

Article

Development of Lightweight Geopolymer Composites by Combining Various CDW Streams

Dimitrios Kioupis ^{1,2,*} , Aggeliki Skaropoulou ¹, Sotirios Tsivilis ¹  and Glikeria Kakali ¹
¹ School of Chemical Engineering, National Technical University of Athens, 15773 Athens, Greece

² Engineering School, Merchant Marine Academy of Aspropyrgos, 19300 Athens, Greece

* Correspondence: dkioupis@mail.ntua.gr; Tel.: +30-2107723284

Abstract: This study regards the development of lightweight geopolymer composites through the valorization of various construction and demolition wastes. Brick waste was utilized as the sole aluminosilicate precursor for the geopolymerization reactions, expanded polystyrene and polyurethane wastes were used as artificial lightweight aggregates, and short polyethylene fibers developed from CDWs reinforced the geopolymer matrix. The curing conditions of the geopolymer synthesis were optimized to deliver a robust geopolymer matrix ($T = 25\text{--}80\text{ }^{\circ}\text{C}$, $t = 24\text{--}72\text{ h}$). Both raw materials and products were appropriately characterized with XRD and SEM, while the mechanical performance was tested through compressive strength, flexural strength, Poisson's ratio and Young's modulus measurements. Then, a comprehensive durability investigation was performed (sorptivity, wet/dry cycles, freeze/thaw cycles, and exposure to real weather conditions). In contrast to polyurethane waste, the introduction of expanded polystyrene (0.5–3.0% wt.) effectively reduced the final density of the products (from 2.1 to 1.0 g/cm³) by keeping sufficient compressive strength (6.5–22.8 MPa). The PE fibers could enhance the bending behavior of lightweight geopolymers by 24%; however, a geopolymer matrix–fiber debonding was clearly visible through SEM analysis. Finally, the durability performance of CDW-based geopolymers was significantly improved after the incorporation of expanded polystyrene aggregates and polyethylene fibers mainly concerning freeze/thaw testing. The composite containing 1.5% wt. expanded polystyrene and 2.0% v/v PE fibers held the best combination of properties: Compr. Str. 13.1 MPa, Flex. Str. 3.2 MPa, density 1.4 g/cm³, Young's modulus 1.3 GPa, and sorptivity 0.179 mm/min^{0.5}.

Keywords: lightweight geopolymers; CDWs; expanded polystyrene waste; polyurethane waste; polyethylene waste; curing conditions; mechanical properties; microstructure; durability



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1. Introduction

Construction and demolition wastes (CDWs) are one of the massive and the most voluminous waste streams generated in the European Union (EU) and globally [1,2]. More specifically, CDWs constitute over 35% of all waste generated by different economic activities in EU (Figure 1a) and they are associated with operations occurring in the construction sector such as the excavation, construction, and demolition of buildings [3]. CDWs are composed of numerous materials such as cement, bricks, plaster, wood, glass, metals, plastic, asbestos and soil, tiles, treated marble, etc., many of which are recyclable. A typical composition [3] is shown in Figure 1b.

Construction and demolition wastes (CDWs) have been classified as a “priority waste stream” by the European Union [4], both from an environmental and economic point of view, as nowadays, they are of considerable interest for recycling and reusing because many of these materials have a high economic value. Furthermore, CDWs have a high environmental impact, mainly due to the large volume of waste that has to be managed. Their disposal is associated with impacts such as the degradation of the natural environment, visual pollution, the production of dust–air pollution, the pollution of surface and groundwater, soil pollution, risks to human health, etc. [5].

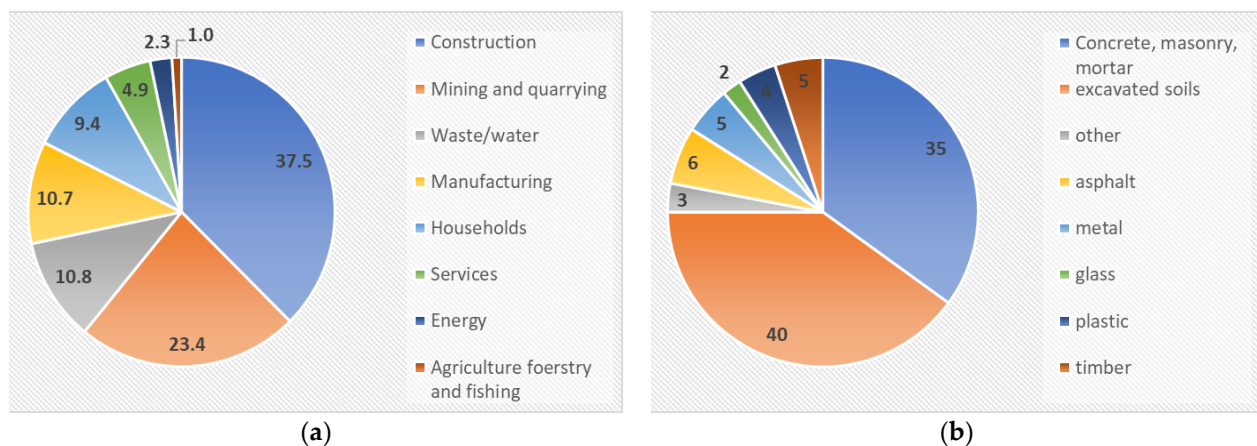


Figure 1. (a) Waste generation by economic activities and households, EU, 2020 (% share of total waste) [3] and (b) typical composition of CDWs [3].

In the European Union, CDW management policy is reflected in a series of measures and directives, the main one being the revised Waste Framework Directive 2008/98/EC, amended 2018/851 [6], which aims at the prevention, recovery, and proper disposal of waste. Following this directive, the average recovery rate in the EU significantly increased to 89% for the year 2020 [3].

To date, the most widespread practice of recycling the mineral part of CDWs is its use as aggregates in various construction projects in order to reduce the consumption of natural resources [7,8]. Indeed, concrete waste has replaced natural coarse aggregates in concrete production, thus increasing the sustainability of concrete technology [9–14]. However, more efforts such as the aforementioned are required to utilize the full recycling potential of CDWs to develop high-added-value products.

The fact that a large part of mineral CDWs are of aluminosilicate origin reveals the great potential of using such wastes as raw materials in the production of geopolymers. Geopolymers [15–17] are sustainable building materials valorizing waste and by-products generated by other industries as raw materials, and thus cut down the consumption of natural resources while at the same time obtaining a smaller environmental footprint than conventional cement [16,18,19]. Recently, several scientific papers have been published [20–26] where concrete and masonry wastes were applied as geopolymer precursors. In most studies, these CDWs are used in combination with supplementary cementitious materials (SCMs) such as ground granulated blast furnace slag [27–30], ferronickel slag [31], OPC [23,32–34], silica fume [35], metakaolin [23,29,36–38], and fly ash [29,38–43]. In all these cases, SCMs play the role of the main active precursor.

However, the synthesis of geopolymers through the use of a CDW source as the sole aluminosilicate precursor is crucial to explore their full geopolymerization potential and, therefore, to maximize their recycling rate, according to EU legislations. A number of studies have dealt with the aforementioned aspect, mainly focused on the synthesis optimization of pastes and mortars [22,33,44–49]. Particularly, parameters such as the type and processing of the CDW precursor, the applied curing conditions, and the type and concentration of the alkali activator were evaluated in terms of microstructure, fresh state, and mechanical properties. Our group has also studied the CDW brick geopolymer synthesis through the performance of a multifactorial design of experiments in a previous study, highlighting the significant parameters of the synthesis [50].

Although considerable progress has been made in CDW synthesis optimization, the literature concerning the development of CDW-based products and their service life performance is limited. A few works so far have dealt with the performance of CDW geopolymer concrete [51], and the potential application of CDWs in the production of bricks, pavers, tiles [25,52], geopolymer composites [53,54], or 3D-printable mixtures [55]. Therefore,

further research in this field is required to explore the application capabilities of CDW-based geopolymers.

A major application field concerns the lightweight building materials market. These materials have had increased demand in recent years since they have numerous advantages such as superior thermal and acoustic performance, the minimization of construction dead load, the construction time, and the labor expenses involved in handling or transportation, and are responsible for significant energy savings within buildings [56–59].

Furthermore, a vital part of building materials' service life is the assessment of their durability performance. It is well known that geopolymer products exhibit similar and, in some cases, better durability performance in relation to ORC [60]. More specifically, studies in the literature show that the geopolymers derived from certain wastes have superior performance when exposed to high temperatures [61], aggressive environments (acids, chlorides, and sulfates) [62–65], and weathering (humidity, freezing, etc.) [63,66,67]. Therefore, it is highly important to conduct such tests in order to assess the effect of geopolymers composition on their long-term performance. Concerning the durability of CDW-based geopolymers, their water absorption [68] and behavior in acid environments [69] and high temperatures [31] have been examined so far for samples prepared with different synthesis parameters, pointing out that a more comprehensive investigation is needed to fully define the durability of the produced materials.

The main scope of this study is to develop durable, lightweight, CDW-based geopolymer composites that can be applied as building materials in the construction sector. The synthesis of the composites was performed using the combinational valorization of four different CDW streams, namely, bricks rejected during the manufacturing process and expanded polystyrene, rigid polyurethane, and polyethylene wastes from construction and demolition activities. To the best of authors' knowledge, this is the first attempt to combine such wastes to produce lightweight building materials with a high CDW recycling rate and low embodied energy.

After the appropriate processing of the CDW materials, brick waste was used as the sole aluminosilicate precursor of the geopolymer synthesis, expanded polystyrene and polyurethane wastes played the role of lightweight aggregates, while PE waste in the form of short plastic fibers reinforced the geopolymer matrix. The curing conditions were also investigated to maximize the geopolymerization of brick wastes. Apart from examining the microstructure and physicochemical and mechanical properties of the composites, a comprehensive experimental setup for the durability investigation was performed to point out variations in the long-term performance depending on the geopolymer synthesis. The combination of brick, EPS, and PE wastes in certain amounts can effectively produce durable lightweight geopolymer composites with a high CDW content (>70%).

2. Materials and Methods

2.1. Raw Materials

Brick waste (BW) used as an aluminosilicate source comes from rejected products of the brick industry that do not conform to quality standards. The brick waste was supplied from NR-GIA BUDOWNICTWO Sp. z o.o. (Poland). Table 1 and Figure 2 present the chemical and mineralogical composition of BW measured using XRF and XRD analysis, respectively. The XRD analysis of a BW geopolymer is also presented in Figure 2, showing no significant changes in the mineralogical content among the precursor and product. The BW was pulverized prior to its use to obtain a mass median particle diameter (d_{50}) of 20 μm (Figure 3). This fineness is in the typical range for raw materials used in the industrial formulation of cementitious materials.

Table 1. Chemical composition of BW.

Source	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	TiO ₂	P ₂ O ₅	Cl	LOI
BW	61.02	18.85	7.87	1.73	3.03	3.40	1.27	0.26	0.89	0.17	0.02	1.32

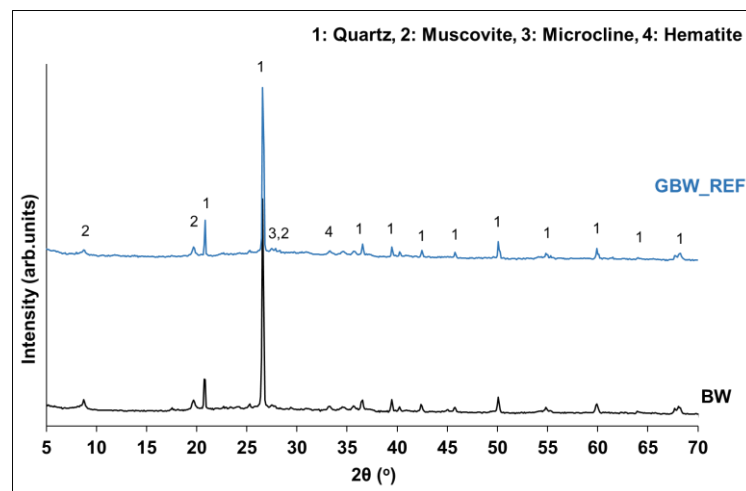


Figure 2. XRD pattern of BW.

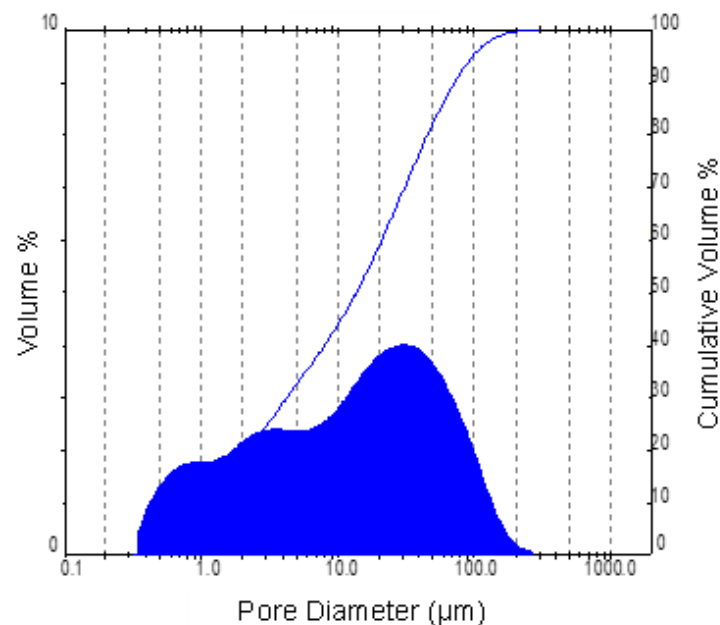


Figure 3. BW particle size distribution.

Expanded polystyrene (EPS) and polyurethane foam (PU) wastes are applied as lightweight aggregates to produce geopolymers of reduced density. These wastes come from construction and demolition activities of the building sector and were provided by NR-GIA BUDOWNICTWO Sp. z o.o. (Poland). The wastes were appropriately processed prior to their use in order to obtain the desired particle size. In particular, fragments of EPS and PU wastes were broken by hand into small pieces (<10 cm) and then were shredded in an appropriate shredding machine. Finally, the resulting material was sieved to obtain aggregates with particle diameters between 1 and 3 mm. This particle size was based on a previous study [70], where commercial EPS beads with the same nominal particle size effectively reduced the density of fly-ash-based geopolymers. Table 2 shows the characteristic properties of EPS and PU aggregates.

Table 2. Properties of lightweight aggregates.

Aggregate	Density (g/cm ³)	Water Absorption (%)	UCS (MPa)	λ (W/mK)	Particles with $d > 2$ mm (%)
EPS	0.038	1.0	0.46	0.035	97.0
PU	0.051	<2.0	0.21	0.025	78.6

The reinforcement of the BW geopolymer matrix was carried out through the incorporation of short plastic fibres developed from polyethylene (PE) C&D wastes (Leitat Technological Center, Terrassa, Spain). The developed PE fibres were alkali-resistant and, therefore, compatible with the chemistry of geopolymers. The crucial PE fibre properties are presented in Table 3. Representative images of the raw materials used for the development of the BW geopolymer composites are shown in Figure 4. All CDW materials (BW, EPS, and PU), as well as the PE fibres, were collected, processed, and developed in the framework of the GreenInstruct project [71,72].

Table 3. PE fibers' properties.

Property	Value
Length (mm)	6–12
Maximum diameter (μm)	331
Minimum diameter (μm)	114
Average diameter (μm)	213
Diameter—standard deviation (μm)	46
Density (g/cm ³)	0.97
Tensile modulus (MPa)	258.60

**Figure 4.** CDW materials used in this study before (up) and after (down) processing: (a) BW, (b) EPS, (c) PU, (d) PE.

2.2. Geopolymer Preparation

The first step of geopolymer synthesis includes the preparation of the activation solution that provides the appropriate alkalinity for the dissolution of the aluminosilicate precursor and, at the same time, the charge-balancing ions (Na^+ or K^+) that will participate in the formation of the aluminosilicate matrix. Apart from the alkaline species, the presence of adequate soluble silicon amounts in the activation solution is crucial for the development of a robust geopolymer matrix. Therefore, the activation solution was prepared through the dissolution of NaOH (>99%, CAS: 1310-73-2) and KOH (>85%, CAS: 1310-58-3) anhydrous pellets in water, and then the addition of soluble Si in the form of SiO_2 solution (50% in H_2O , colloidal dispersion, CAS: 7631-86-9). The mixing of the geopolymer precursor (BW)

with the activation solution and the aggregates/fibers was performed with a standard mortar mixer (Controls 65-L0005). Then, the prepared geopolymer slurries were casted in metallic molds, cured at 25–80 °C for 24–72 h, and, finally, demolded and kept in plastic bags. The geopolymers' preparation was based on the optimization of BW geopolymer synthesis reported in a previous study using the following synthetic ratios: Si/Al = 3.0 (the Si/Al molar ratio expresses the overall amount of Si involved in the geopolymerization process originating from both the brick precursor and activation solution), R/Al = 0.75 (the R/Al molar ratio expresses the alkalinity of the activation solutions in terms of the Na and K contents), Na/Na+K = 0.35 (the Na/Na+K molar ratio expresses the kind of the alkali in the activation solution), and solids/liquids = 5.2 (solids/liquids is the mass ratio that expresses the workability of the geopolymer pastes related the total solid mass with water mass) [50]. Therefore, the composition of the geopolymer matrix in all of the samples was kept constant: 70.1% wt. BW, 7.9% wt. SiO₂ solution, 2.7% wt. NaOH, 7.1% wt. KOH, and 12.2% wt. H₂O.

In the first part of the experiment, the curing conditions of the geopolymer synthesis were optimized in order to maximize the geopolymerization of the BW. More specifically, reference geopolymer specimens were prepared and cured at varying conditions ($T = 25, 50$, or 80 and $t = 24, 48$, or 72 h) and then mechanically tested under compression and bending. Then, lightweight geopolymers were prepared with the incorporation of EPS (0.5–3.0% wt.) and PU (1.0–6.0% wt.) aggregates following the same synthesis procedure as before. The aggregates were added to the mixes after the preparation of the BW geopolymer slurries. Finally, the incorporation of fibers was examined to improve the mechanical behavior under bending. The composites were developed by incorporating the PE fibers at varied contents (0.5–2.0% v/v). As in the case of lightweight-geopolymer preparation, PE fibers were added to the geopolymer mixes after the preparation of the geopolymer slurries.

Figure 5 presents a flowchart of the experimental procedure, while Table 4 presents the geopolymer mixes prepared in this study.

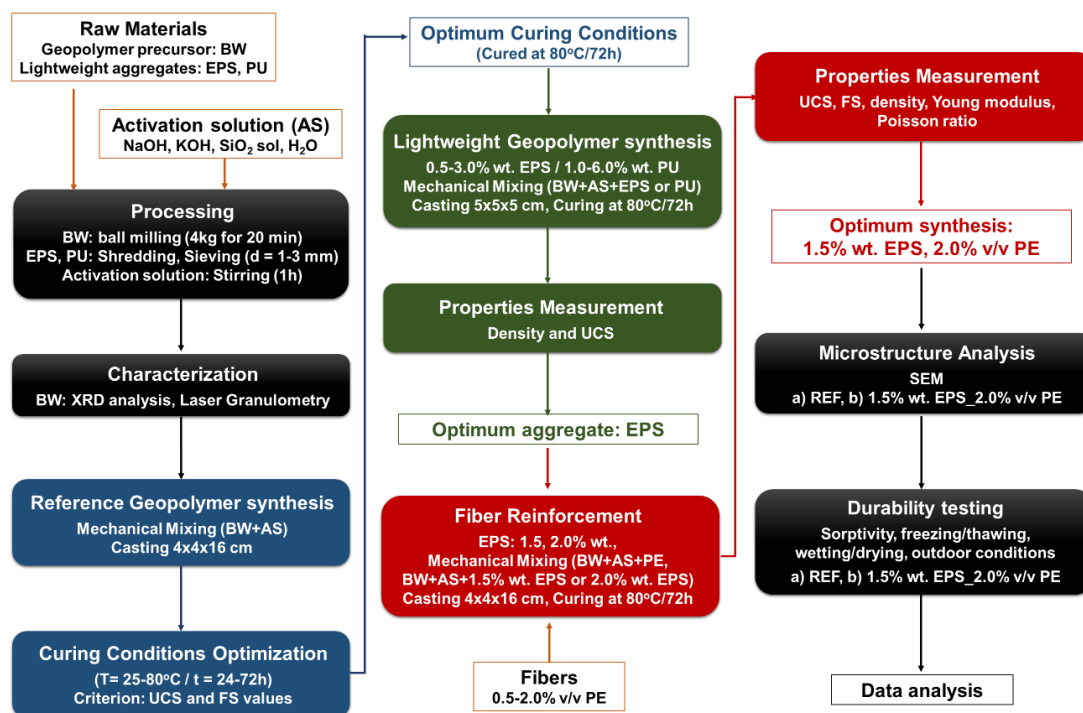


Figure 5. Flowchart of experimental procedure.

Table 4. BW geopolymer mixes prepared in this study.

Samples	EPS * (% wt.)	PU * (% wt.)	PE Fibers (% v/v)
GBW_REF	-	-	-
GBW_0.5EPS	0.5	-	-
GBW_1.0EPS	1.0	-	-
GBW_1.5EPS	1.5	-	-
GBW_2.0EPS	2.0	-	-
GBW_2.5EPS	2.5	-	-
GBW_3.0EPS	3.0	-	-
GBW_1.0PU	-	1.0	-
GBW_2.0PU	-	2.0	-
GBW_3.0PU	-	3.0	-
GBW_4.0PU	-	4.0	-
GBW_5.0PU	-	5.0	-
GBW_6.0PU	-	6.0	-
GBW_REF_0.5PE	-	-	0.5
GBW_REF_1.0PE	-	-	1.0
GBW_REF_1.5PE	-	-	1.5
GBW_REF_2.0PE	-	-	2.0
GBW_1.5EPS_0.5PE	1.5	-	0.5
GBW_1.5EPS_1.0PE	1.5	-	1.0
GBW_1.5EPS_1.5PE	1.5	-	1.5
GBW_1.5EPS_2.0PE	1.5	-	2.0
GBW_2.0EPS_0.5PE	2.0	-	0.5
GBW_2.0EPS_1.0PE	2.0	-	1.0
GBW_2.0EPS_1.5PE	2.0	-	1.5
GBW_2.0EPS_2.0PE	2.0	-	2.0

* Brick-waste-based.

2.3. Mechanical Properties

The mechanical performance of the BW geopolymers was evaluated through compressive strength (UCS) and flexural strength (FS) measurements according to the EN 196-1 and ASTM C109 standards after 7, 28, or 90 days of aging, depending the experiment. The geopolymers were casted in cubic ($5 \times 5 \times 5$ cm) or prismatic ($4 \times 4 \times 16$ cm) molds (Figure 6). For each synthesis, 3 specimens were prepared and tested.

**Figure 6.** BW geopolymers subjected to flexural and compressive strength measurements.

Young's modulus and Poisson's ratio were also measured following the ASTM C 469 standard. Cylindrical specimens (20×10 cm) were prepared and tested 7 days after their preparation. The measurements were performed by using strain gauges supplied by KYOWA Co., Ltd. (Tokyo, Japan).

2.4. Analytical Characterizations

Brick waste was characterized through X-ray diffraction (XRD) on a Bruker D8 ADVANCE X-ray diffractometer. Diffractograms in a 2θ range $5\text{--}70^\circ$ with 0.1° step size and 1 sec per step were collected and then were evaluated by using Diffrac.Eva v3.1 software.

The microstructure of the BW geopolymers was examined by using a JEOL JSM-5600 scanning electron microscope equipped with an OXFORD LINK ISIS 300 energy-dispersive X-ray spectrometer (EDX). Fragmented pieces of the samples were gold-coated prior to the measurement.

2.5. Durability Tests

Sorptivity tests, following the ASTM C1585 standard, were performed to determine the water absorption of the BW geopolymers through capillary suction. The specimens were dried at 105°C for 24 h prior to testing. Then, the lower side surface of each specimen was covered with a water-impermeable film. The specimens were placed on special supports so that they were submerged to a depth of approximately 5 mm. The water mass absorbed by the specimens was measured after 10, 20, 30, 45, 60, 90, and 120 min from the beginning of the test. The sorptivity test was based on measuring the rate of water absorption attributed to the capillary pores in a geopolymer sample. Sorptivity (S) is defined as:

$$S = \frac{i}{\sqrt{t}}$$

i : total increase in the specimen's mass per adsorption surface area unit (g/mm^2).

t : time (min).

The long-term stability of the BW geopolymers was determined through repeated cycles of wetting/drying (ASTM D 559M) and freezing/thawing (ASTM C 666). In these tests, material losses and shape changes produced during the cycles determined the stability of the examined samples. Prior to testing, the examined specimens were water-saturated and then weighed. A total of 50 cycles were performed for each test. Concerning the wetting/drying tests, each cycle included the immersion of the specimens in water ($25 \pm 2^\circ\text{C}$) for a period of 4 h and then the drying at $80 \pm 5^\circ\text{C}$ for 4 h. In the freezing/thawing tests, each cycle included the freezing of the specimens at $-18 \pm 2^\circ\text{C}$ for a period of 4 h and then immersion in water ($25 \pm 2^\circ\text{C}$) for another 4 h. The mass of the specimens was recorded after a period of 5 to 10 cycles.

The stability of the BW geopolymers was also tested by exposing the BW geopolymer specimens to real weather conditions (Athens, Greece) for a period of 6 months (October 2019 to March 2020). The stability was evaluated in terms of mass and shape changes that were periodically measured.

For each sample, 3 cubic ($5 \times 5 \times 5$ cm) specimens were prepared and tested, while compressive strength measurements were conducted after the end of the tests.

3. Results and Discussion

3.1. Curing Conditions Optimization

The curing of the geopolymer specimens is an essential requirement for the completion of the geopolymerization reactions. Using improper conditions in terms of temperature and time may result in either incomplete geopolymerization or the degradation of the reaction products [45]. In this study, the optimization of curing conditions delivered a robust binding matrix (geopolymer) from BW that was effectively applied for the preparation of lightweight geopolymer composites though the inclusion of lightweight aggregates and short plastic fibers.

Figure 7 presents the effect of curing time (a) and temperature (b) on the mechanical properties of the BW geopolymers. To evaluate the curing time, the prepared specimens were cured at a constant temperature of 80°C , while those prepared for evaluating the

curing temperature were cured at a constant time of 72 h. Finally, the mechanical properties were measured 7 days after the synthesis of the specimens.

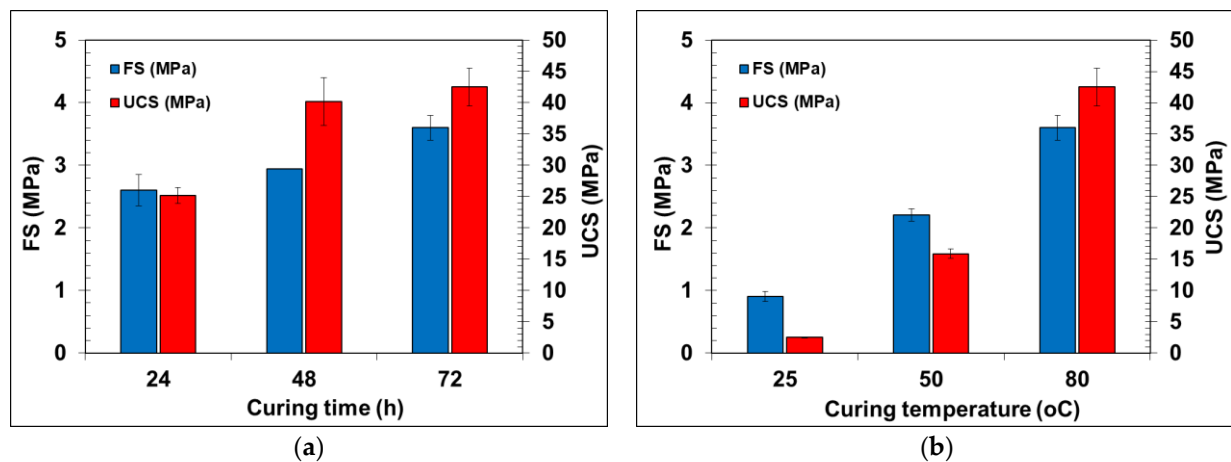


Figure 7. Effect of curing time (a) and temperature (b) on geopolymer's properties.

As was expected, an increase in the time or temperature enhanced the mechanical performance of the BW geopolymers. This is attributed to the ion diffusion phenomena among the solid and liquid phase of the geopolymer slurries [73]. In particular, the increase in curing time prolongs the diffusion duration, while the increase in curing temperature accelerates the diffusion. Both cases are beneficial to the geopolymerization reactions up to a point, with the effect of temperature being more significant.

The results showed that robust BW geopolymers can be prepared after curing at 80 °C for 72 h, having a UCS and FS of 42.5 and 3.6 MPa, respectively. These conditions will be applied for the preparation of lightweight geopolymer composites even though they are relatively more intense than those usually applied for the geopolymerization of other aluminosilicate sources such as fly ash [70] due to the lower geopolymerization potential of BW [50].

Concerning the aging of the specimens (Figure 8), a variation in aging time (7, 28, and 90 days) did not significantly change the values of UCS and FS at any tested curing time (24, 48 and 72 h), revealing that the geopolymerization reactions were almost completed 7 days after the synthesis. Indeed, any variation in the strength value lies within the standard deviation range. From the aforementioned, the mechanical strength tests were performed 7 days after the preparation of the specimens except the durability testing (after 28 d).

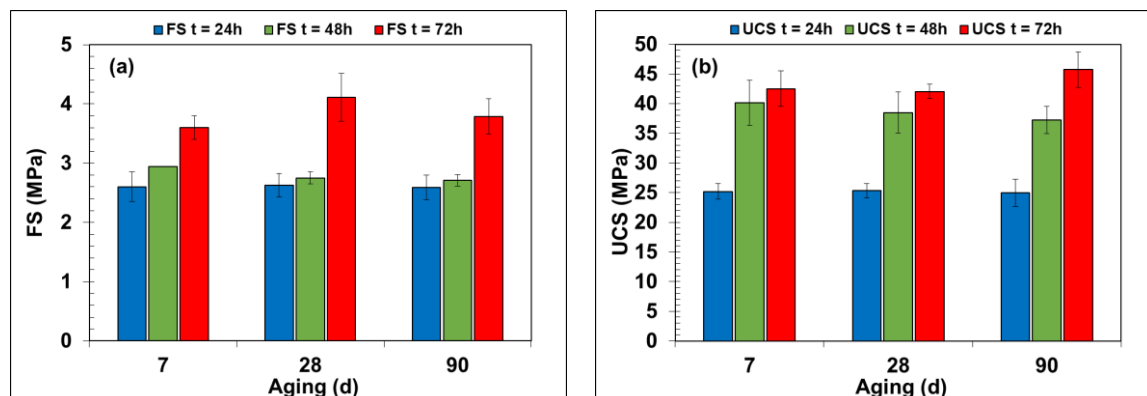


Figure 8. Effect of aging time on geopolymer's (a) FS and (b) UCS.

3.2. Lightweight BW Geopolymers

The impact of EPS introduction on the properties of the BW geopolymers is shown in Figure 9. The use of EPS as lightweight aggregate in the geopolymer synthesis, as expected from its hollow structure, promotes the preparation of low-density building materials [70,74–76]. Indeed, the introduction of 3% wt. EPS into the geopolymer matrix lowered the density from 2.1 to 1.0 g/cm³, corresponding to a 52% decrease. Apart from the density, the EPS introduction affected the mechanical strength of the specimens. More specifically, 3% wt. EPS addition led to a reduction in UCS by 85%. The aforementioned observations are linked with the high-volume fraction capacity of EPS into the geopolymer specimens causing the dilution of the geopolymer matrix and, subsequently, the deterioration of the mechanical properties. The product that contained 3% wt. EPS had a UCS of 6.5 MPa and density of 1.0 g/cm³.

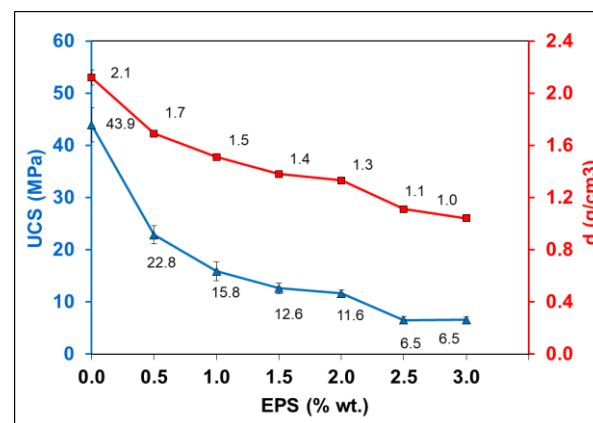


Figure 9. Geopolymers' properties (UCS and density) when incorporating EPS waste.

Correspondingly, Figure 10 depicts the effect of PU introduction on the properties of the BW geopolymers. In contrast to the EPS behavior, the addition of PU to the geopolymer mixes did not result in a significant reduction in the density of the BW geopolymers. Indeed, the addition of 6% wt. PU resulted in just a 25% drop in the density values, while at the same time, the UCS was reduced by 75%. This behavior may be related to the higher compressibility of PU than EPS (Table 2) during the mixing and molding of the geopolymer slurries. Similar results on the property alterations were reported by Kismi et al. [77].

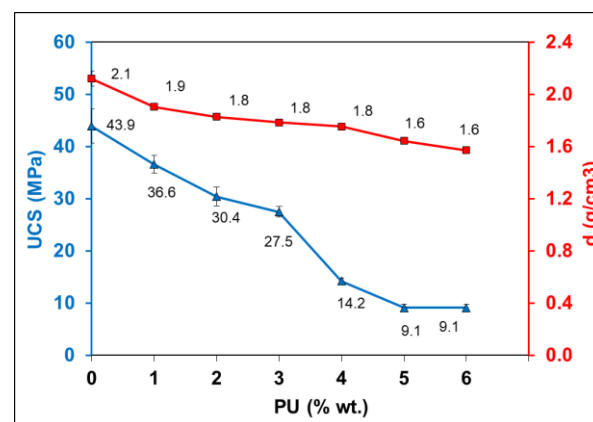


Figure 10. Geopolymers' properties (UCS and density) when incorporating PU waste.

The results demonstrated that EPS waste can be effectively used as artificial lightweight aggregates in the preparation of CDW lightweight geopolymer matrices, fulfilling, at the same time, the minimum mechanical strength requirements. The EPS introduction

(0.5–3.0% wt.) resulted in the development of products with densities ranging from 1.0 to 1.7 g/cm³ and a UCS ranging from 6.5 to 22.8 MPa. The developed products, depending the EPS concentration, can be used as structural ($\leq 0.5\%$ wt. EPS) or nonstructural ($>0.5\%$ wt. EPS) building materials [78].

3.3. Fiber Reinforcement

Both the reference and lightweight BW geopolymers were subjected to fiber reinforcement in order to evaluate the performance of the PE fibers. Concerning lightweight geopolymers, GBW_1.5EPS and GBW_2.0EPS were selected since higher EPS contents led to products with limited mechanical properties (UCS < 10 MPa). Figure 11 presents the effect of PE fiber incorporation on the properties of the GBW_REF (a), GBW_1.5EPS (b), and GBW_2.0EPS (c) composites, respectively.

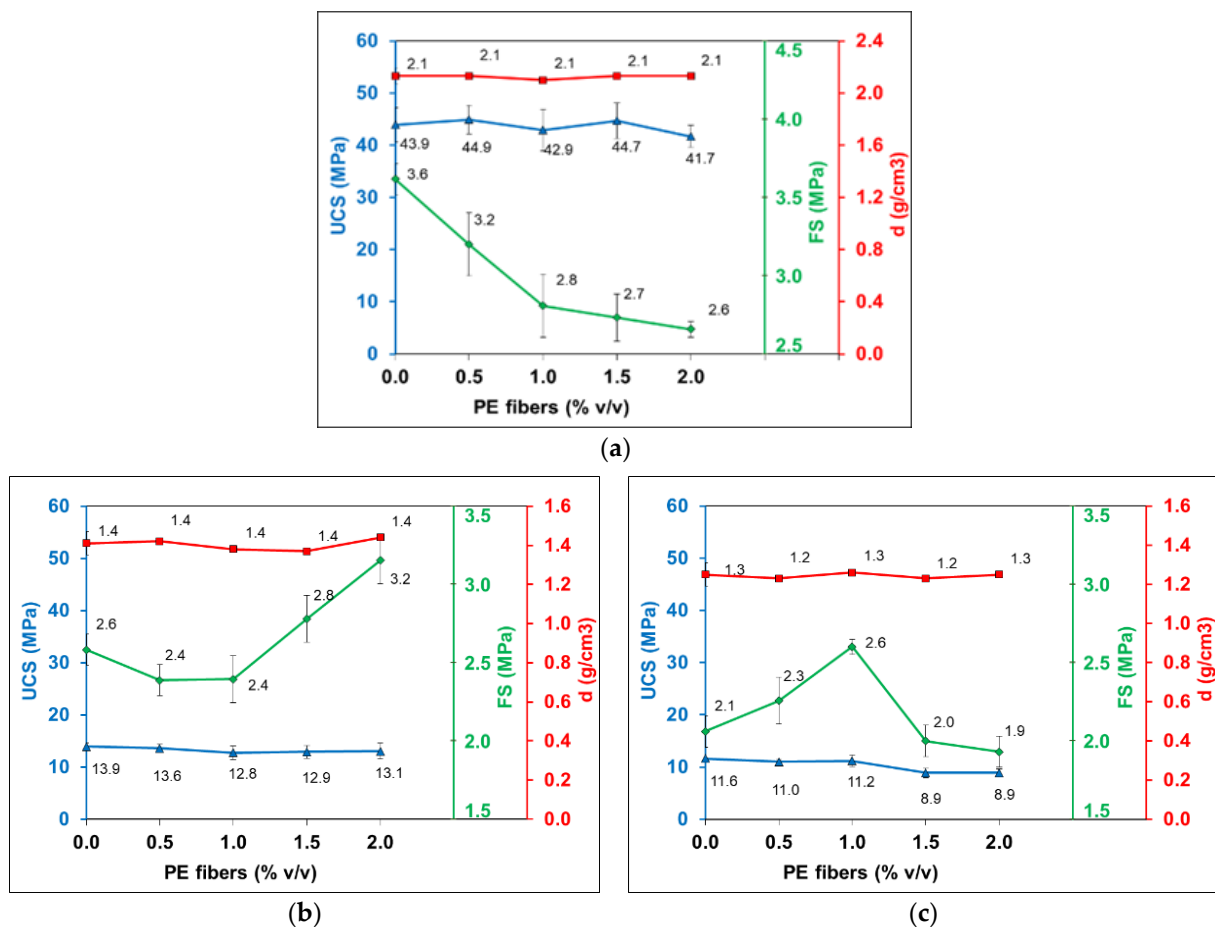


Figure 11. Properties (UCS, FS and density) of fiber-reinforced geopolymers containing: (a) 0% wt. EPS, (b) 1.5% wt. EPS, and (c) 2.0% wt. EPS.

The reinforcement of the reference geopolymer (Figure 11a) showed that PE fibers had a negative impact on the FS of the produced composites. More specifically, the progressive introduction of PE fibers inside the geopolymer matrix led to worse behavior under flexion. As a result, the GBW_REF_2.0PE sample exhibited a 28% lower FS value (2.6 MPa) in relation to its unreinforced counterpart (3.6 MPa). This behavior is in contrast to other authors' observations when using polyethylene fibers [79], where an increase in the fiber content up to 2% led to an improvement in the bending behavior of the geopolymer composites. This might be attributed to the significantly lower resistance under tension of the developed PE fibers from CDWs (Table 2) in relation the commercial PE fibers used in other studies [80]. The UCS and density of the specimens seemed to be nearly constant throughout the PE fiber incorporation range.

The reinforcement of the geopolymer containing 1.5% wt. EPS (Figure 11b) showed an enhancement of the FS values for the PE fibers' content higher than 1% *v/v*. The composite prepared with 2% *v/v* PE fiber content exhibited the highest FS value (3.2 MPa), which was 23% higher than that of the unreinforced geopolymer. These results are in accordance with the related literature, where it was reported that 2% *v/v* PE fiber content is the optimum one for enhancing the flexural performance of the geopolymer composites [79]. Contents higher than 2% *v/v* result in fiber aggregation inside the geopolymer matrix, negatively affecting the mechanical performance of the composites [81]. As in the case of GBW_REF, the UCS and density of the specimens remained almost unaffected during fiber reinforcement.

In the case of the GBW_2.0EPS geopolymer (Figure 11c), a slight increase in FS values was observed up to 1% *v/v* PE fiber content. Beyond this content, a considerable decrease in FS values was recorded, possibly associated with the high dilution of the binding matrix (geopolymer) that cannot homogeneously integrate both EPS aggregates and PE fibers. This was also reflected in the slightly lower values of UCS of the composites containing 2.0% *v/v* PE fibers. From the aforementioned, it is clear that even though PE fibers developed from CDWs have poor behavior under tension in comparison to their commercial counterpart, they can effectively reinforce samples with low and moderate mechanical properties such as lightweight geopolymers. Indeed, the fiber reinforcement of GBW_1.5EPS and GBW_2.0EPS samples led to an enhancement in flexural strength by about 24% when 2.0 and 1.0% *v/v* PE fiber contents were added, respectively.

The sample containing 1.5% wt. EPS and 2.0% *v/v* PE fibers held the best combination of properties for the development of a lightweight composite since it exhibited the highest resistance under bending (FS of 3.2 MPa) combined with a density and UCS of 1.4 g/cm³ and 13.1, respectively. Therefore, this sample was chosen for further investigation in terms of microstructure analysis and durability testing. For comparison reasons, GBW_REF was also tested to reveal any differences in the structure and performance of the samples.

Table 5 presents the modulus of elasticity and Poisson's ratio values of the GBW_REF and GBW_1.5EPS_2.0PE samples. The incorporation of EPS waste and PE fibers in the geopolymeric matrix had an effect on the reduction in the elasticity modulus. Both EPS and PE fibers possess low values of elasticity modulus and the dilution of the geopolymeric matrix with such materials leads to products with improved ductile behavior. The Poisson ratio of the GBW_1.5EPS_2.0PE lies within the typical range (0.1–0.2) of the low-weight cementitious materials [82,83]. Concerning the reference material, a higher value of Poisson's ratio was measured. This fact is in good accordance with the Poisson ratio values of cement pastes and mortars which generally possess values between 0.20 and 0.35 [83].

Table 5. Young's modulus and Poisson's ratio of GBW_REF and GBW_1.5EPS_2.0PE.

Sample	Young Modulus (GPa)	Poisson Ratio
GBW_REF	4.3	0.27
GBW_1.5EPS_2.0PE	1.3	0.11

3.4. Microstructure

The microstructure of the reference geopolymer (a, b) and its fiber-reinforced counterpart (c, d, e, and f) is presented in Figure 12. It is worth mentioning that the examined samples are fragments of specimens subjected to mechanical strength tests. GBW_REF appears to be relatively dense (a, b) with pores with diameters between 5 and 500 μm . GBW_1.5EPS_2.0PE exhibits a slightly higher concentration of pores that affects the final porosity of the geopolymer matrix (c, d). This porosity acts synergistically with the EPS particles in the reduction in samples' density.

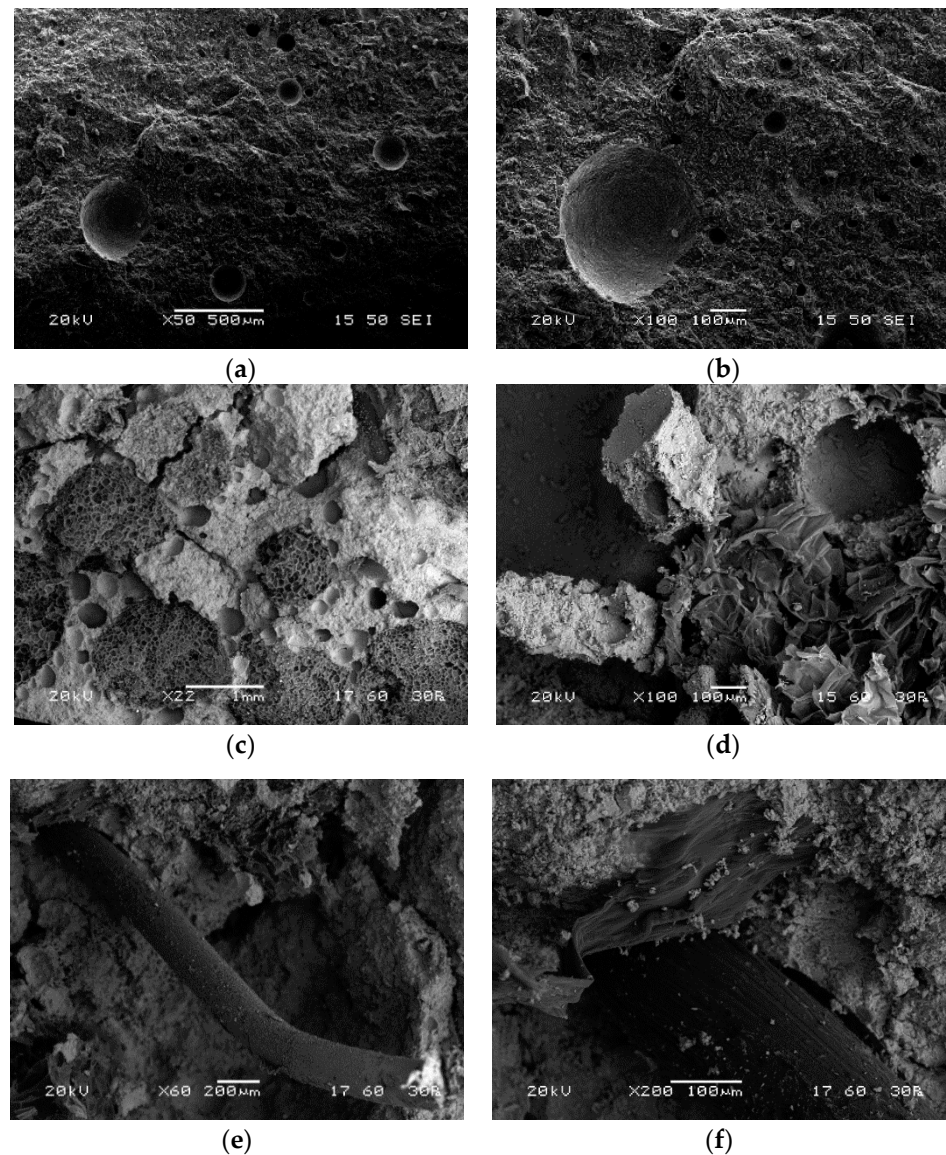


Figure 12. SEM micrographs of GBW_REF (a,b) and GBW_1.5EPS_2.0PE (c–f) samples.

Apart from that, the EPS particles had good cohesion with the geopolymer matrix, while they seemed to remain relatively uncompressed. The fibers' surface and texture as well as the fiber–matrix interface can act as indicators of the bonding quality between the fiber and geopolymer matrix [84]. In this case, the fiber surface (e and f) was smooth since few geopolymer products covered them, clearly showing the debonding between the matrix and PE fibers. Indeed, a factor that negatively affects the matrix–fiber bonding is the hydrophobic nature of PE fibers [80]. Furthermore, the PE fiber–matrix interface (f) also presented a weak bonding that reveals a pull-out failure mechanism. The aforementioned observations explain the moderate effect that the PE fibers had on the improvement in the flexural behavior of the samples. Therefore, modifications can be adopted for PE fiber synthesis to improve the geopolymer matrix–fiber surface bonding. Furthermore, no considerable deformation in the shape and surface of the fibers was observed.

A more sophisticated method of analysis is X-ray-computed tomography, which can accurately define the microstructure of such materials in terms of aggregate and fiber distribution, composite porosity, crack paths, etc. [85,86].

3.5. Durability Performance

3.5.1. Sorptivity Tests

In Figure 13, the water absorption plots of the GBW_REF and GBW_1.5EPS_2.0PE samples are presented. The GBW_1.5EPS_2.0PE specimens exhibited a little higher water absorption rate in relation to the reference sample. This observation agrees well with the findings of microstructure analysis, where the lightweight product exhibited higher porosity than the reference, resulting in higher water absorption via capillary suction. Furthermore, the loose connection between the matrix and PE fibers may also result in increased absorption.

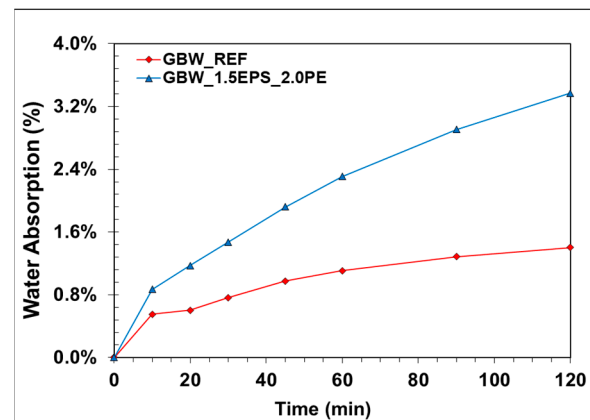


Figure 13. Water absorption (%) vs. time (min) for the reference and reinforced samples.

In order to export the sorptivity values of the samples, diagrams of water absorption per unit area vs. square root of time ($t^{0.5}$) were plotted. The slope of the curves indicates the sorptivity magnitude. Indicative sorptivity curves are shown in Figure 14 for the GBW_REF and GBW_2.0EPS_1.0PE specimens.

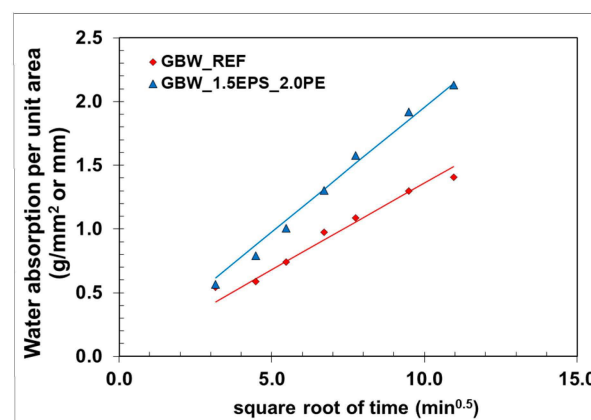


Figure 14. Association between water absorption per unit area and square root of time.

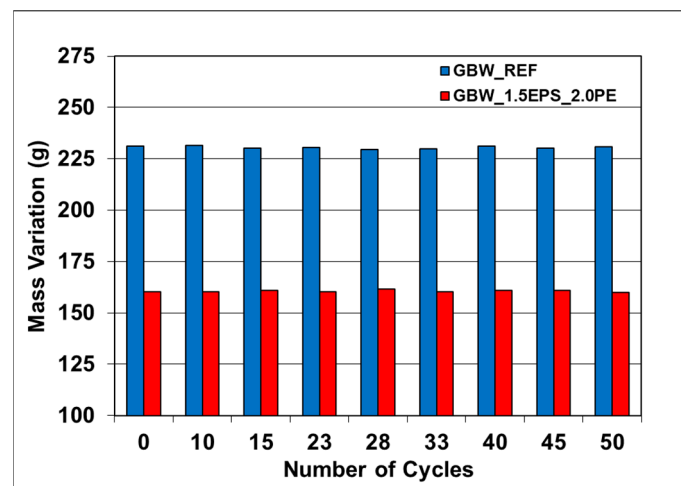
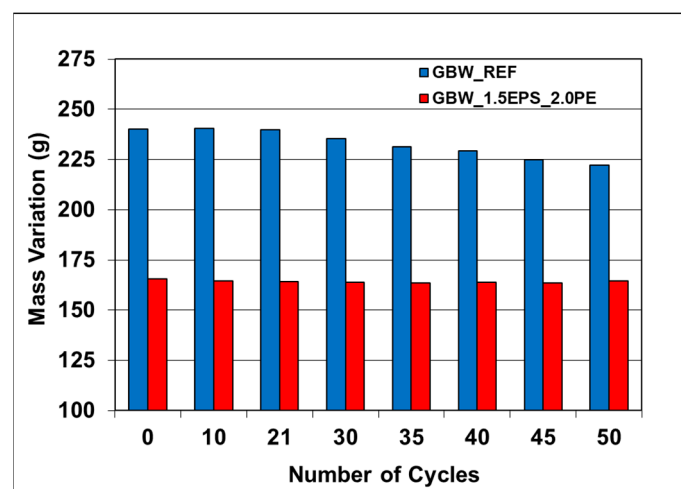
The sorptivity values of the tested samples along with the R^2 factors of linear regression are presented in Table 6. The values' fitting is near excellent for all the tested specimens. The sorptivity of GBW_REF was calculated at $0.131 \text{ mm}/\text{min}^{0.5}$, while that of GBW_1.5EPS_2.0PE was found to be a little higher ($0.179 \text{ mm}/\text{min}^{0.5}$). Similar results were reported in the literature regarding EPS incorporation in geopolymer matrices [70,87]. Furthermore, the sorptivity values of both samples lay within the acceptable range set for the sorptivity of cementitious materials [88].

Table 6. Sorptivity and R^2 values of tested geopolymers.

Samples	Sorptivity (mm/min ^{0.5})	R^2 (%)	Average S (mm/min ^{0.5})
GBW_REF	0.1295	97.9	0.1309
	0.1271	96.8	
	0.1361	98.0	
GBW_1.5EPS_2.0PE	0.1840	98.6	0.1790
	0.1974	98.4	
	0.1583	97.4	

3.5.2. Long-Term Stability Tests

In this section, durability tests concerning repeated wetting and drying, freezing and thawing, and exposure to outdoor conditions (Athens, Greece) are presented. The performance of the samples was evaluated in terms of the mass stability and compressive strength after the end of the durability tests. Figures 15–17 present the mass stability of the GBW_REF and GBW_1.5EPS_2.0PE geopolymers when subjected to wet/dry cycles, freeze/thaw cycles, and exposure to outdoor conditions, respectively. Figure 18 shows the mechanical performance of the specimens before and after the completion of durability testing.

**Figure 15.** Wet/dry cycles.**Figure 16.** Freeze/thaw cycles.

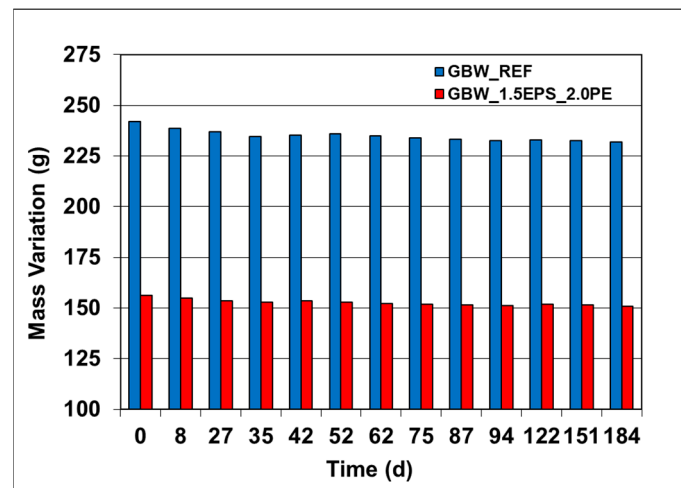


Figure 17. Exposure to weather conditions in Athens, Greece.

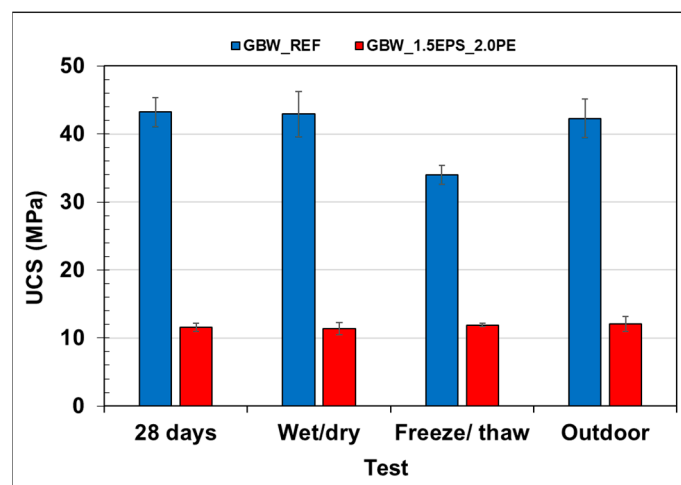


Figure 18. Mechanical performance after the end of durability tests.

Concerning wet/dry cycles (Figure 15), the masses of all the specimens, regardless of their composition, remained practically constant after the end of 50 cycles. Indeed, the mass variation was measured to be as low as 0.3%. This stability indicates a satisfactory durable behavior in both the GBW_REF and GBW_1.5EPS_2.0PE samples. The composite sample had a corresponding behavior when subjected to freeze/thaw tests (Figure 16) since it also presented a nearly zero mass variation (less than 0.4%). However, the reference sample showed a progressive weight reduction (up to 7.5%), a fact that was also confirmed by the visual inspection of the specimens, where extended corrosion was observed (Figure 19). A drop in the weight of the specimens after the conduction of 50 freeze/thaw cycles was also observed for geopolymers prepared with ceramic powder, slag, and fly ash precursors [70,89]. EPS and PE fibers seem to act as stabilizing agents in the geopolymer synthesis, preventing the extended corrosion.

The performance of the geopolymers after their exposure to real weather conditions (Athens, Greece) for a 6-month period is presented in Figure 17. The masses of the geopolymer specimens fluctuated depending on the daily weather conditions; however, their variation was rather small. In particular, the GBW_REF and GBW_1.5EPS_2.0PE specimens exhibited $\pm 1.21\%$ and $\pm 1.02\%$ mass variations after 184 days of testing, respectively.

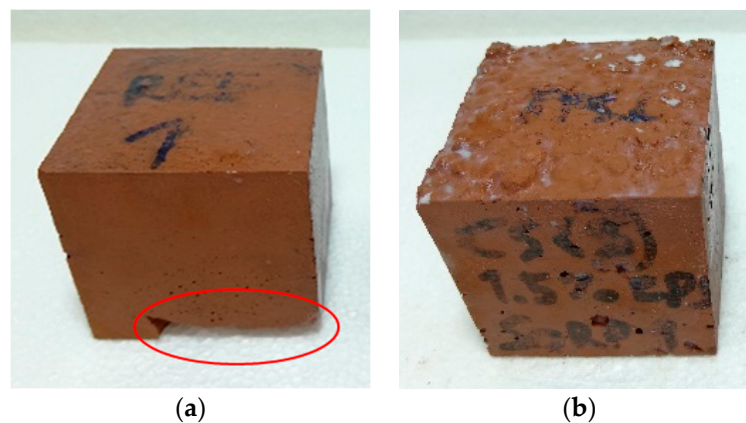


Figure 19. GBW_REF (a) and GBW_1.5EPS_2.0PE (b) specimens after the end of freeze/thaw testing. Corrosion is observed in GBW_REF sample (red cycle).

Regarding the mechanical performance of the samples (Figure 18), GBW_2.0EPS_1.0PE exhibitde very good mechanical stability after the conduction of the durability tests. As was observed, the compressive strength of the geopolymer composites remained practically unaffected (with a deviation lower than 5%). In a similar manner, the GBW_REF specimens exhibited good mechanical stability except after the end of the freeze/thaw tests, where a reduction in the compressive strength by 20% was measured. This fact was expected since the reference geopolymer specimens presented a total mass loss of 7.5%.

4. Conclusions

This study suggests a way to develop durable, lightweight geopolymer composites by valorizing different CDW streams. CDW brick was used as the aluminosilicate precursor, EPS and PU wastes lowered the total weight of geopolymers, and PE fibers from CDWs reinforced the geopolymer matrix. Apart from the physicochemical and mechanical characterization of the products, a comprehensive investigation of the durability performance was performed. The conclusions of this study can be summarized as follows:

- Intense curing conditions ($T = 80\text{ }^{\circ}\text{C}$ and $t = 72\text{ h}$) are required to deliver a robust binding matrix from CDW brick ($\text{UCS} = 42.5\text{ MPa}$ and $\text{FS} = 3.6\text{ MPa}$) that can be used as a basis for the incorporation of low-density aggregates and short plastic fibers. These curing conditions limit the application of the developed lightweight composites to the precast industry.
- EPS and PU wastes can be valorized in the synthesis of CDW-based geopolymers as artificial lightweight aggregates for reducing the total weight of the products. However, EPS achieved a better combination of density and mechanical strength, mainly due to its lower compressibility. Indeed, the maximum EPS content (3% wt.) achieved a density and UCS decrease of 52 and 84%, respectively, while the corresponding PU content (6% wt.) decreased the density and UCS by 25 and 75%, respectively. A control in the EPS content can lead to the development of geopolymeric materials for construction ($\leq 0.5\%$ wt.) or nonconstruction ($> 0.5\%$ wt.) applications.
- The use of short PE fibers developed from CDWs can effectively reinforce lightweight BW geopolymers, enhancing their bending performance by up to 24%. The lightweight product containing 1.5% wt. EPS and 2.0% v/v PE fibers held the best combination of properties: $\text{UCS} = 13.1\text{ MPa}$, $\text{FS} = 3.2\text{ MPa}$, density = 1.4 g/cm^3 , and Young's modulus = 1.3 GPa .
- The durability testing of the CDW-based geopolymers showed that the incorporation of lightweight aggregates and fibers (1.5% wt. EPS and 2.0% wt. PE fibers) into the geopolymer matrix modified their performance. Indeed, the freezing performance of the lightweight geopolymer composite containing 1.5% wt. EPS and 2.0% v/v PE fibers was greatly improved in relation to the reference sample. The sorptivity of the afore-

mentioned sample ($0.179 \text{ mm/min}^{0.5}$) lay within the accepted range of cementitious materials, even though it was slightly higher than that of the reference sample.

- The developed geopolymer composites incorporated a high percent of CDW materials (higher than 70% on a solid precursor basis), revealing the high potential of geopolymer technology in the field of the circular economy.

A further step of this research work will be the modification of geopolymer synthesis through the replacement of the highly corrosive activation solution with solid activators developed from wastes. In this way, the production of CDW lightweight geopolymer composites will be simpler, safer, and more environmentally friendly.

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