for the 3D printing of materials are the following:

- Nozzle diameter = 10 mm
- Layer heigh t= 10 mm
- Printing feed = 30 mm/sec
- Infill angle = 45°/-45°
- Infill density level = 100%
- Printing direction = inside out
- Deposition height = 22 mm

**<u>Note:</u>** The viscosity of the geopolymeric paste was checked before and during printing. The printing process of this geopolymer slurry should not exceed half an hour since the preparation of the slurry, under laboratory conditions, i.e., RH = 46% and T = 35 °C.



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

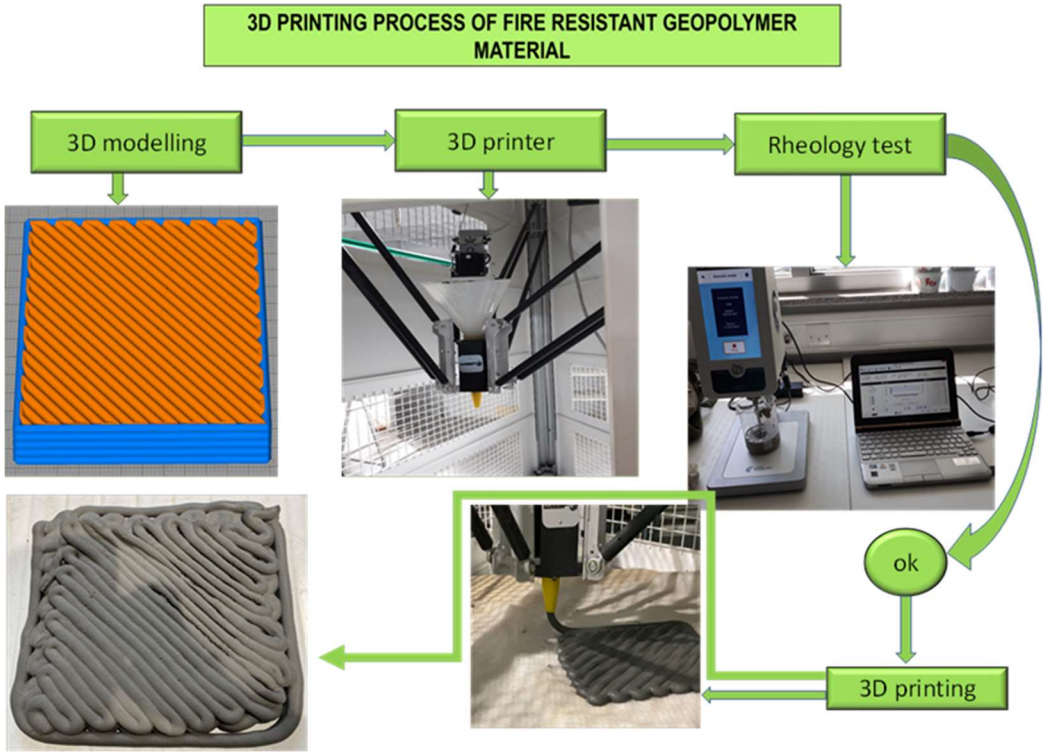



**Figure 6**. 3D printing process execution.



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

# 3. Development of Fiber Reinforced Geopolymer Concrete (FRGC)

In the current work, a ternary blend geopolymer binder (FA, GGBS, and silica fume) was combined with sand and potassium silicate ($K_2SiO_3$) at a molar ratio of 1.25 to create the geopolymer mortar matrix. The current investigation's mixes utilized total binder and silica sand quantities of 775 kg/m$^3$ and 1054 kg/m$^3$, respectively. These values were derived from an earlier study [(2)].

## 3.1 Material and Mix Proportions

A total of fourteen different mix combinations were examined, each with a different fibre type, and fibre volume percentage (Table 5).

**Table 5**: Mixture compositions of fibre reinforced geopolymer concrete.

| MIX ID | OPC (Kg/m$^3$) | FA / Binder | GGBS / Binder | SF/ Binder | Sand (Kg/m$^3$) | $K_2SiO_3$ / Binder | Water / Binder | Fibre $V_f$ (%) |
|---|---|---|---|---|---|---|---|---|
| OPC mortar | 650 | - | - | - | 1525 | - | 0.35 | 0 |
| GP mortar | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 0 |
| 2ST6 | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 2 |
| 3ST6 | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 3 |
| 1ST13 | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 1 |
| 2ST13 | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 2 |
| 3ST13 | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 3 |
| 3[ST6-ST13] | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 3 |
| 1HE | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 1 |
| 1HE-1ST13 | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 2 |
| 1HE-2ST13 | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 3 |
| 1PVA | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 1 |
| 2PVA | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 2 |
| 1Glass | - | 0.5 | 0.4 | 0.1 | 1052 | 0.12 | 0.25 | 1 |

Figure 7 and Table 6 provide details about the fibres that were employed. The fiber-reinforced geopolymer composite was prepared using a Pan Mixer ZZ 75 HE high shear mixer from Zyklos. Sand, alkaline liquid, and geopolymer binder consisting of Silica Fume (SF), Fly Ash (FA), and Ground Granulated Blast-furnace Slag (GGBS) were used. Water and alkaline activator were added after the completion of the initial mixing (5 minutes) of the dry materials of the geopolymer binder and they were mixed for four minutes. Then fibres and silica sand were added and mixing

[2] Al-Majidi, M.H., Lampropoulos, A., Cundy, A., and Meikle, S.: 'Development of geopolymer mortar under ambient temperature for in situ applications', Construction and Building Materials, 2016, 120, pp. 198-211.



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

lasted for an additional four minutes, for a total mixing time of thirteen minutes. The specimens were demoulded, cured at room temperature, and covered with plastic sheets until the testing of the examined specimens.


**Figure 7**. Geometry and shape of fibres used in this study.

**Table 6**: Fibres properties used in this study.

| Fibre type | Geometry | Length (mm) | Diameter (mm) | Aspect ratio (L/D) | Fibre strength (MPa) | Density (Kg/m$^3$) | E (GPa) |
|---|---|---|---|---|---|---|---|
| Steel (ST6) | Micro | 6 | 0.16 | 37.5 | 2250 | 7850 | 200 |
| Steel (ST13) | Micro | 13 | 0.16 | 81.25 | 2250 | 7850 | 200 |
| Steel (HE1050) | Macro | 50 | 1 | 50 | 1150 | 7850 | 200 |
| Glass | Micro | 13 | 0.13 | 100 | 1620 | 2700 | 74 |
| PVA | Micro | 12 | 0.015 | 800 | 1560±325 | 1300 | 29.5 |

## 3.2 Experimental equipment and test procedures

Compression, flexural, and direct tensile tests were conducted to evaluate the mechanical properties of the examined mixtures. The strength (compressive and tensile) development over time was also examined through the mechanical testing. Scanning electronic microscopy (SEM) was also used to evaluate the microstructure of the examined mixes.

For the evaluation of the compressive strength, nine cubes with 50 mm sides were tested for each mixture to study the compressive strength development over the curing period (3, 7 and 28 days). A Denison Avery 2000 KN testing machine was used for these tests with a loading rate of 180 KN per minute.



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

For the flexural tests, standard prisms (100 x 100 x 500 mm) were tested at 28 days to evaluate the flexural strength characteristics. An Instron testing equipment was used and the loading points were spaced 1/3 of the span length apart, with a span length of 450 mm. Details of the flexural test setup are shown in Figure 8. The prisms were loaded into the testing apparatus at a constant deflection rate of 0.24 mm/min using a "closed loop" operation. To minimize any induced displacements at the supports during that loading, two Linear Variable displacement Transducers (LVDTs) were mounted to a yoke frame (refer to Figure 8).

"Dog bone" shaped samples (13 mm by 50 mm mid cross section) were used to assess the direct tensile strength (Figure 9a). To measure displacement along the small cross section, a steel frame with one LVDT was used over a 105mm gauge length (Figure 9b). The test was conducted at 3, 7, 14, and 28 days to evaluate the tensile strength development of the examined geopolymer specimens. The tests were conducted under a constant loading rate of 0.4 mm/min, and measurements were recorded until the maximum load was reached. The testing also continued in the post-cracking region to evaluate the full stress strain characteristics and the energy absorption of the examined mixes.

After the tensile testing was completed, samples of Plain Geopolymer and FRGC were extracted from the cracked samples for microstructure investigation. The samples were examined using scanning electron microscopy (SEM) (Zeiss; model of LEO 1455VP) after the required treatment.

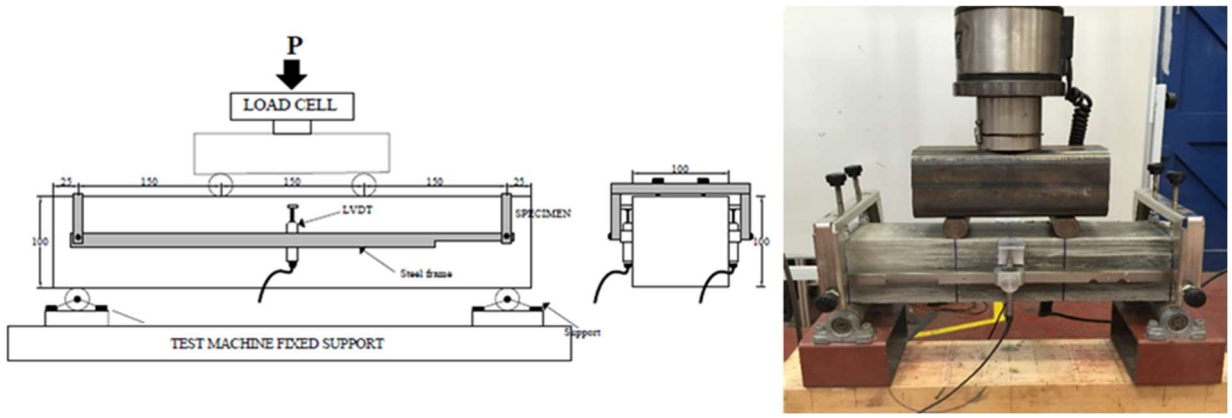



**Figure 8**. Bending specimen geometry (**a**); and test set-up (**b**).



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

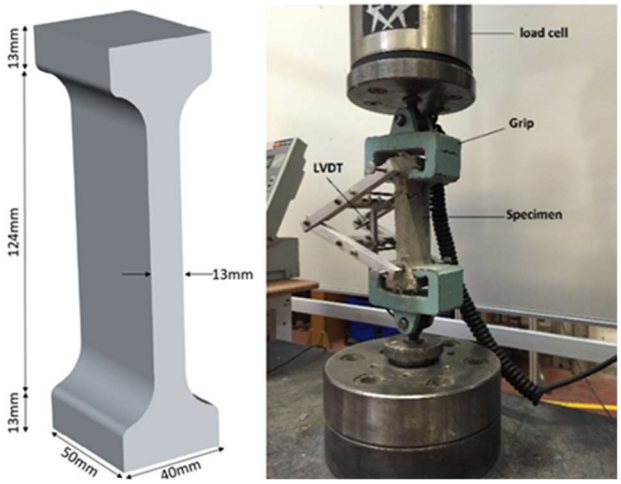



**Figure 9**. Direct tensile specimen geometry (**a**); and test set-up (**b**).

## 3.3 Results

**Compressive Strength Test**

The compressive strength development of strain hardening geopolymer concrete over curing time was examined, using cubes with 50 mm side (Figure 10). This cubic size was valid only for micro fibre mixtures as the macro fibre length exceeded the limit of the cube size. Each data point corresponds to an average of three specimens.

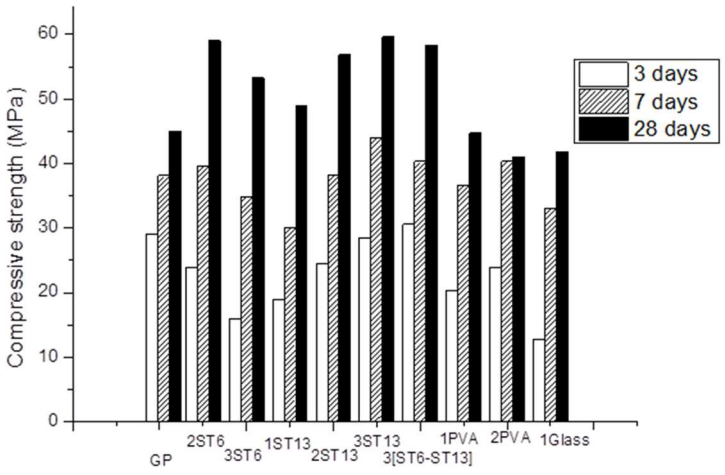



**Figure 10**. Compressive strength of various FRGC mixes based on 50 mm cubes.

The results show that, for all the examined mixes, the compressive strength of SHGC increases with the curing time. The seven-day compressive strength appears to be increased by 31%, 64%,



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION
*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

48%, and 80% for the PG, 2ST6, 2ST13, 2PVA, and 1Glass mixtures, respectively when compared to the 3 days results. The results were further increased at the 28 days where the compressive strength was 45 MPa, 59 MPa, 57 MPa, 45 MPa, and 42 MPa for PG, 2ST6, 2ST13, 2PVA, and 1Glass mixes, respectively.

## Flexural strength test

The main benefit of the use of fibres is the enhanced ductility and energy absorption which is evidenced by the experimental results Figures 11 and 12 show the impact of various volume fractions and fibre kinds on the load-deflection relationships, respectively.

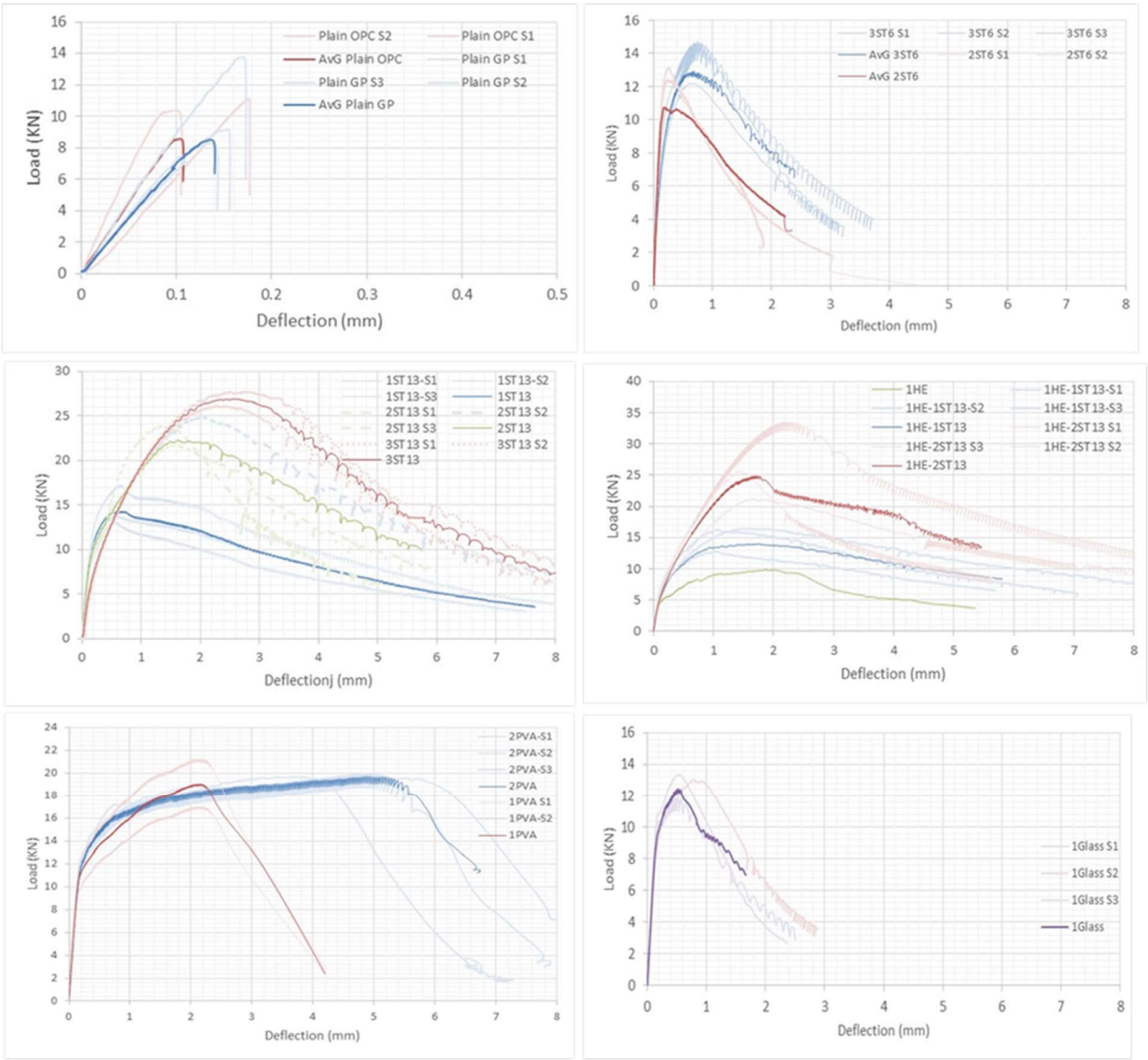



**Figure 11**. Load-deflection relationships: plain geopolymer mortar and OPC mortar (a); 2ST6 and 3ST6 mixtures (b); 1ST13, 2ST13 and 3ST13 mixtures (c); 1HE, 1HE-1ST13 and 1HE-2ST13 mixtures (d); 1PVA and 2PVA mixtures (e); and 1Glass mixture (f).



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

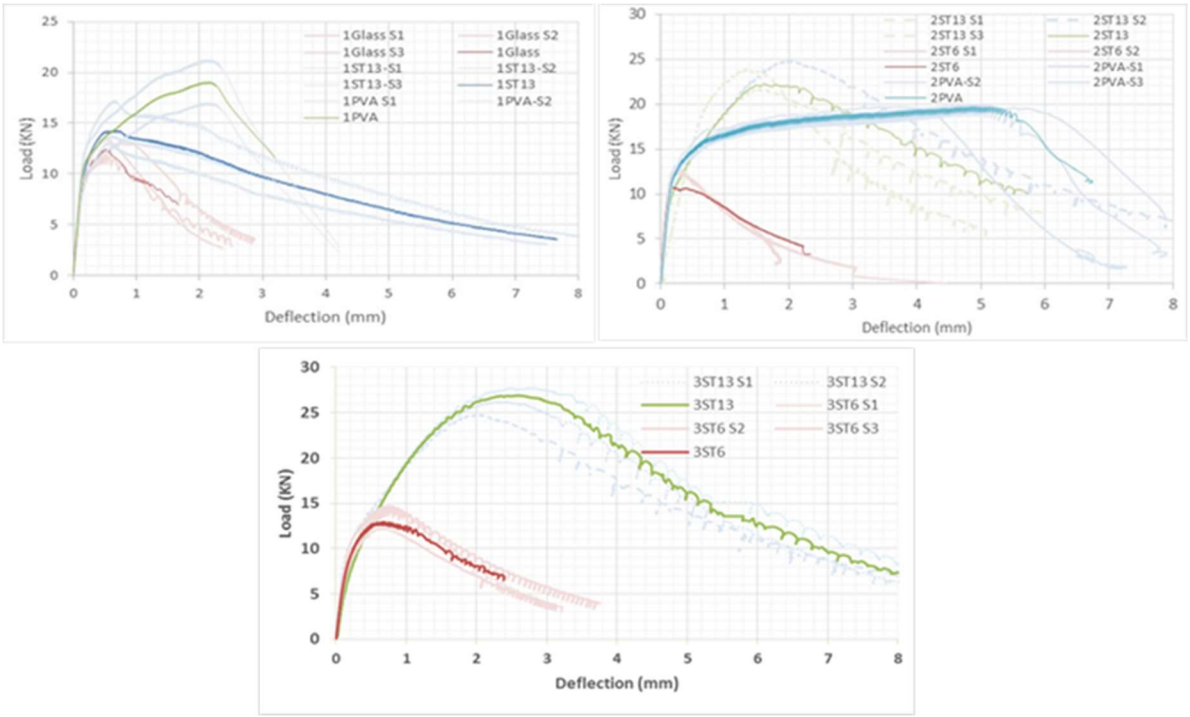



**Figure 12**. Comparisons of load-deflection relationships for variant fibre types within the same volume fraction: 1Glass, 1ST13, and 1PVA mixtures (a); 2ST6, 2ST13 and 2PVA mixtures (b); and 3ST13 and 3ST6 (C).

The plain geopolymer concrete specimens broke into two parts because of cracking that started in the middle and spread swiftly to the top. Steel fibre addition had a beneficial effect on all the geopolymer combinations' post-cracking performance. All the fibre-reinforced geopolymer combinations had a similar behaviour with the load increasing nonlinearly up to the ultimate load followed by an almost linear part up to the first peak. Then in the post-peak region the load is reduced until the failure. Increment of fibre volume fraction and use of fibres with higher aspect ratios led to enhancement of the load bearing capacity of FRGC (Figures 11b-c).

The load-deflection curve of a geopolymer composite with macro hooked end fibre HE1050 is shown in Figure 11d. The load-deflection relation is characterised by three distinct segments when only hooked end fibres are used: the linear section, the strain hardening section, and the strain softening section, with low flexural strength equal to 3.8 MPa.

Regarding PVA-FRGC (Figure 11e), a significantly enhanced deflection capacity was observed, with the load-deflection curve displaying distinct first and second peaks. There was not any significant effect on the final flexural load when the volume fraction of PVA fibres was increased.

Figure 11f shows the load-deflection relationship for the glass fibre reinforced geopolymer specimen. It is evident that the use of glass fibre increases the peak load by 50% as compared to the unreinforced geopolymer. Nonetheless, for the same volume fraction, the peak load of glass is



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

lower than that of steel and PVA, and strain-softening failure is identifiable in the post-crack behaviour with subsequent limited deflection capacity. This is attributed to the poor bond between the glass fibre and the geopolymer matrix.

**Direct tensile test**

The effect of fibre type, aspect ratios, volume fractions, and curing time on the tensile strength of the investigated mixes was investigated through direct tensile tests. Geopolymer specimens at 3, 7, 28, and 90 days were studies to examine the effect of the curing age on the stress-strain relationship of FRGC (Figure 13 and 14).

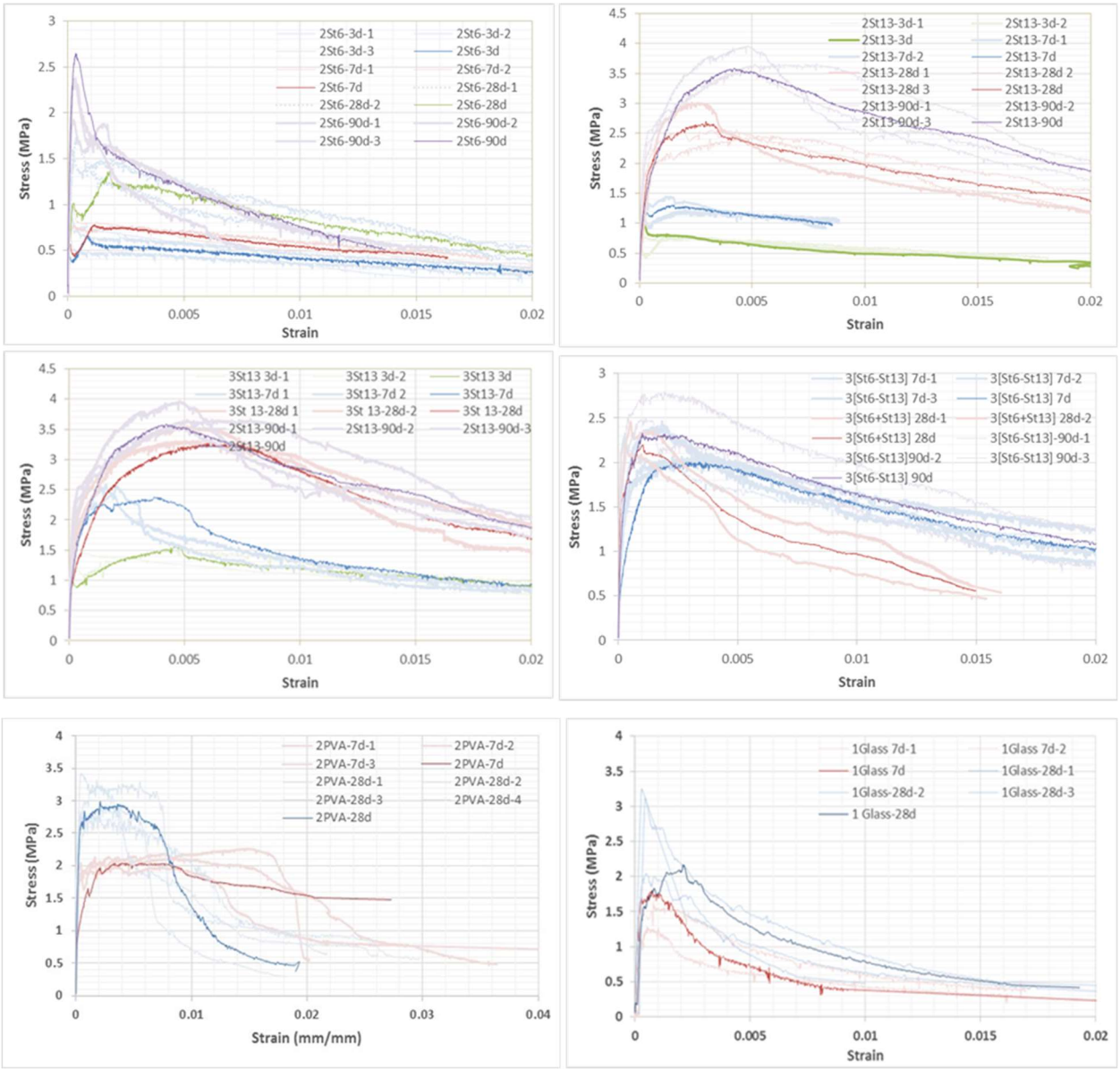



**Figure 13**. Effect of curing time on the stress strain relation under tensile strength: 2ST mixtures (a); 2ST13 mixtures (b); 3ST13 mixture (C); 3 hybrid 3(ST6-ST13) mixture (D); 2PVA mixture (E); 1Glass mixture (F).



Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

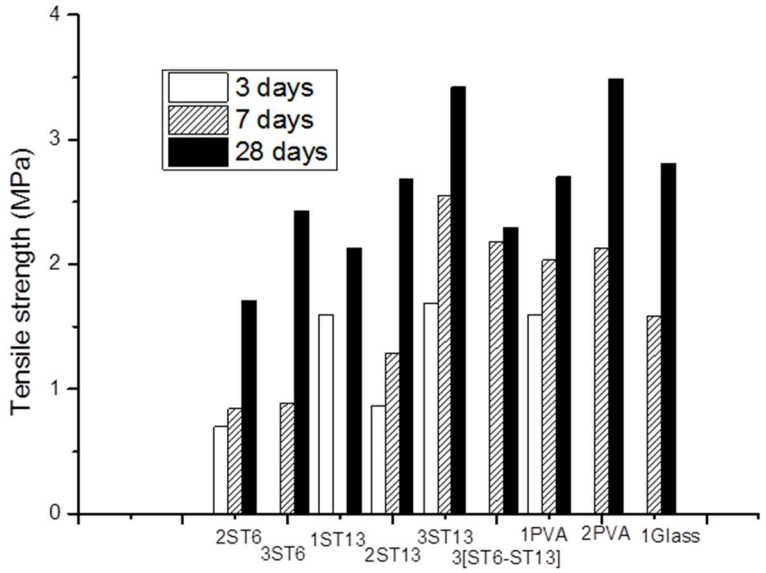



**Figure 14**. Ultimate tensile strength versus curing time.

Figures 13 and 14 demonstrate the significant effect of the curing time on the ultimate tensile strength and the post-cracking behaviour of the tensile stress-strain curves. Tensile performance of the 2ST6 mix cured at room temperature is around 0.7 MPa and 0.85 MPa, respectively, at early ages (3 and 7 days). In comparison to the specimens reinforced with 2ST13, this early strength is lower (at around 1.3 MPa and 1.43 MPa, respectively). Compared to all specimens reinforced with the two different steel fibre aspect ratios, the 2PVA-FRGC specimens have the highest tensile strength.

The tensile strength of SFRGC (0.855 MPa and 1.3 MPa for 6 mm and 13 mm fibre length, respectively) is less than that of 2PVA-FRGC at 7 days, with a tensile strength of approximately 2.13 MPa. This is attributed to the strong early interfacial bond formed between the PVA fibres and the geopolymer matrix as compared to the steel fibre reinforced composite. After seven days, the tensile strength of every SFRGC specimen had dramatically increased. As compared to their respective values at early ages, the results demonstrate a similar trend, with high values for all geopolymer reinforced with various fibres.

In comparison to the corresponding combinations at 7 days, the tensile strength of the 2ST6, 2ST13, 2PVA, and 1Glass mixtures increased by 104%, 95%, 65%, and 77% at 28 days. For curing at room temperature, the geopolymerization process improves over a sufficient amount of time, strengthening which allows the improvement of the bond between the matrix and the reinforcing fibre. Figure 11 further demonstrates how the length of curing affects the post-



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION
*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

cracking behaviour of FRGC. Nevertheless, the failure type remains constant in all the curing ages: strain hardening behaviour for geopolymer mixtures reinforced with PVA and ST13 fibre, and strain softening for short lengths of steel fibres.

**Scanning Electronic Microscopy (SEM)**

The fiber-geopolymer matrix bond properties were assessed by scanning electron microscopy (SEM) analysis of the fibre surface texture and fiber-matrix interfaces. Figure 8(a-h) displays the SEM images of the fiber-reinforced geopolymer composite with steel, PVA, and glass fibres, and the plain geopolymer mortar. The plain geopolymer mortar is illustrated in Fig. 18a, which shows a thick geopolymer microstructure and a dark, well-connected structure. Based on previously published investigations, this mixture is an ideal ternary geopolymer mixture [3]. When calcium from GGBS is combined with silica fume and FA as sources of silica, more calcium-alumino-silicate hydrate (C-A-S-H) gel is formed as a result of geopolymerization.

Figure 8 (c-h) [1] displays images of the FRGC's fracture surfaces from samples taken after the end of the tensile testing. Regardless of the fibre type, it is evident that the geopolymerization process has been successful occurred and all the examined samples have a similar, well-connected geopolymer matrix.

The geopolymer matrix covering the steel fibre surface at the fracture surface is visible in the SEM pictures. This shows a reasonably strong link between the steel fibres and the geopolymerization product, preventing the fibre in the FRGC sample from pulling out. The PVA-FRGC mixture exhibits coarser-surfaced PVA fibres, and thickening of the fibres is observed due to the accumulation of geopolymer hydration products on the PVA fibre surface. This indicates a strong link between the PVA fibres and the geopolymer matrix.

Given that PVA exhibits strain hardening behaviour and good post-crack resistance, this is consistent with the experimental data. Conversely, glass fibre exhibits a rather flat surface inside the geopolymer composite. The mechanical behaviour of FRGC is in clear agreement with these fiber-matrix interface measurements. While the effect of glass fibre was less noticeable, the addition of PVA and steel fibres greatly increased the tensile strength and post-crack behaviour. Furthermore, the fibres' unaltered diameter suggests that the alkaline geopolymer matrix has little to no degradative effect on the steel fibres.



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION
*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

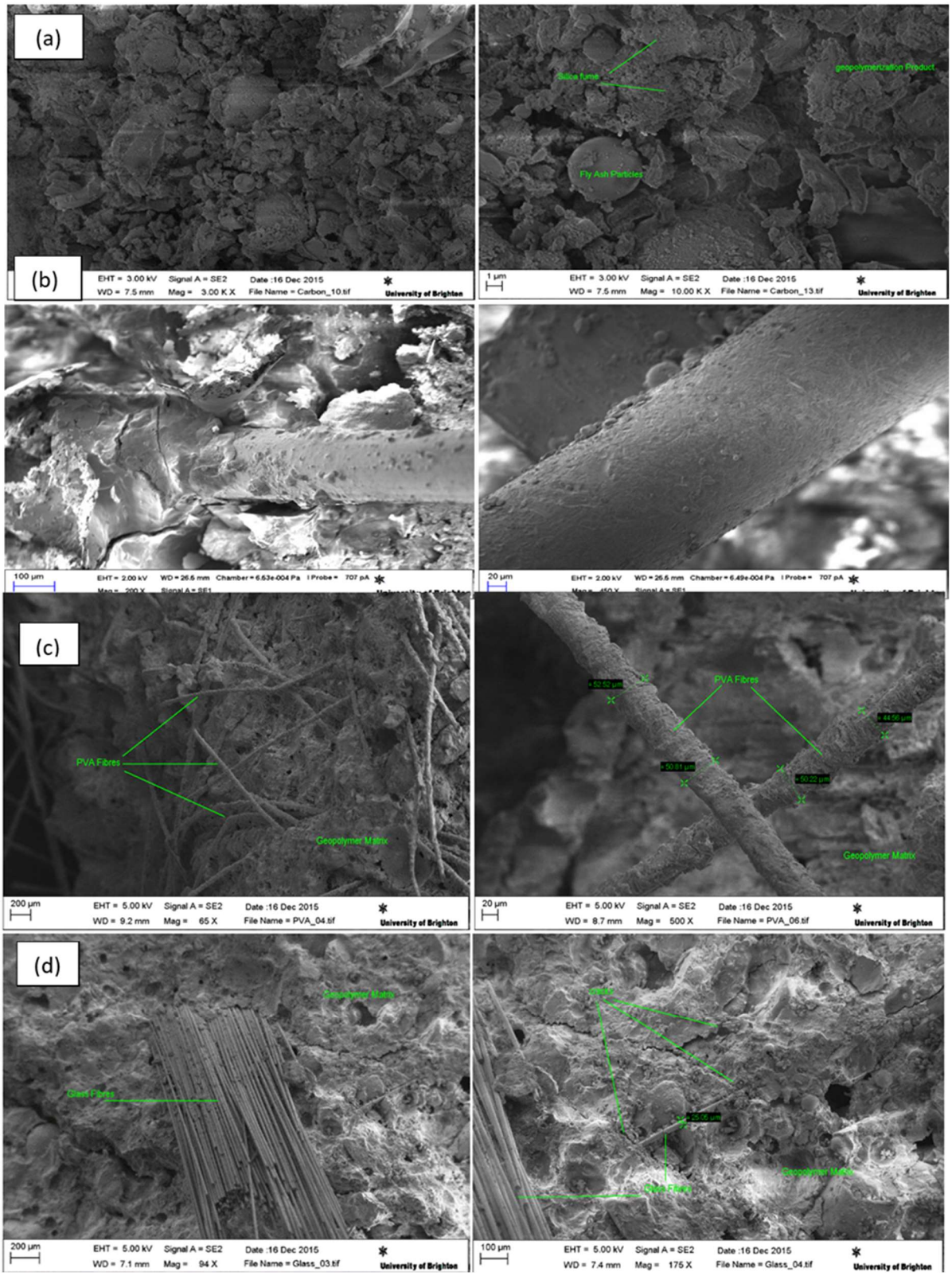



**Figure 15**. SEM micrographs of plain geopolymer mortar x3,000 magnification (a); geopolymer mortar x10,000 magnification (b); perpendicular to the fracture surface of steel fibre/geopolymer composites x3,000 magnification (c); and perpendicular to the fracture surface of steel fibre/geopolymer composites x5,000 magnification (d).



Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

## 4. Conclusions

From the investigation for the development of an innovative, sustainable and low-cost Smart Composite Geopolymer Concrete (SCGC) the following conclusions are drawn:

- The compressive strength of the GGBFS-based SCGC materials developed in this study was importantly affected by the S/L ratio and the concentration of the alkali hydroxide solution used in the activator. The SCGC material with solid-to-liquid ratio S/L = 3.2 g/mL and 7M NaOH solution in the alkaline activator reached 132 MPa compressive strength and 8.6 flexural strength.
- The addition of aggregates (silica sand), silica fume and fibres (steel, PPE or basaltic) did not improve the mechanical strength of SCGC.
- After the exposure of the optimized SCGC to high temperatures (600, 800 and 1050 °C), extended cracking appeared on specimens' surfaces, leading to a significant decrease of their residual compressive strength. This was more profound after exposure at 800 and 1050 °C. Although the density of SCGC remained almost unchanged, a mass loss of about 18% occurred after the exposure of materials to each temperature, while a linear shrinkage of about 8% was observed at the highest tested temperatures of 800 and 1050 °C.

Regarding the parallel study for the development of a Fibre-Reinforced Geopolymer Concrete (FRGC) with enhanced strain hardening characteristics, the following key conclusions were derived:

- The fibre type, volume percentage, and fibre aspect ratio were proved critical parameters for the mechanical properties of FRGC, affecting its compressive, tensile and flexural strength.
- When steel fibres were added to the geopolymer concrete, the specimens showed an increase in compressive strength of 15–25 MPa. The addition of Glass and PVA fibres did not significantly affect the compressive strength.
- The compressive strength of FRGC with steel fibres was considerably impacted by increasing the steel fibre content and aspect ratio. When 3% of steel fibres of 13 mm in length were employed, the highest compressive strength values were obtained; in this instance, the compressive strength value was approximately 70 MPa.
- Higher volume fractions and the use of longer straight steel fibres lead to improved mechanical performance in terms of post-crack behaviour and tensile strength.



Co-funded by the European Union
Republic of Cyprus
RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

- In case of SFRGC with 3% steel fibre volume fraction and 13mm long fibres, twenty times higher deflection capacity at the peak load was observed when compared to specimens without fibre, and 4 times higher than the respective results for 3% steel fibres with 6mm length.
- The maximum flexural strength of SFRGC with 13mm steel fibres was higher than the respective results for ST6, PVA and glass fibre.
- Compared to the use of macro 1% HE only, the hybrid 1%VF HE fibre with micro fibre (1% and 2% $V_f$) shows deflection capacity two to four times greater.
- In conclusion, these results demonstrates that, even in the absence of an enhanced temperature treatment, the FRGC with the addition of steel and PVA fibres has increased flexural and tensile strength characteristics in addition to enhanced energy absorption while strain hardening characteristics can also be achieved.



Co-funded by the European Union

Republic of Cyprus

RESEARCH & INNOVATION FOUNDATION

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

**Acknowledgements**

*The project is implemented under the programme of social cohesion "THALIA 2021-2027" co-funded by the European Union, through Research and Innovation Foundation.*

*Το έργο υλοποιείται στο πλαίσιο του Προγράμματος Πολιτικής Συνοχής «ΘΑΛΕΙΑ 2021-2027» με τη συγχρηματοδότηση της ΕΕ, μέσω του Ιδρύματος Έρευνας και Καινοτομίας.*