Development of an Innovative Insulation Fire Resistant Façade

from the Construction and Demolition Waste

DEFEAT

INTEGRATED/0918/0052

DELIVERABLE D5.3

MATERIAL PROTOTYPE WITH FIRE AND INSULATION PROPERTIES IN SANDWICH TYPE

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EXECUTIVE SUMMARY

Deliverable *D5.3-Material prototype with fire & insulation properties in sandwich type* includes the activities performed to develop and test the prototype of the Composite Material developed in the DEFEAT project. It concerns an innovative composite building façade consisted of two layered geopolymers, a fire-resistant and a thermal insulation, both based on the Construction and Demolition Waste (CDW). Deliverable D5.3 describes two prototypes of the DEFEAT Composite Material (DEFEAT-CM), since the optimization of materials in WP5 of the project resulted in two fire-resistant (Brick Fire Resistant - BFR and Ceramic Tile Fire Resistant - CTFR) and one thermal insulation (Ceramic Tile Thermal Insulation - CTTI) geopolymers.

The development and testing of the DEFEAT-CM prototypes, the BFR-CTTI and the CTFR-CTTI, was performed at both, small and large lab-scale. The relevant activities include the validation of the casting process that was used for the production of the DEFEAT-CM, as well as the verification of the DEFEAT-CM main properties, at both small and large lab-scale.

According to the results of the design, development and testing of the DEFEAT-CM prototypes, the casting process is suitable for the scaling up of the Composite Material production. The bonding between the two individual layered geopolymers of the DEFEAT-CM was proved strong enough for operating as an integrated component. Furthermore, most of the properties targeted in the DEFEAT project for the Composite Material were achieved in a relevant environment.

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1. Introduction

Deliverable ***D5.3-Material prototype with fire & insulation properties in sandwich type*** describes the activities carried out for the development and testing of the *DEFEAT Composite Material* (***DEFEAT-CM***) prototype. The optimization of materials performed in WP5 resulted in two optimized fire-resistant geopolymers, the BFR (based on brick waste) and the CTFR (based on ceramic tile waste) and one thermal insulating, the CTTI (based on ceramic tile waste). Based on the combinations of these optimized geopolymers, two prototypes of the *DEFEAT-CM* are presented and discussed in this deliverable:

(i) the ***BFR-CTTI***, which consists of the optimum brick waste-based fire-resistant geopolymer (BFR) and the optimum ceramic tile waste-based thermal insulating geopolymer (CTTI)
(ii) the ***CTFR-CTTI***, which consists of the optimum fire-resistant and thermal insulating geopolymers, both based on ceramic tile waste (CTFR and CTTI, respectively)

The ***DEFEAT-CM prototypes*** are considered early samples for future commercialization of the innovative fire- and heat-resistant building façades developed in the project, utilizing construction and demolition waste (CDW). The prototypes were built to test the concept of the composite fire- and heat-resistant building façade, as well as to validate its production process, improve any imperfection and ensure reproducibility. For the production of the DEFEAT-CM prototypes, only the direct casting method was used, as the 3D-printing process, which was also proposed and studied in the project, presented significant difficulties in the printing of geopolymers (more details are given in the deliverable *D6.1-Report on the material engineering on the production method*).

The DEFEAT-CM prototypes and their production process were firstly designed and implemented at a small laboratory scale (small-scale) in the laboratory of the Frederick Research Center (FRC). The process flowsheet followed for their production is illustrated in Fig. 3 of the project deliverable *D6.2-Flowsheet of material production*. The samples of the small-scale prototypes were of dimensions 50x50x50 cm (cubic specimens) and used to test and validate the innovative building façade DEFEAT-CM and its production process at lab-scale, so as for both to reach a **Technology Readiness Level (TRL) of 4**.

The small-scale prototypes and the results from their lab-scale testing were used as reference for the implementation of the integrated DEFEAT-CM prototypes at a larger scale (up-scale). These activities took place at the laboratory of RECS, with the cooperation of FRC and UCY. The up-scale prototypes of the innovative DEFEAT-CM façade were prepared with dimensions of 15 cm length, 15 cm width and 5 cm thickness, of which 1.5 cm refers to the fire-resistant geopolymer and 3.5 cm refers to the thermal insulating geopolymer foam, following the same production flowsheet with the small-scale prototypes. The fire-resistant and thermal insulating geopolymers composing the DEFEAT-CM were integrated in one component, so that their configuration to be similar (or very close) to their final application, in almost all respects. The up-scale prototypes were used to validate the DEFEAT-CM and its production process at an relevant operating environment, in order for both of them to reach a **TRL 5**.

The DEFEAT-CM prototypes fulfilled the requirements of a complete testing case and eventually, validated the innovative DEFEAT building façade and its production process toward a reliable and affordable design of commercial products. The information included in this deliverable may also support several activities in WP7, since the technical information (mix designs, properties measurements, etc.) here presented can be used for the activities related to the production of the innovative DEFEAT-CM building façades at a pilot-scale and their demonstration in an operational environment.

2. Small-scale prototype of the *DEFEAT Composite Material*

2.1 Validation of the production process

Table 1 below summarizes the synthesis conditions followed to prepare the geopolymers combined into the small-scale DEFEAT-CM prototypes.

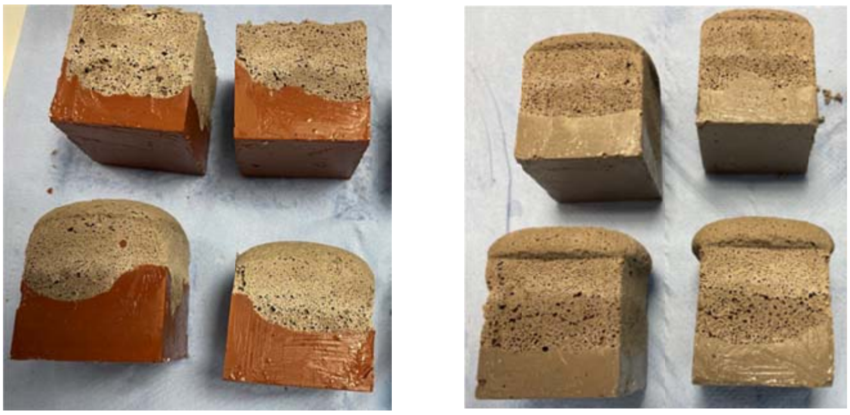
Table 1. Synthesis and curing conditions of the small-scale DEFEAT-CM prototypes

Parameter	**BFR**[(1)]	**CTFR**[(1)]	**CTTI**[(1)]
S/L ratio, g/mL	2.5	3.4	3.3
Alkaline solution / Concentration, Molarity	KOH / 8M	KOH / 8M	KOH / 8 M
Alkali activator	KOH and Na_2SiO_3 x H_2O solutions	KOH and Na_2SiO_3 x H_2O solutions	KOH and Na_2SiO_3 x H_2O solutions
$Na_2SiO_3xH_2O$ / KOH, v/v in the activator	1.6 : 1	1.6 : 1	1 : 1
Al powder, % wt	-	-	0.1
Curing temperature, °C	70	70	70
Curing time, days	7	7	7

[(1)]Abbreviations: BFR: waste brick-based fire resistant geopolymer (compact); CTFR: waste ceramic tile-based fire resistant geopolymer (compact); CTTI: waste ceramic tile-based thermal insulating geopolymer (foamed)

For the production of the small-scale prototypes, the casting method was used. Briefly, they were produced according to the following procedure:

First, the paste of the fire-resistant geopolymer was prepared and set aside. Next, the thermal insulating geopolymer paste was prepared, just before the addition of the foaming agent. Then, the fire-resistant geopolymer paste was mixed for 2-3 min and poured into the molds, forming a layer of 1.5 cm thickness. Immediately after that, the foaming agent (Al powder) was added in the thermal insulating geopolymer paste, mixed for 5-10 s and molded into the same molds as and above the fire-resistant layer, to a thickness of 3.5 cm. Once the foaming procedure was completed, the molds were placed in the oven that was pre-heated at 70 °C, and left for curing at this temperature for 7 day. After curing, the specimens of prototypes were demolded and used to validate the properties of interest. In Fig. 1, the small-scale DEFEAT-CM prototypes prepared as above-described are shown.



(a) (b)

Figure 1. Small-scale DEFEAT-CM prototypes (a) BFR-CTTI and (b) CTFR-CTTI.

As shown in Fig. 1, the interface of the two geopolymers did not form a plane parallel to the base of the cube, but in opposite, it was irregular and seems like the upper material (thermal insulating foam) to sink into the lower one (fire-resistant compact material). The lack of flatness at the interface of materials was observed in both the prototypes prepared (Fig. 1) and attributed to the low viscosity of both the fresh fire-resistant geopolymers (BFR and CTFR), which was not sufficient to withstand the stresses created by the fresh paste of the heat insulating geopolymer (CTTI), resulting in the sinking of the upper material into the lower one. The phenomenon was more intense at the fire resistant geopolymer based on waste bricks (BFR) and indeed, in the center of materials interface, since the vertices of the fire-resistant layer appeared to raise (Fig. 1a). In the CTFR-CTTI prototype, this interface irregularity was noticeably smaller (Fig. 1b).

In order to overcome this phenomenon, some modifications in the molding process of the composite material were decided: the paste of the fire-resistant geopolymer was prepared and left aside to set for 20 min at ambient temperature; this paste was mixed for 30 s and molded, when the paste of the thermal insulation geopolymer was prepared; then this last paste was molded over the first one. This setting time was considered adequate for the fire-resistant geopolymers paste to harden sufficiently, in order to form a flat surface during the bonding with the thermal insulation geopolymer. Before casting the thermal insulation geopolymer paste, the upper surface of the fire-resistant geopolymer was slightly etched, in order to improve the materials bonding. In addition, after the molding of prototypes, these were left at ambient conditions for 60 min, before putting them in the oven. In Fig. 2, the small-scale prototypes prepared with these modifications in their production process, are shown.

Figure 2. Small-scale DEFEAT-CM prototypes based entirely on waste ceramic tiles (CTFR-CTTI).

As seen in Fig. 2, the two geopolymers (compact and foamed) are bonded to a flat interface, which formed a plane parallel to the base of the cubic sample.

2.2 Verification of properties of the small-scale prototypes

In order to verify the significant properties of the fire-resistant and the thermal insulating materials in the *DEFEAT Composite Material* as well, the small-scale DEFEAT-CM samples were subjected to certain tests and measurements, including compressive strength and density. In order to verify their behavior in fire case, they were exposed to the elevated temperatures of 800 and 1000 °C for 2 hours (details on the experimental set-up and procedure followed in this test are given in the Final Periodic Report of the project, at the description of the WP5 activities). The compressive strength, density, mass loss and dry (linear) shrinkage of the exposed specimens were also tested and measured after this test.

In the following Table 2, the basic properties of the small-scale DEFEAT-CM components based entirely on waste ceramic tiles (CTFR-CTTI) are presented. For comparison purposes, the properties of the two individual materials that make up the composite one are also included in Table 2.

Table 2. Properties of the CTFR-CTTI small-scale prototypes and its individual materials

DEFEAT-CM small-scale prototype	Temperature °C	Density kg/m^3	Mass loss %	Dry linear shrinkage %	Compressive strength MPa
CTFR	50 (after curing)	1743	-	-	33.1
	800	1569	6.7	-	13.2
	1050	1578	9.9	-	36.6
CTTI	70 (after curing)	574		-	1.1
CTFR-CTTI	70 (after curing)	889	-	-	9.4
	800	896	7.5	1.5	12.9
	1050	853	8.0	4.0	10.2

According to the results presented in Table 2, it is obvious that the properties of the DEFEAT-CM are strongly affected by those of the thermal insulation geopolymer (CTTI). This is reasonable, taking into account that more than the two third of the composite material consist of the thermal insulation geopolymer foam. However, the compressive strength of the small-scale DEFEAT-CM samples after curing, as well as after exposure to elevated temperatures, is considered adequate for a building façade, since it was higher than the typical one of a gypsum board (2.4 – 2.7 MPa)[1]. Similarly, the density of the small-scale DEFEAT-CM samples is comparable to that of typical gypsum boards used as passive fire protective and thermal insulating building materials (600 - 1000 kg/m^3). The mass loss and dry shrinkage of the DEFEAT-CM samples are similar to those of the individual geopolymers, as expected.

Regarding the bonding of the two individual geopolymers in the DEFEAT-CM, it was excellent. The fire-resistant (compact) and the thermal insulating (foamed) geopolymer materials remained strongly bonded after the exposure of the prototype samples to 800 and 1050 °C, as shown in Figs 3 and 4, respectively. In addition, no cracks were observed on the surface of the small-scale prototype samples, after their exposure to the elevated temperatures.

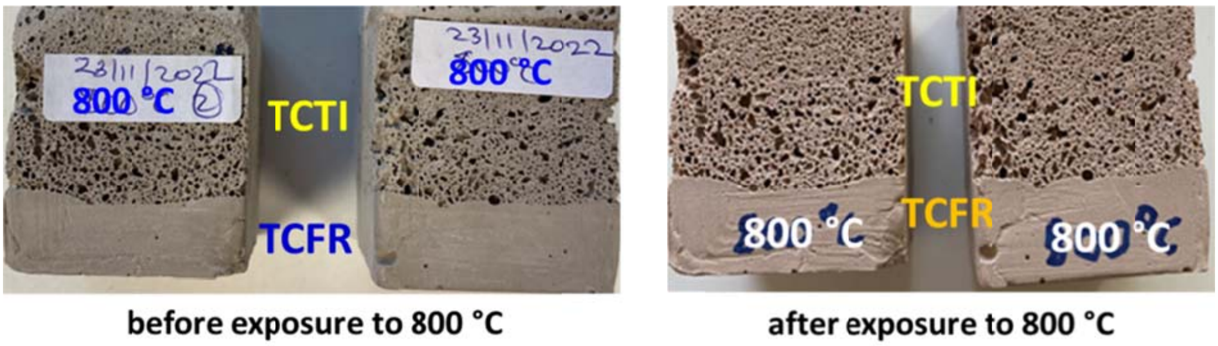


Figure 3. Small-scale CTFR-CTTI prototype before and after exposure to 800 °C.

[1]https://www.americangypsum.com/sites/default/files/2022-01/ga-235_gypsum_board_typical_mechanical_and_physical_properties.pdf

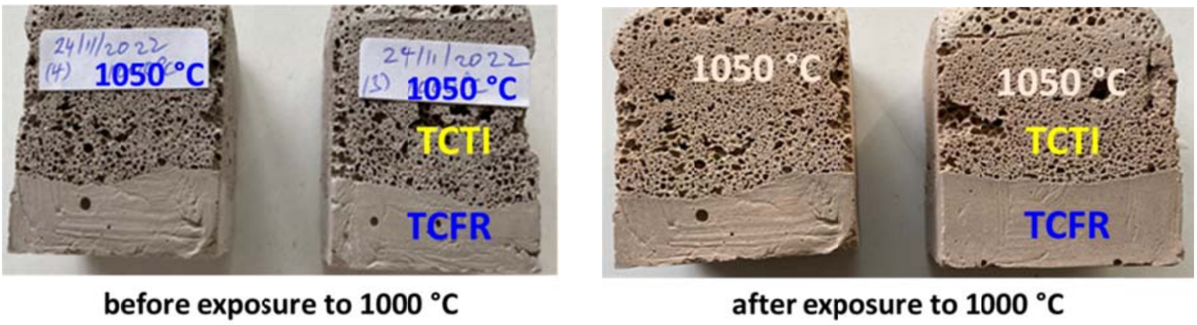


Figure 4. Small-scale CTFR-CTTI prototype before and after exposure to 1050 °C.

The good bonding of the two geopolymers in the CTFR-CTTI integrated component was also confirmed during the compressive strength testing of the relevant samples exposed to 800 and 1050 °C. As observed in the fragments of the small-scale CTFR-CTTI samples presented in Fig. 5, these were broken in the foam part, which developed very low to negligible compressive strength as an individual material, leaving the bonding area of the two geopolymers unaffected. It is worth noting that the direction of the load applied to the specimens tested was parallel to the interface of the two geopolymers composing the DEFEAT-CM.

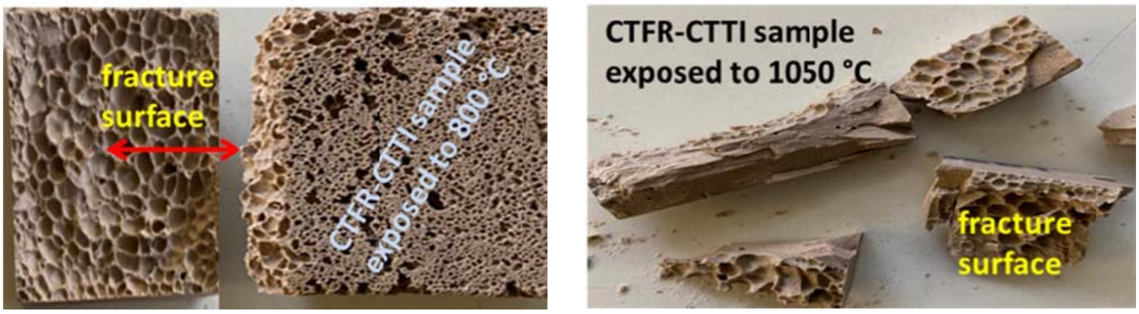


Figure 5. Fragments the CTFR-CTTI prototype samples exposed to 800 and 1050 °C and subjected to tests of compression strength.

According to the above-presented results, the small-scale prototypes correspond qualitatively and quantitatively the most important goals set in the project for the DEFEAT Composite Material.

3. Development of the *DEFEAT Composite Material* Prototype

3.1 Validation of the production process in large scale

The findings from the small-scale prototyping process of the *DEFEAT Composite Material* were used to produce the project prototype in the appropriate dimensions. For the up-scaling of the DEFEAT-CM, some modifications to the production parameters were performed, in order to improve the geopolymer pastes workability and the foaming process. These modifications were deemed necessary due to slight variation in the raw materials properties (small differences in chemical composition and particle size distribution), since these are solid wastes (waste bricks and tiles). For this reason, several lab-scale tests were carried out before achieving the desired workability and the suitable foaming of the geopolymer pastes for the production of the prototype. These tests resulted in a set of parameters for each individual geopolymer participating in the DEFEAT-CM. Based on them, two trials for each composite material developed in the DEFEAT project, the BFR-CTTI and the CTFR-CTTI, were carried out to verify the repeatability of the production process. Table 3 below summarizes the process parameters followed in these trials.

Table 3. Production process parameters of the up-scaled DEFEAT-CM prototypes

Process parameter	1st production trial		2nd production trial	
	Fire-resistant geopolymer			
	BFR(1)	**CTFR(1)**	**BFR(1)**	**CTFR(1)**
S/L ratio, g/mL	2.9	5.2	3.4	5.2
Alkaline solution / Concentration, Molarity	KOH / 8M	KOH / 8M	KOH / 8 M	KOH / 8 M
Solutions of the alkali activator	KOH and $Na_2SiO_{3\,x}H_2O$	KOH and $Na_2SiO_{3\,x}H_2O$	KOH and $Na_2SiO_{3\,x}H_2O$	KOH and $Na_2SiO_{3\,x}H_2O$
$Na_2SiO_3xH_2O$ / KOH, v/v in the activator	1.6 : 1	0.9 : 1	1.6 : 1	0.9 : 1
	Thermal insulation geopolymer			
	CTTI(1)	**CTTI(1)**	**CTTI(1)**	**CTTI(1)**
S/L ratio, g/mL	5	5	5	5
Alkaline solution / Concentration, Molarity	KOH / 8M	KOH / 8M	KOH / 8 M	KOH / 8 M
Solutions of the alkali activator	KOH and $Na_2SiO_{3\,x}H_2O$	KOH and $Na_2SiO_{3\,x}H_2O$	KOH and $Na_2SiO_{3\,x}H_2O$	KOH and $Na_2SiO_{3\,x}H_2O$
$Na_2SiO_3xH_2O$ / KOH, v/v in the activator	1 : 1	1 : 1	1 : 1	1 : 1
Al powder, % wt	0.5	0.5	0.5	0.5
	Curing process			
Curing temperature, ^oC	70	70	70	70
Curing time, hours	24	24	24	24

(1)Abbreviations: BFR: waste brick-based fire resistant geopolymer (compact); CTFR: waste ceramic tile-based fire resistant geopolymer (compact); CTTI: waste ceramic tile-based thermal insulating geopolymer (foamed)

Comparing the parameters values given in Table 3 for the DEFEAT-CM prototypes with those given in Table 1 for the small-scale prototypes, it is obvious that there are significant differences in the solid to liquid ratio, as well as in the ratio of the alkaline solutions in the activator. However, the DEFEAT-CM prototypes produced according to the data given in Table 3 for trials 1 and 2 looked macroscopically identical (Fig. 6), thus demonstrating the production process reproducibility.

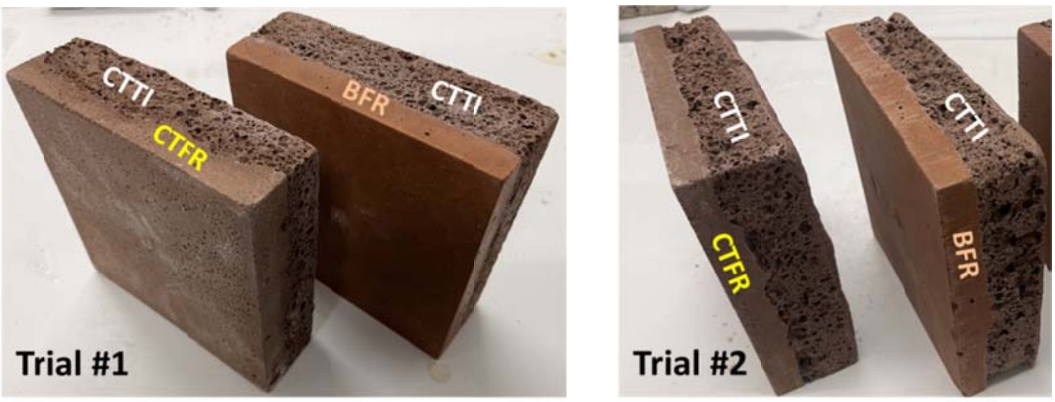


Figure 6. The DEAFET-CM prototypes.

3.2 Verification of properties of the up-scale prototypes

The density of the DEFEAT-CM was selected as a measure to verify the success of the prototype upscaling. In Table 4, the densities of the small-scale and up-scale prototypes of the BFR-CTTI and CTFR-CTTI composite materials are given. The estimated density values of the same components, based on the dimensions of the used specimens and their foam and compact sections is also included in Table 4.

Table 4. Density of the small-scale and up-scale DEFEAT-CM prototypes

	DEFEAT-CM Prototype	
	BFR-CTTI	CTFR-CTTI
	Density, kg/m^3	
Estimated	869	925
Small-scale	886	913
Up-scale	1008	960

As seen in Table 4, there are not significant deviations between the values of density that have been estimated and those of the small- or up-scale prototypes experimentally measured, as well as between the density values of the small- and up-scale prototypes. This finding is important for the validation of the DEFEAT-CM production process upscaling.

Except of density, the adhesion strength of the two individual geopolymers composing the DEFEAT-CM prototype was also measured through pull-off tests performed in accordance with EN1542. On these tests, the resistance of the two geopolymers to separate from each other (adhesive bonding) was also evaluated through the failure mode of the specimens tested. The dimensions of the specimens used in the tests were 150 x 150 x 100 mm; the fire-resistant geopolymer (compact) covered a specimen part with dimensions 150 x 150 x 30 mm, while the rest specimen part of 150 x 150 x 70 mm dimensions was filled from the insulating geopolymer (foam).

In Fig. 7, the apparatus and testing set-up used to measure the adhesion strength between the two individual geopolymers in the DEFEAT-CM prototypes, is shown (details are given in the Final Report of the DEFEAT project, in the section of WP5 activities). The tensile force applied to the specimens was continuously increased at a rate of 0.05 ± 0.01 MPa/s, until failure occurred. It is worth noting that, as the used apparatus was manually operated (Fig. 7), the operators took extra care to ensure that there were no significant fluctuations in the applied force. This was done to minimize any potential inconsistencies or variations that could affect the test results. According to the results of the pull-off tests, the maximum force applied to the specimens before they failed was below 100 N (which is a value below the recording capacity of the testing equipment), indicating that the bonding between the individual geopolymers was exceptionally weak. However, the closer observation of the specimens after the pull-off tests revealed that they failed in their foam section, next to the interface of the two materials, indicating strong adhesive bonding between them. The tested specimens of the CTFR-CTTI prototype presented in Fig. 8 confirm this observation.

Figure 7. Tensile bond strength equipment

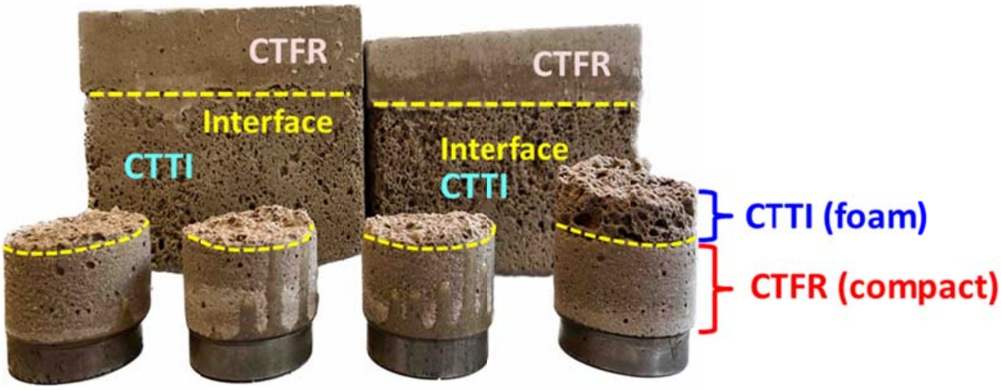


Figure 8. Specimens of the CTFR-CTTI prototype after the pull-off adhesion test.

As seen in Fig. 8, the failure surface of all four specimens of the CTFR-CTTI prototype was located in their foam part (thermal insulating geopolymer), right after the interface of the two geopolymers. The same failure mode was also observed in the small-scale prototypes described in Section 2 of this deliverable (Fig. 5). In general, the foam geopolymers developed in the DEFEAT project to be used as thermal insulation materials presented very low mechanical strength, which was expected as they consisted of 70-85% voids (details are given in the deliverable *D5.1-Report on the fire and insulation design*). Extremely low mechanical strength is common in this kind of geopolymers and is attributed to the foam structure with the high percentage of voids, which is the most significant cause of their failure even at very low loads.

The coloring of the DEFEAT-CM was also studied and verified to the up-scale prototypes, using either food colors or cement pigments. The colors were added into the alkali activator, before its mixing with the geopolymer precursor. The resulted colored specimens of prototypes are shown in Fig. 9.

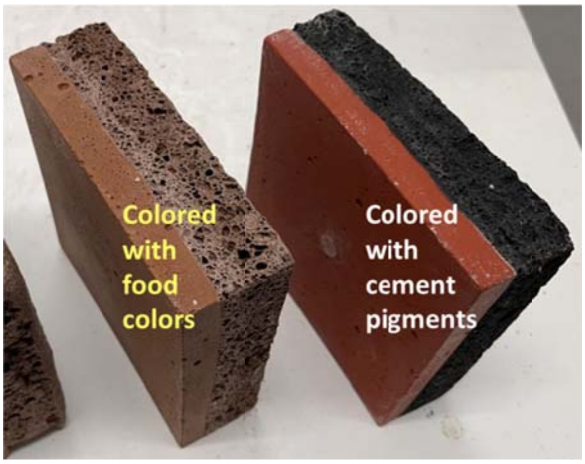


Figure 9. Colored specimens of the CTFR-CTTI prototype.

4. Conclusions

Deliverable *D5.3- Material prototype with fire and insulation properties in sandwich type* is dealing with the development and testing of the innovative DEFEAT Composite Material (DEFEAT-CM) prototype. Based on the optimization of the individual fire-resistant and thermal insulation geopolymers composing the DEFEAT-CM, two prototypes were implemented and validated. The casting production process used for the DEFEAT-CM was tested and validated in lab-scale and in a larger scale, as well. Based on this process, the production of the DEFETA-CM as an integrated component was implemented. The development of the DEFETA-CM prototype was implemented in two levels: in small laboratory scale and in large laboratory scale. The most important conclusions of this deliverable are the following:

In general, the DEFETA-CM prototypes developed in both small and large scale satisfied the requirements initially defined in the project. The experimental results of the prototypes’ testing provided evidence that the individual fire- and heat-resistant geopolymers combined in the DEFETA-CM can work together successfully in an integrated system.

The production process has been proved reliable and repeatable for the up scaling of the DEFEAT-CM. Moreover, the verification of the basic properties and adhesion bonding in the prototypes of the integrated DEFEAT-CM system are solid data for the commercialization process of the DEFEAT-CM.

A fine tuning of the very basic process parameters related to the workability and setting of the geopolymer pastes and the foaming process is necessary for the upscaling of the casting production process due to slight variations in composition and particle size that presented the waste materials used as geopolymer precursors (Construction and Demolition Waste, CDW).

The achieved TRL 4-5 for the DEFEAT-CM represents a bridge from the scientific lab-scale research to the innovative building facade engineering, while the experimental results taken could be used in the demonstration activities in WP7.

Acknowledgements

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