

Sustainable Materials for Energy Improvement and Fire Protection of Buildings

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Abstract— This paper deals with the development of innovative sustainable materials by utilizing Construction and Demolition Waste. The new materials are based on the alkali-activation of waste bricks and ceramic tiles and are foam and compact geopolymers with proper physical and mechanical properties to be used for heat insulation and the passive fire protection of buildings, respectively. The paper investigates the basic physical and mechanical properties of the developed geopolymers and compares them with the properties of conventional materials currently used in similar applications. The compact geopolymers were proved thermally stable upon firing at 600 °C, 800 °C and 1050 °C, appearing only negligible surface cracks. Their residual compressive strength was ranged from 20 to 35 MPa, while their mass loss was measured in between 3 and 9 %, after firing at the tested temperatures. The geopolymeric foams presented densities lower than 1000 kg/m³ with adequate mechanical performance. According to the results of this study, the new materials are promising to be used for passive fire protection and heat insulation of buildings. Further intensive research for the optimization of both kinds of materials is necessary.

Keywords— Geopolymer, Heat Insulation, Fireproofing Materials.

I. INTRODUCTION

In Europe, buildings are responsible for more than 40% of energy consumption and 36% of energy-related GHG emissions [1]. The most important cause of this issue is the low energy performance of buildings, especially of the older ones that were built without the relevant standards. More than 220 million building units in EU, representing about 85% of its building stock, were built before 2001, while 85-95% of the buildings exist today will still be standing in 2050 [1]. In order to achieve the goal set by the European Green Deal for climate neutrality in 2050, the renovation of the EU building stock is necessary as it will improve their energy performance, contributing thus to the reduction of energy demand and the related emissions. In addition to energy improvement, the passive fire protection of the EU building stock is essential. During the last years, several fires occurred in Europe, mainly in the Mediterranean countries, where the summers have become particularly dry, caused significant loss of human life and property and led to extensive structural damages with serious consequences for regional communities and economies.

The commercial heat insulating products mostly used in buildings are foams and boards based on extruded and

expanded polystyrene, polyurethane, mineral and glass wool and cellulose, as well as lightweight concrete based on expanded perlite. Among them, mineral wool and expanded perlite are also used as firewalls in buildings and constructions, since they can withstand also fire conditions [2-4]. Although the organic-based materials used as heat insulators present very low thermal conductivity (0.02 – 0.05 W/m·K), serious drawbacks, such as the high energy demand and the dangerous and toxic emissions in their manufacturing and the low diffusivity of moisture, prevent their widespread application. In addition, the high manufacturing cost and the large energy requirements of the inorganic-based thermal insulators limit their application in large constructions. Therefore, there is a need for new low-cost and sustainable materials with insulating and fire-resistance properties that could meet the needs for thermal insulation and fire safety of residential buildings.

In this direction, geopolymers are advantageous materials that combine low production costs with excellent performance. It concerns for pioneering inorganic polymers formed through the alkali activation of aluminosilicate sources rich in reactive silicate and aluminate phases. The resulted materials possess very good physical, mechanical and thermal properties, which are comparable to those of advanced materials currently used in buildings and constructions. The processing temperature of geopolymers is lower than 100 °C and their embodied energy and CO₂ footprint are extremely low compared to those of cement [5]. Therefore, geopolymers can serve as green construction materials with immense potential for sustainable development in applications with specific mechanical and thermal requirements.

The development of geopolymers with thermal insulation and fire resistance properties has been studied to a very limited extend. According to published research, fly ash from thermal power plants and slag from metallurgical industries are the most studied raw materials for the development of such geopolymers [6-17]. Kioupis et al. [6] studied the development of porous fly ash-based geopolymers, using metallic Si as foaming agent and achieved to produce lightweight geopolymers with apparent density of 840 – 1550 g/cm³ and compressive strength of 2.08 – 14.88 MPa. Ramos et al. [7] investigated the synthesis and properties of foam geopolymers based on blast furnace slag (BFS), using H₂O₂ as foaming agent and achieved to control the porosity of the obtained materials. The addition of microspheres [8] or

metakaolin [9] in fly ash geopolymeric precursors resulted in materials with density of 250-500 kg/m³, compressive strength of 520-683 kPa and thermal conductivity of 0.07-0.12 W/m·K.

Mixtures of fly ash and metakaolin were also investigated for the development of geopolymers with thermal stability at high temperatures [10]. However, the mechanical strength of the obtained materials was constantly decreased with the increase of temperature, while several cracks appeared on their surfaces from 300 °C. Similarly, geopolymers prepared using class F fly ash and Na- and K-based activators were found unsuitable for refractory insulating applications, as they showed high shrinkage and low thermal stability during firing tests taken place in the temperature range of 800-1200 °C [11]. In opposite, the compressive strength of geopolymeric pastes based on the alkaline activation of granulated Ca-BFS and exposed to temperatures from 200-1000 °C for 1 h, was constantly increased achieving almost twice the value that it had before the firing of materials [12]. Furthermore, the reverse-side temperature of BFS-based geopolymers exposed to 1100 °C flame equilibrated at less than 400 °C after 35 min [13-14]. Sakkas et al. [15-16] developed fire resistant geopolymers based on the activation of ferronickel slag with KOH solution, which were tested according to the RWS temperature-time curve and proved able to withstand successfully a fire under such severe conditions. Combinations of fly ash and slag in different ratios were also used to prepare geopolymers that were exposed at high temperatures, up to 800 °C [17]. The residual strength of the obtained materials was decreased exponentially as their initial strength was increased and this behavior was attributed to the decreased of the specimens' ductility observed with the increase of the initial compressive strength.

The present paper investigates the alkali activation potential of Construction and Demolition Waste (CDW). The focused materials are compact geopolymers with high compressive strength and thermal stability at high temperatures that are suitable for the passive fire protection of buildings and foam geopolymers with low density and satisfying mechanical strength to be used as thermal insulators in buildings. CDW is recognized in EU as a priority waste stream for recycling and reuse and although it is one of the heaviest and most voluminous solid wastes worldwide, its valorization remains a great challenge. CDW comprises a wide group of waste materials, including concrete, bricks, tiles, gypsum, wood, glass, metals, plastic, organic materials and excavated soil. The high SiO₂ and Al₂O₃ content in certain CDW streams such as bricks, ceramic tiles and concrete makes interesting their utilization in the production of geopolymers.

In this study, the geopolymerization of brick and ceramic tile wastes is investigated and the resulted materials are evaluated in terms of apparent density, mechanical strength and thermal stability at high temperatures, up to 1050 °C.

II. EXPERIMENTAL

A. Materials and Methods

Brick waste (BW) and ceramic tile waste (CTW) were used as geopolymeric precursors in this study. Both raw materials were obtained from a CDW recycling plant in Cyprus (Resource Recovery Cyprus – RRC) and sorted

manually. After crashing, the raw materials were grinded, using a Los Angeles ball mill with rotation speed 2000 round/s and subsequently, they were sieved to a particle size below 300 µm. The chemical composition of raw materials was determined according to the X-Ray Fluorescence Spectroscopy method (Spectro XEPOS XRF spectrometer). Their mineralogical analysis was performed by the X-Ray Diffraction method (D8 Bruker, CuK α radiation, $\lambda=1.5418$ nm, 2 θ /min) and the analysis of their particle size was carried out on a laser particle size analyzer (Malvern Mastersizer S500). Density and water absorption of raw materials were also determined according to the ASTM 128-88. The chemical composition of both raw materials is given in Table 1.

TABLE I. CHEMICAL ANALYSIS OF BRICK WASTE (BW) AND CERAMIC TILE WASTE (CTW).

Oxide	BW (% wt.)	CTW (% wt.)
SiO ₂	55.96	65.30
Al ₂ O ₃	14.97	15.36
FeO	10.64	8.98
CaO	8.04	1.55
MgO	4.25	3.85
K ₂ O	3.91	3.93
Na ₂ O	0.69	1.03
TiO ₂	1.53	-

As shown in Table 1, both raw materials are rich in silicon oxide (55.96 and 65.30 % wt. for BW and CTW, respectively) and in aluminum oxide (14.97 and 15.36 % wt. for BW and CTW, respectively), which indicates high potential for them to be used as geopolymeric precursors. Furthermore, FeO content is quite similar in both raw materials, while CaO content is much higher in BW than in CTW. The raw materials also contain similar amounts of MgO and K₂O and traces of Na₂O and TiO₂.

The mineralogical analysis of BW revealed feldspars (anorthite-CaAl₂Si₂O₈ and albite-NaAlSi₃O₈) and quartz (SiO₂) as the main phases and diopside (MgCaSi₂O₆) and hematite (Fe₂O₃) as secondary. According to XRD analysis of CTW, it was mostly dominated by quartz (SiO₂) with minor contributions of feldspars (anorthite-CaAl₂Si₂O₈ and orthoclase-KAlSi₃O₈) and hematite (Fe₂O₃). A slightly amorphous phase of Si-Al composition was also present in CTW.

According to the particle size distribution analysis, CTW had particle size lower than 300 µm with mean diameter $d_{50} = 48.34$ µm, while BW was slightly finer than the CTW, since it had particle size lower than 250 µm with mean diameter $d_{50} = 35.35$ µm. Both raw materials had quite similar bulk density (1.63 g/cm³ for BW and 1.82 g/cm³ for CTW), while the water absorption was much higher for BW (25.57 %wt.) than for CTW (15.77 % wt.).

For the alkali-activation of the geopolymeric precursors, alkaline solutions prepared by mixing sodium silicate solution (Na₂O = 8%, SiO₂ = 27%) and potassium hydroxide (KOH) solution were used. The KOH solution was prepared by dissolving KOH anhydrous pellets of analytical grade (99.5% purity) in distilled water and its concentration was selected to

be equal to 8 mol/L (M) in all the experiments, considering that at lower alkaline concentrations the content of OH⁻ ions is inadequate to facilitate the dissolution of silicate and aluminate phases and therefore promote geopolymerizations [5]. For the preparation of the foam geopolymers, aluminum powder (-325 mesh, 99.5% purity) and/or hydrogen peroxide (H₂O₂) solution (30% wt.) were used as foaming agents.

To prepare both foam and compact geopolymers, the alkaline activating solution was first mixed with the geopolymer precursor in a laboratory mixer, until to obtain a homogeneous paste. The formed paste was poured in cubic molds (50 x 50 x 50 cm) and left for curing. The curing procedure of both types of developed geopolymers, foam and compact, took place in an oven, operating at 70 °C for 7 days for the foam geopolymers and at 50 °C for 5 days in the case of the compact ones. When curing was completed, the specimens were demolded and left for ageing at room temperature. After an ageing period of 7 days, the compressive strength, apparent density and porosity of the developed geopolymeric foams was determined. The compact geopolymers were tested for compressive strength, apparent density, shrinkage and mass loss after ageing for 7 days and 28 days. All tests and measurements were carried out in triplicate and the mean values are reported in the relevant tables and figures included in this study.

B. Tests and Analyses

The compact geopolymers were exposed at 600 °C, 800 °C and 1050 °C for 7 h, using a laboratory-scale muffle furnace with the capacity of 1200 °C max temperature. After each firing test, the specimens were left at ambient conditions for ageing periods of 7 days and 28 days, before compressive strength, density, shrinkage and mass loss to be determined.

The compressive strength of materials was tested on a 2000 KN electro-hydraulic mechanical testing machine, by applying load of 6 MPa/s. The shrinkage of specimens was calculated as percentage of their initial volume and the apparent density of materials was determined as the ratio of mass to volume of specimens. The measurements of specimens' mass were also used to determine the mass loss of materials, after being exposed at the different temperatures. Before the mass and volume measurement, all specimens were dried in an oven at 105 °C for 24 h.

III. RESULTS AND DISCUSSION

A. Thermal Stability of Geopolymers

The compact geopolymers developed in this study are intended for use in the passive fire protection of buildings and therefore, their mechanical strength and structural stability at high temperatures are considered particularly important. Table 2 below presents the synthesis conditions of the compact geopolymers developed in this study.

TABLE II. EXPERIMENTAL CONDITIONS OF COMPACT GEOPOLYMERS PREPARATION.

Parameter	BFR	CTFR
S/L, g/mL	2.5	3.4
[KOH], M	8	8
Na ₂ SiO ₃ / KOH ratio	1.6: 1	1.6: 1
Curing time, days	7	7
Curing temperature, °C	50	50
Firing time, h	2	2
Firing temperature, °C	600, 800, 1050	600, 800, 1050

In Figure 1, the compressive strength of the brick- and ceramic tile- based compact geopolymers (BFR and CTFR, respectively) after curing and their exposure at elevated temperatures is plotted as a function of temperature and ageing time.

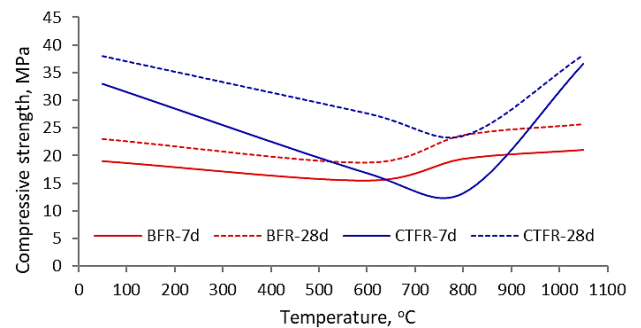


Fig. 1. Compressive strength of BFR and CTFR compact geopolymers versus curing and firing temperature.

As seen in Fig. 1, the compressive strength of CTFR geopolymers after curing was substantially higher than that of the BFR materials. Actually, compressive strength of the CTFR geopolymer was 33 MPa and 38 MPa after ageing for 7 days and 28 days, respectively, while the corresponding values of compressive strength for BFR geopolymers were 19 MPa and 23 MPa, respectively. The existence of the amorphous aluminosilicate phase in the CTW raw material provided the geopolymeric system with easily dissolved Si and Al, which enhanced the development of the Si- and Al-based polymerized network in the materials matrix [18-20], leading to the production of more compact and stable structures. Furthermore, the high calcium content in the BW raw material favored the formation of C-S-H gel instead of the N-A-S-H one (where C=Ca, N=Na, A=Al, S=Si and H=H₂O) that characterizes the geopolymers [21], keeping thus relatively low the strength of the BFR material. The ageing process also improved compressive strength (Fig. 1), as it favored the formation of more ordered geopolymeric networks and denser materials.

According to Fig. 1, the exposure of geopolymers at high temperatures affected the compressive strength, especially in the case of the CTFR geopolymer. More precisely, the residual compressive strength of CTFR was substantially reduced up to 800 °C and then, it was sharply increased as temperature raised to the highest value tested (1050 °C). At this temperature, the reached compressive strength was slightly higher than the initial one, regardless the ageing period (7d or 28d). The residual compressive strength of BFR

geopolymer followed a similar trend, but with less abrupt changes (Fig. 1). It was slightly decreased up to 600 °C and then, increased up 800 °C and remained almost unchanged up to 1050 °C. The decrease of compressive strength can be related to the softening of the geopolymeric matrix, which happens at about 550-650° C [22], while the improve of compressive strength at 1050° C can be attributed to the sintering process taken place between the unreacted solid particles and the melted geopolymeric binder, which results in more durable structures [23].

The density of both BFR and CTFR geopolymers has been remarkably decreased after their exposure at 600 °C. Actually, the density of BFR decreased from 1550 kg/cm³ to 1430 kg/cm³ and that of CTFR, from 1736 kg/m³ to 1580 kg/m³, as the temperature was increased from ambient to 600 °C. Then on, the density of both geopolymers remained almost unchangeable up to 1050 °C. Moreover, both materials presented very low mass loss, in the range of 6 – 9 %wt. and negligible shrinkage, after their exposure at the temperatures tested in this study.

During firing up to 1050° C, there were no signs of spalling or deformation for both studied geopolymers. Only small size cracks appeared on the specimen's surface of both materials at 800 °C, which became more intense and longer after their exposure at 1050 °C.

Figure 2 below compares the maximum application temperature of commercial heat insulating materials, which are also used today as fire barriers in buildings and constructions [24], with the compact geopolymers developed in this study.



Fig. 2. Maximum temperature of application for typical insulating materials used also as fire resistant [24] (BFR and CTFR are the geopolymers developed in this study).

According to Fig. 2, the geopolymers developed in this study can operate at higher temperatures than calcium silicate, which is considered as an excellent fire resistant materials and therefore, they have a great potential to be used for the passive fire protection of buildings and other structures.

B. Properties of Foam Geopolymers

Table 3 below summarizes the experimental conditions followed for the development of the geopolymeric foams.

TABLE III. EXPERIMENTAL CONDITIONS OF FOAM GEOPOLYMERS

Parameter	BF	CTF
S/L, g/mL	1.8; 2.1; 2.2; 2.5; 3.0	2.1; 2.2; 2.4; 2.6; 3.3
[KOH], M	8	8
Na ₂ SiO ₃ / KOH ratio	1: 1	1: 1
Al powder, % wt (in paste)	0.1; 0.5; 2.0	0.1; 0.2; 0.4
H ₂ O ₂ , % vol (in activator)	2.2; 2.4; 3.0	3.4; 4.0; 5.0
Curing time, days	7	7
Curing temperature, °C	70	70
Ageing time, days	7	7

In Figures 3 and 4, the apparent density and the compressive strength of BF and CTF foam geopolymers are plotted versus the quantity of the foaming agent added, Al powder and H₂O₂ solution, respectively.

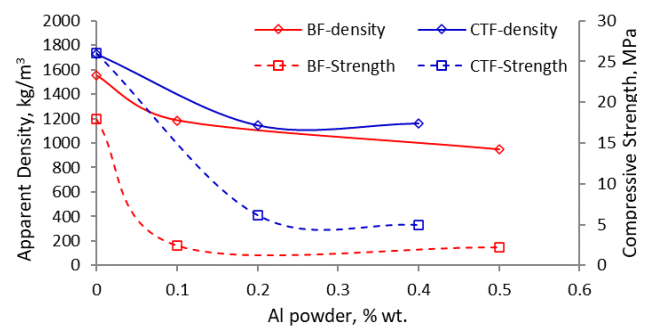


Fig. 3. Apparent density and compressive strength versus the Al powder additions in the geopolymeric paste for the BF geopolymers at S/L = 2.1 g/mL and for CTF geopolymers at S/L = 2.6 g/L.

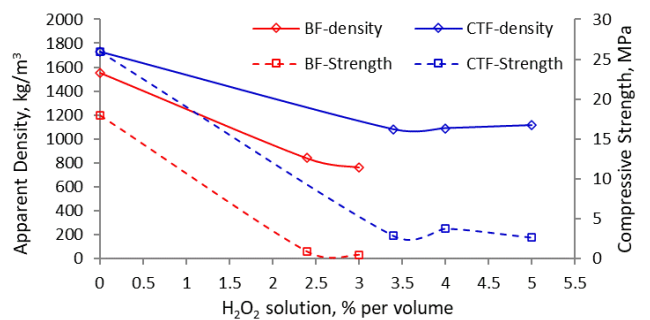


Fig. 4. Apparent density and compressive strength versus the H₂O₂ solution additions in the geopolymeric paste for the BF geopolymers at S/L = 2.1 g/mL and for CTF geopolymers at S/L = 3.3 g/L.

As seen in Figs 3 and 4, the density of the foam geopolymers decreased with the increase of foaming agent. Moreover, the decrease of the materials density decreased their compressive strength, as well. This relationship between density and compressive strength is reasonable, as the decrease of materials density involves the increasing of their porosity and consequently, the decrease of the mass per unit volume that affects the materials cohesion. As known, the porosity of materials changes reversibly to their density and increases as the density decreases.

Porosity expresses the voids that exist per unit volume of a material and is an important index for lightweight materials. Due to porosity, materials become lighter. However, the increase of porosity has an upper limit for the foam materials.

Further increase of porosity by this upper limit involves the formation of huge size pores that expanded uncontrollably, destroying the foamed matrix of the material. Porosity and consequently density affects also the thermal properties of materials. According to relevant publications, there is a linear relationship between the thermal conductivity coefficient and the density of geopolymeric foams [22-25]. Therefore, foam geopolymers with low density is expected to have very good thermal conductivity.

Based on the experimental results given in Figs 3 and 4, it is possible to develop geopolymer foams with densities lower than 600 kg/m³ by using WB and CTW raw materials. The authors of this paper currently focus on the development of foamed geopolymers with density close to 450 kg/m³ and thermal coefficient < 0.1 W/(m·K). Given that the geopolymeric materials are sustainable, non-combusting, non-smoking and not-toxic materials, achieving thermal conductivity similar to that of the commercial foam concrete (0.2 W/m·K) [26] with satisfying mechanical properties (about 2 MPa) comprises an important challenge for further improvement and up-scaling of the foam geopolymers developed in this study.

IV. CONCLUSIONS

Brick waste (BW) and ceramic tile waste (CTW) from CDW are suitable precursors for the development of geopolymers to be used as fire-resistant and heat insulating materials in buildings.

The compact geopolymers BFR and CTFR developed in this study were proved thermally stable after exposure at temperatures up to 1050 °C for 2 h. The temperatures 600 °C and 800 °C were considered as critical for the properties of BFR and CTFR, respectively; after their exposure at these temperatures, density and compressive strength were decreased. At 1050 °C, the compressive strength of both materials was improved and reached almost the initial one that they had developed after curing. The changes in mechanical strength were attributed to structural transformations occurred in the geopolymeric matrix of materials.

The foam geopolymers prepared by both foaming agents used in this study, Al powder and H₂O₂ solution, achieved densities lower than 1000 kg/m³ with satisfying mechanical strength. However, further research is necessary to achieve materials with properties comparable to those of the commercial products.

The geopolymers developed in this study have the potential to be effectively used for the passive fire protection and the thermal insulation of buildings. As density and compressive strength of both types of materials are crucial parameters for their application in buildings, it is necessary to define their exact use and the relevant specifications for their further investigation and up-scaling.

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