

## PHYSICO-CHEMICAL AND MECHANICAL PROPERTIES OF ECO-FRIENDLY ULTRA-LIGHTWEIGHT GEOPOLYMERS CONTAINING SILICA SAND FLOUR AND CALCIUM CARBONATE.

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### ABSTRACT

Low-cost lightweight geopolymer mortars based on ground granulated blast furnace slag, calcium carbonate, and silica sand flour were investigated as lightweight building materials. The effect of two chemical foaming agents such as hydrogen peroxide ( $H_2O_2$ ) and sodium perborate tetrahydrate ( $NaBO_3 \cdot 4H_2O$ ) on bulk density, porosity, and compressive strength was studied. FTIR, XRD, XRF, and SEM were used to investigate the raw materials and selected samples of prepared lightweight geopolymers. The use of grinding calcium carbonate and silica sand flour enhanced the compressive strength, workability, and homogeneity of the geopolymer.

The lightweight geopolymer has given about 3.30 - 17.60 and 1.70 - 6 MPa of compressive strengths at 28 days of curing with bulk densities from 584 to 1340  $kg/m^3$  and 745 to 1770  $kg/m^3$  in the case of  $H_2O_2$  and  $NaBO_3 \cdot 4H_2O$ , respectively. The images of morphology and microstructure results indicate that the porosity and pore size increase with the increase of the foaming agent due to the release of oxygen gas upon its decomposition. According to the results of bulk density MC-H1, MC-H1.25, and MC-B2.5 mixes can be considered ultra-lightweight geopolymers with proper compressive strength. The results show that hydrogen peroxide mixes have better performance in the physico-chemical and mechanical properties than sodium perborate mixes.

**Keywords:** Lightweight geopolymers; hydrogen peroxide; sodium perborate; grinding calcium carbonate; silica sand flour; calcium silicate hydrate.

### 1. INTRODUCTION

The world is now seeing a state of lopsidedness in climatic conditions with record precipitation, destroying surges, and timberland fires because of worldwide warming coming about from greenhouse gas outflows, essentially carbon dioxide. The recent climate conference in Egypt (COP-27, 197 nations, 2022) stressed the completion of the terms of the Paris agreement (COP-27, 2021) by reducing greenhouse gases to reduce the global temperature by 2 degrees Celsius, decrease carbon dioxide emanations by 45 % by 2030 compared to the 2010 level and gradually reducing the use of coal [1].

Many studies have shown that building materials and cement are responsible for 8% of global carbon dioxide emissions [2]. In 2020, the world produced six billion tons of cement. One ton of ordinary Portland cement (OPC) produces a ton of carbon dioxide ( $CO_2$ ) as a by-product of limestone calcination [3]. More considerable efforts have as of

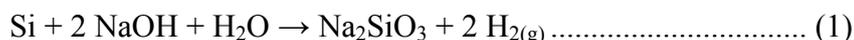
late been attempted to discover an alternative Eco-friendly to ordinary Portland cement [4]. Geopolymers are one of the most suitable alternative Eco-friendly materials. Geopolymeric material can be prepared by condensation reaction between materials that contain silica and alumina in the amorphous phase with highly alkaline activators. Aluminosilicate material may be natural such as metakaolin or a by-product such as a slag and fly ash. Commonly, the alkaline solution could be sodium or potassium hydroxide (NaOH/ KOH) with/without sodium or potassium silicates ( $\text{Na}_2\text{SiO}_3/\text{K}_2\text{SiO}_3$ ) with different silica modulus. The modern form of geopolymeric material used in insulation is lightweight Geopolymers [5]. Lightweight Geopolymers are low-density materials that have good chemical, physical, and thermal properties with low thermal conductivity and fire resistance [6], appropriate compressive strength, low toxicity, low cost of transport, low carbon dioxide ( $\text{CO}_2$ ) emission, low energy consumed in the production process [7,8], lower setting times, rapid hardening, and low shrinkage [9].

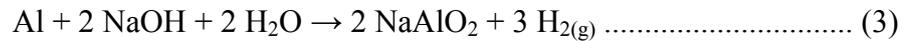
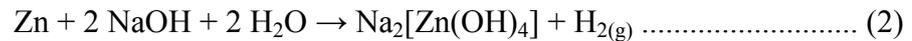
The American Concrete Institute (ACI 318M-11) classifies light geopolymer concrete into three types based on the following properties: compressive strength, thermal, and conductivity bulk density.

Type I is a lightweight structural concrete having a compressive strength of over 17 MPa, a thermal conductivity between 0.4 and 0.7 W/mK, and a density of 1440–1840  $\text{kg/m}^3$ . Whereas Type II is structural concrete and light insulating with a compressive strength of 3.4 to 17 MPa, the thermal conductivity is between 0.22–0.43 W/mK, and a bulk density of 800–1400  $\text{kg/m}^3$ . Type III is considered an insulating lightweight concrete with compressive strength values between 0.7–3.4 MPa, the thermal conductivity of 0.065–0.22 W/mK, and a density of 240–800  $\text{kg/m}^3$  [10, 11].

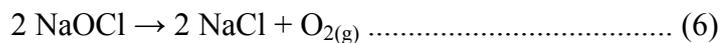
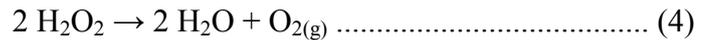
There are two ways to produce lightweight geopolymers using the strategy of lowering density [12].

1. Using a lightweight aggregate to replace a portion of solid raw materials such as (a) Inorganic substances like Vermiculite, Perlite, expanded clay, and glass aggregates [13-17]. (b) Organic compounds like cellulose, polymers made of polystyrene, polyethylene, and polyurethane. According to some experts, these organic compounds (polystyrene and polyurethane) are poisonous and combustible. However, inorganic minerals like perlite and vermiculite are expensive [18, 19].
2. Incorporation of the foaming agent into a paste (in this case geopolymers called foamed or aerated geopolymers) regards the best method for the manufacture of lightweight geopolymers [7, 20-22]. Some techniques, such as mechanical, chemical, thermal, and irradiation approaches, can produce foamed geopolymers. In the chemical method, the foaming agent generates foams or gases (oxygen and hydrogen) into geopolymer paste followed by producing a porous structure with low bulk density according to the following reactions:
  - i- The reaction between the chemical foaming agent with an alkaline activator such as Si, Zn, and Al powders [9, 23-25].





ii- Decomposition of some chemical foaming agents such as hydrogen peroxide [26-28], sodium perborate [19, 29], and sodium hypochlorite. [7, 30]



Since utilizing hydrogen peroxide results in a more equal distribution of pores than Al powder, according to V. Ducman and L. Korat (2016) [31], we shall investigate it in our work. In general, metallic particles created inhomogeneous foams as a result of the gas being produced locally from a constant source until it was exhausted [32]. Using calcium carbonate in geopolymer improves the mechanical properties and the compressive strength [34]. According to A. Antoni et al (2015) [34] when the content of calcium carbonate is not more than (15%) by weight of the geopolymer mixture, the compressive strength, and the workability increase. Grinding calcium carbonate has an essential role in improving these properties [35], so it was used less than 10 microns.

W. Wongkeo (2017) [36] studied the effect of calcium carbonate with aluminum powder acting as a foaming agent. This study concluded that the presence of calcium carbonate by 10% improved the physical properties of the lightweight geopolymer, but the lowest density reached was 825 Kg/m<sup>3</sup>, and the porosity did not exceed 35%, and the images showed that the foam distribution was not ideal.

Therefore, the main objective of this study improves the lightweight geopolymer properties which are made of a mixture of ground granulated blast furnace slag, silica sand flour, and calcium carbonate. Using silica sand flour decreases the bulk densities of the prepared lightweight geopolymers on the other hand calcium carbonate enhances the compressive strength. Replacement of 60% of (GGBFS) with silica sand flour and calcium carbonate decreases the final cost of the prepared geopolymer with locally available raw materials.

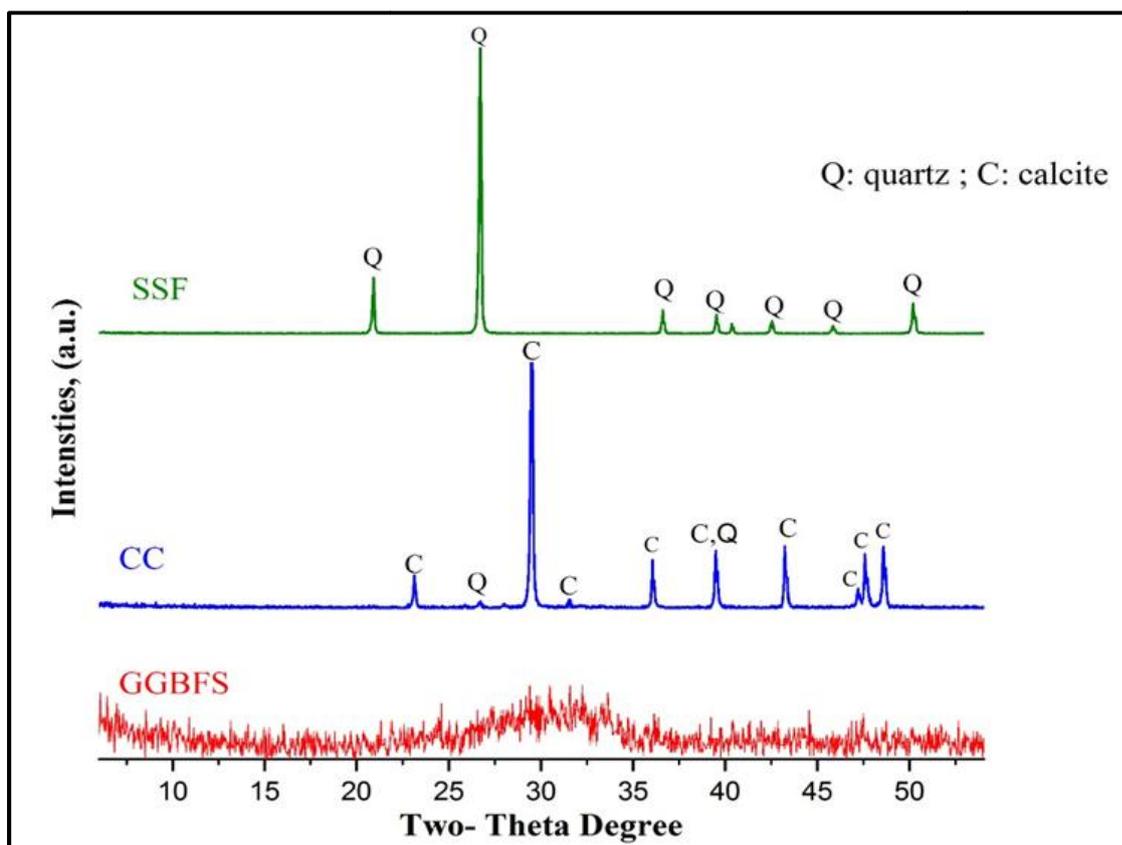
## 2. EXPERIMENTAL WORK

### 2.1. Materials

The ground granulated blast furnace slag (GGBFS) used in this work was supplied by (Iron and Steel Company, Helwan, Giza Governorate, Egypt). The silica sand flour (SSF) and calcium carbonate (CC) were provided by an Engineering Company for Mining (KNOUZ), El-Sadat City, Egypt. The chemical composition by XRF and XRD patterns of the granulated blast-furnace slag, silica sand flour, and calcium carbonate are shown in table 1 and figure 1, respectively.

**Table (1): Chemical compositions of the ground granulated blast furnace slag (GGBFS), silica sand flour (SSF), and calcium carbonate (CC).**

Oxides wt. %	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	MnO	SO <sub>3</sub>	L.O.I
<b>GGBFS</b>	35.3	9.02	0.35	36.4	8.27	0.84	0.2	0.5	3.20	3.14	0.01
<b>SSF</b>	99.16	0.69	0.013	0.11	---	0.01	0.003	---	---	---	---
<b>CC</b>	2.1	0,09	----	54.01	0.18	0.01	0.04	---	---	0.04	43.50



**Fig. 1: XRD patterns of the raw materials: ground granulated blast furnace slag (GGBFS), calcium carbonate (CC), and silica sand flour (SSF).**

The used alkaline activator prepared a mixture between sodium hydroxide and liquid sodium silicate. Sodium hydroxide in the form of flakes obtained from Alkout Industrial Projects Company, Kuwait, 97.5% purity. Liquid sodium silicate (Na<sub>2</sub>O = 13.9 %, SiO<sub>2</sub> = 32.5 %, solid content 46.4%, density = 1.55 g/mL at 20°C) provided by Targochem for Chemical Industries, Borj Al-Arab City, Egypt.

Two different types of chemical foaming agents, hydrogen peroxide and sodium perborate were used. Hydrogen peroxide from Evonik Company, Germany with the following properties: purity of 50% by weight, a density of 1.196 g/mL at 20°C, and pH of 1.50. Sodium perborate tetrahydrate was obtained from Belinka Company, Slovenia with the following properties: white crystals, odorless and water-soluble with a molar mass of 153.9 g/mol.

## 2.2. Procedure for Preparing Lightweight Geopolymers Mortars

### 2.2.1. Preparing of alkaline solution

Firstly, prepare sodium hydroxide solution with a concentration equal to 4 mol/L by dissolving 160 grams of flakes of sodium hydroxide in water and completing the solution to one liter. The prepared sodium hydroxide and liquid sodium silicate (LSS) were mixed with a 1:1 weight ratio, then kept for 24h at room temperature before use to allow for equilibration [37].

### 2.2.2. Preparing lightweight geopolymers

#### 2.2.2.a. In the case of hydrogen peroxide ( $H_2O_2$ ) mixes

The ground granulated blast furnace slag, silica sand flour, and calcium carbonate were mixed in a porcelain ball mill for 15 minutes with a (4:5:1) weight ratio as a solid part of the mixture. The alkaline solution was separately mixed with different concentrations of hydrogen peroxide (0.5, 0.75, 1, 1.25 wt. %). The solid part was gradually added to the liquid and then mixed for 5 minutes to complete the homogeneity of the mortar.

#### 2.2.2.b. In the case of sodium perborate tetrahydrate ( $NaBO_3 \cdot 4H_2O$ ) mixes

The ground granulated blast furnace slag, silica sand flour, and calcium carbonate with (4:5:1) weight ratios were mixed with different percentages of sodium perborate tetrahydrate (1, 1.5, 2, 2.5 wt. %) by porcelain ball mill for 15 minutes. The alkaline activator was placed in the mixer then the solid part was gradually added then mixed for 5 minutes.

## 2.3. Mix Design

The mixed design is illustrated in table 2. The fresh mortars were cast in polystyrene mold with diameters 150\*150\*100 mm and then kept to set at room temperature for 48 hrs. before being removed from the molds then the samples were cut into a parallel piped with 30\*30\*30 mm dimension and kept in the air at room temperature (20-25°C) for 7 and 28 days.

**Table (2): Mix design of lightweight geopolymers mortars samples.**

Mix Code	GGBFS, g	SSF, g	CC, g	AA, g	$NaBO_3$ , g	$H_2O_2$ , g
MC-00	40	50	10	32.5	0	0
MC-H0.5	40	50	10	32.5	0	0.5
MC-H0.75	40	50	10	32.5	0	0.75
MC-H1	40	50	10	32.5	0	1
MC-H1.25	40	50	10	32.5	0	1.25
MC-B1	40	50	10	32.5	1	0
MC-B1.5	40	50	10	32.5	1.5	0
MC-B2	40	50	10	32.5	2	0
MC-B2.5	40	50	10	32.5	2.5	0

## 2.4. Methods

The chemical composition of the ground granulated blast furnace slag (GGBFS), silica sand flour (SSF), and calcium carbonate (CC) can be determined using X-ray

fluorescence (XRF), model Philips PW/1404. The test was run using Rh- $\alpha$  (rubidium) radiation tube at 50 Kv and 50 mA. The investigated samples were prepared as pellets using the manual pressing machine of 20 tonnes load. The traditional method according to ASTM (D7348-2008) for determination of the loss on ignition was used.

Phase identification of raw materials and prepared lightweight geopolymers mortars were investigated by the XRD technique. PAN analytical X-Ray diffraction equipment model X'pert PRO with a secondary monochromator, Cu-radiation ( $\lambda = 1.542\text{\AA}$ ) at 45k.v., 35M.A. A continuous mode was used for collecting data in the  $2\theta$  range from 5 – 50° and a scanning speed of 0.04° /sec before testing, samples were prepared by fine grinding to 25  $\mu\text{m}$  using a HERZOG grinder (Herzog Co., Germany). The functional groups and chemical bonds were identified using Fourier-transform infrared spectroscopy (FTIR) spectrometer type Nicolet is 10, Thermo Fisher, USA FTIR. IR spectra were recorded in the range 400-4000  $\text{cm}^{-1}$  using a KBr binder with a resolution of 4  $\text{cm}^{-1}$  at room temp. The bulk density of lightweight geopolymers was measured by the geometric method at 28 days of hydration by drying the sample for 48 hrs. at 105°C in an electric oven according to the following equation (7):

$$d = m/V \dots\dots\dots (7)$$

Where d; is bulk density, m; is the mass of a parallelepiped-shaped sample cut from a larger foam, and V; is the volume of the sample.

The porosity was carried out using an Archimedes method according to ASTM C642-13.

The porosity of lightweight geopolymers was obtained by equation (8) [10, 38].

$$\text{Porosity (\%)} = \left(1 - \frac{\text{Bulk density}}{\text{apparent density}}\right) \times 100\% \dots\dots\dots (8)$$

The compressive strength test of lightweight geopolymers was achieved by the CONTROLS machine according to ASTM C109 standards. The compressive strength test was performed after 7 and 28 days of curing the sample at room temperature. Before the test, the cubic specimens (3  $\text{cm}^3$ ) were dried for 48 hrs. at 105°C in an electric oven, three samples were tested to obtain an average value of the compressive strength. The morphology and microstructure of the cut surface of lightweight geopolymers were investigated by scanning electron microscope (SEM) model Quanta 250 FEG (Field Emission Gun) with an accelerating voltage 30 kV and a high-resolution camera, Canon model EOS 250D, U.S.A.

### 3. RESULTS AND DISCUSSION

#### 3.1. Starting Material Characteristics

The chemical composition of the ground granulated blast-furnace slag (GGBFS), silica sand flour (SSF), and calcium carbonate (CC) measured by x-ray fluorescence (XRF) is illustrated in table 1. The major oxides of Granulated blast-furnace slag are silica ( $\text{SiO}_2$ ), alumina ( $\text{Al}_2\text{O}_3$ ), calcium oxide (CaO), and magnesium oxide (MgO). Silica

sand flour (SSF) consists of over 99 wt.% SiO<sub>2</sub>. Calcium carbonate (CC) consists of over 54 wt. % CaO that indicate is pure calcium carbonate. The XRD patterns of Granulated blast-furnace slag, silica sand flour, and calcium carbonate are shown in figure 1. XRD indicated that silica sand flour is mainly composed of crystalline quartz, while XRD of Granulated blast-furnace slag shows a halo hump between 2θ= 20-40, indicating the amorphous nature of the material [39]. The XRD patterns of calcium carbonate indicated that calcium carbonate is mainly composed of crystalline calcite.

### 3.2. Bulk Density and Porosity

The bulk density and porosity are shown in figure 2, for lightweight geopolymers prepared with various weight ratios of hydrogen peroxide and sodium perborate tetrahydrate which were aged for 28 days at room temperature. In general terms, the increase of foaming agents leads to a decrease in bulk density and an increase in total porosity due to the presence of voids generated by oxygen gas produced from the decomposition of hydrogen peroxide and sodium perborate tetrahydrate as shown in equations (4 and 7). The bulk density of control sample MC-00 is 1930 kg/m<sup>3</sup>, while the bulk density values of lightweight geopolymers with different values of H<sub>2</sub>O<sub>2</sub> are 1340, 815, 622, and 584 kg/m<sup>3</sup> for MC-H0.5, MC-H0.75, MC-H1, and MC-H1.25, respectively as shown in figure (2a).

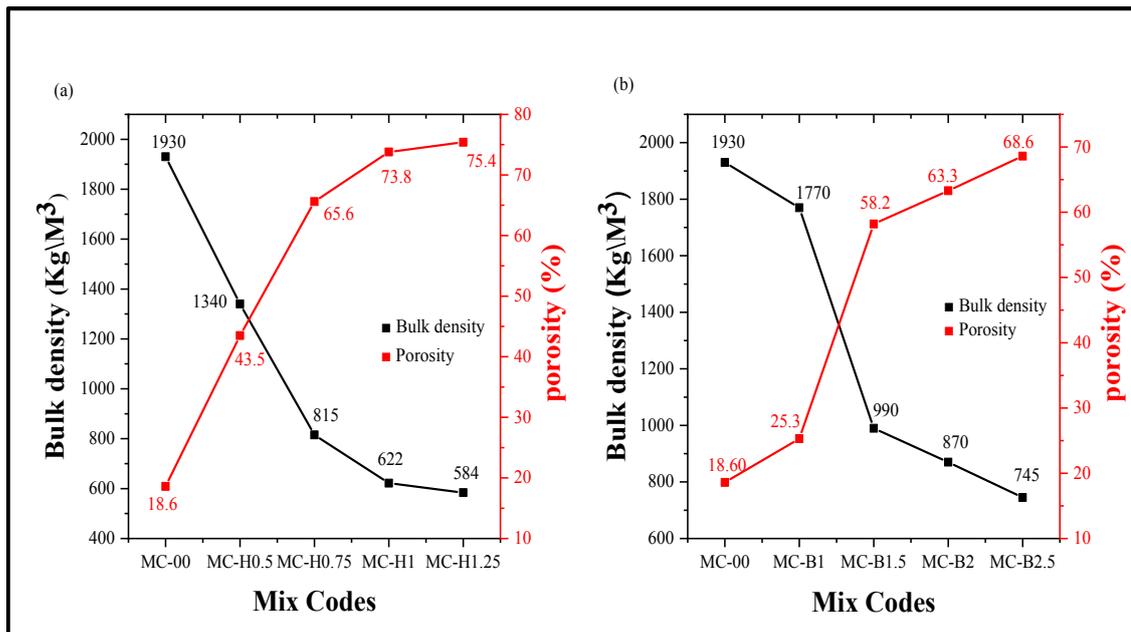


Fig. 2: (a) Bulk density and porosity of hydrogen peroxide mixes and control sample. (b) Bulk density and porosity of sodium perborate mixes and control sample.

MC-H1.25 mix exhibited the lowest bulk density and the highest total porosity (75.40%) due to the increase in the hydrogen peroxide content in the geopolymeric matrix when compared with other mixes (MC-H0.5, MC-H0.75, and MC-H1). While the bulk density values of lightweight geopolymers with different values of NaBO<sub>3</sub>.4H<sub>2</sub>O were 1770, 990, 870, and 745 kg/m<sup>3</sup> for MC-B1, MC-B1.5, MC-B2, and

MC-B2.5, respectively as shown at figure 2b. MC-B2.5 mix gave the lowest bulk density and the highest total porosity (68.60%) while the mixture of (MC-B1) showed a higher bulk density with porosity up to (25.30%) compared with other sodium perborate mixes. According to the results of bulk density MC-H1, MC-H1.25, and MC-B2.5 mixes can be considered ultra-lightweight geopolymers with acceptable compressive strength because of the bulk density of less than  $800 \text{ kg/m}^3$  [40, 41] and regarded as insulating material according to the American Concrete Institute (ACI 318M-11) classification for lightweight geopolymers [11, 22].

### **3.3. Compressive Strength**

The compressive strength results of lightweight geopolymers prepared with various ratios of hydrogen peroxide and sodium perborate tetrahydrate aged for 7 and 28 days at room temperature were plotted in figure 3. The compressive strengths of the control sample MC-00 are 26.14 and 27.1 MPa at 7 and 28 days, respectively. In general, an increase in the foaming agent ratio leads to a decrease in the strength of samples so the strength is directly affected by the foaming agent content. The results indicate that all samples have given reasonable compressive strength values according to the American Concrete Institute (ACI 318M-11) classification for lightweight concrete based on compressive strength and density measurement.

At higher foaming agent concentrations, the formed bubbles fused, resulting in large voids and a decrease in strength. In the case of hydrogen peroxide mixes, figure 3a, the compressive strength values were 5.80, 2.40, 1.60, and 1.04 MPa at 7 days of curing and 6, 3.60, 2.40, and 1.70 MPa at 28 days for MC-H0.5, MC-H0.75, MC-H1, and MC-H1.25, respectively. On the other hand, MC-H0.5 and MC-H0.75 mixes can be classified (Class-II) because the compressive strength values lay between 17 to 3.40 MPa and the bulk density range between  $800\text{-}1400 \text{ kg/m}^3$  [11]. At the same time, MC-H1 and MC-H1.25 mixes can be classified as insulating lightweight concrete (Class- III) because the compressive strength values are higher than 0.7 MPa and the bulk density 622, and  $584 \text{ kg/m}^3$ , respectively.

In the case of sodium perborate mixes, the compressive strength values were 13.90, 3.90, 2.60, and 2.40 MPa at 7 days of curing and 17.60, 5.30, 3.60 and 3.30 MPa at 28 days for MC-B1, MC-B1.5, MC-B2, and MC-B2.5, respectively, figure 3b.

The MC-B1 mix is given the highest compressive strength value because of a lower dose of sodium perborate and it can be classified as structural lightweight concrete (Class-I).where the MC-B1.5, and MC-B2 mixes can be classified as structural and insulating lightweight concrete (Class-II). Finally, the MC-B2.5 mix can be considered insulating lightweight concrete because the compressive strength values lied between 3.4 to 0.7 MPa and the bulk density is less than  $800 \text{ kg/m}^3$ . The relation between the compressive strength, bulk density, and porosity for hydrogen peroxide and sodium perborate mixes is shown in figure 4 (a, b). By comparison, it can be concluded that the sodium perborate mixes had given higher compressive strength and bulk density values with lower porosity values compared with hydrogen peroxide mixes at the same foaming agent content.

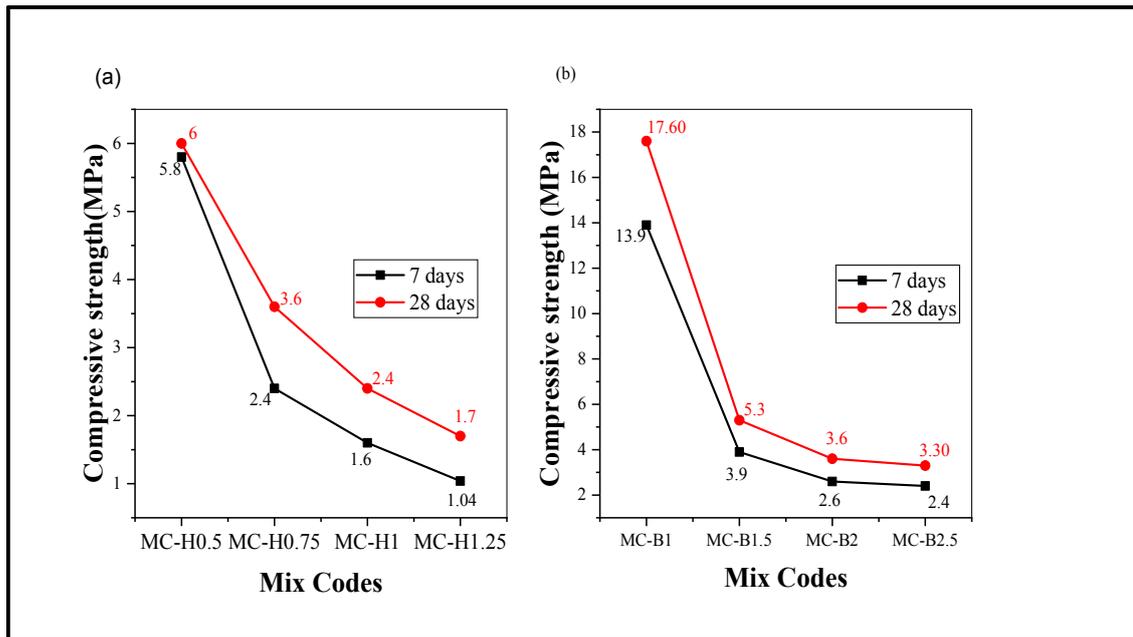


Fig. 3: (a) Compressive strength values of hydrogen peroxide light geopolymer mixes at 7 and 28 days. (b) Compressive strength values of sodium perborate lightweight geopolymer mix at 7 and 28 days.

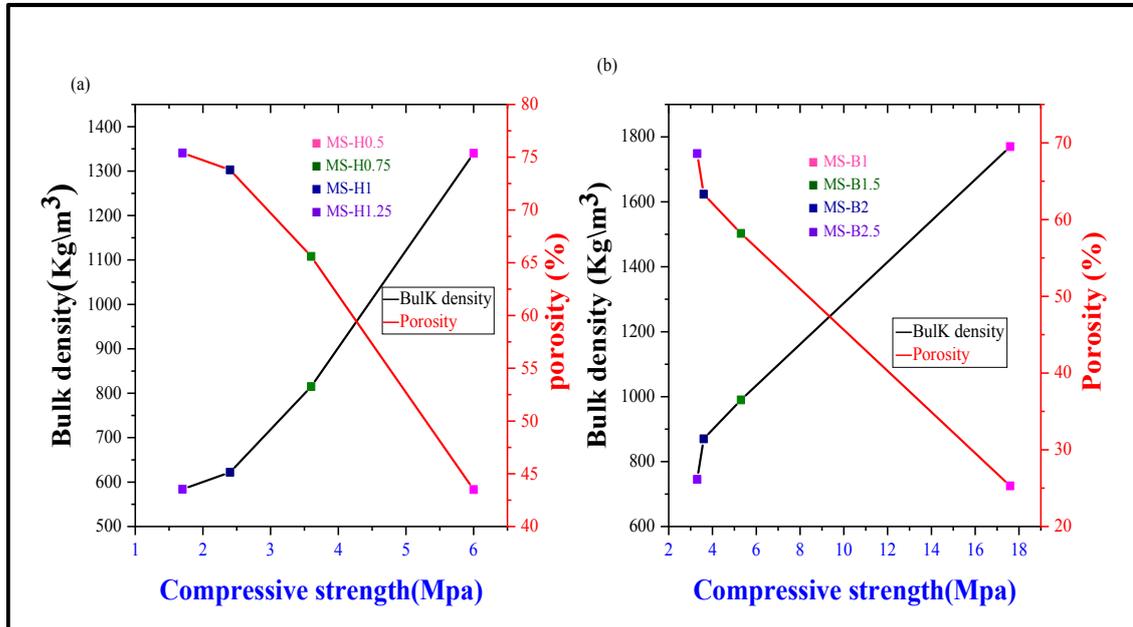


Fig. 4: (a) Relation between the compressive strength, bulk density, and porosity for hydrogen peroxide mixes. (b) Sodium perborate mixes.

### 3.4. FTIR Analysis

The FTIR spectra of three selected samples: MC-00, MC-H1.25, and MC-B2.5 are presented in figure 5 to study the effect of hydrogen peroxide and sodium perborate on covalent bonds in geopolymers. Typically, the vibrational bands of aluminosilicate

geopolymers appeared in all the samples. The broad bands are located less than 500  $\text{cm}^{-1}$  due to the strong vibration bending of (Si-O-Si and O-Si-O) bonds. Symmetric stretching vibration bands located at 693, 778, and 1093 for (Si-O-Si and Al-O-Si) bonds appeared at the same wave number and intensity [42,43]. The bands typical of carbonate phases are seen: at 1442, 1425  $\text{cm}^{-1}$ . These stretching vibration bands of C-O bond bands are connected with the presence of  $\text{CaCO}_3$  in the geopolymers matrix due to the low dissolution of calcite in an alkaline solution [19, 37].

The broad band at 3442  $\text{cm}^{-1}$  is referred to as stretching vibration O-H bonds in the geopolymer matrix's hydrated water. On the other hand, the weak characteristic band at 1648  $\text{cm}^{-1}$  can be attributed to the bending vibration of O-H from water molecules and hydroxyl groups of sodium hydroxide and sodium silicate [45]. From FTIR spectra, the absence of new vibration bands with foaming agent samples (MC-H1.25, MC-B2.5) which refer to all by-products from the decomposition of sodium perborate to sodium metaborate was encapsulated in the geopolymerization reaction in addition to the lower concentration of foaming agent compared with the raw materials of geopolymers [46].

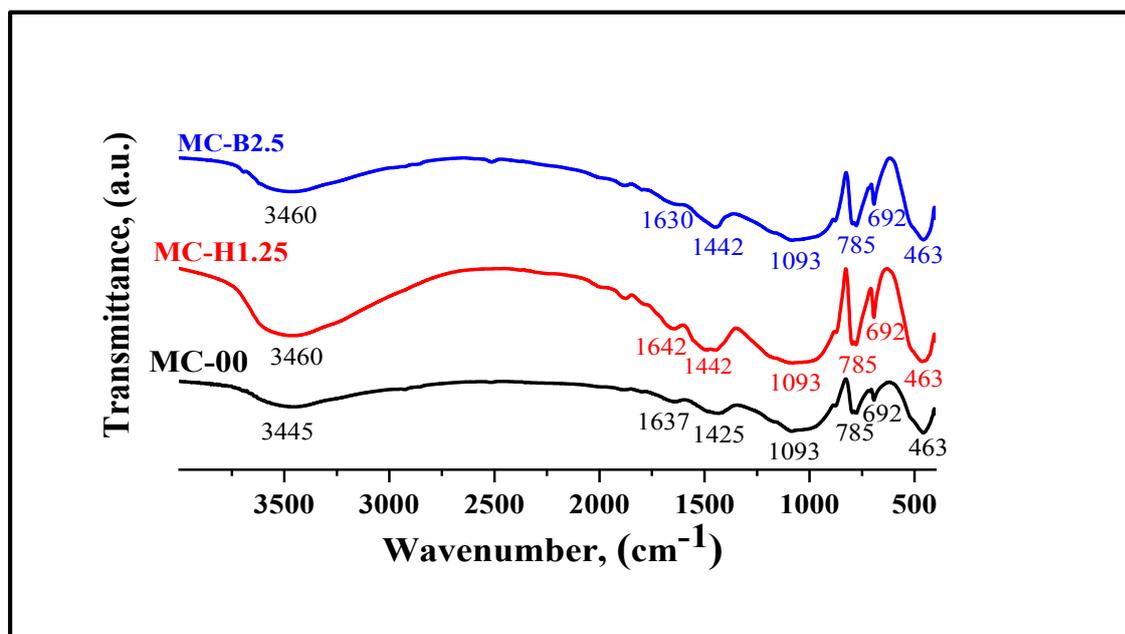
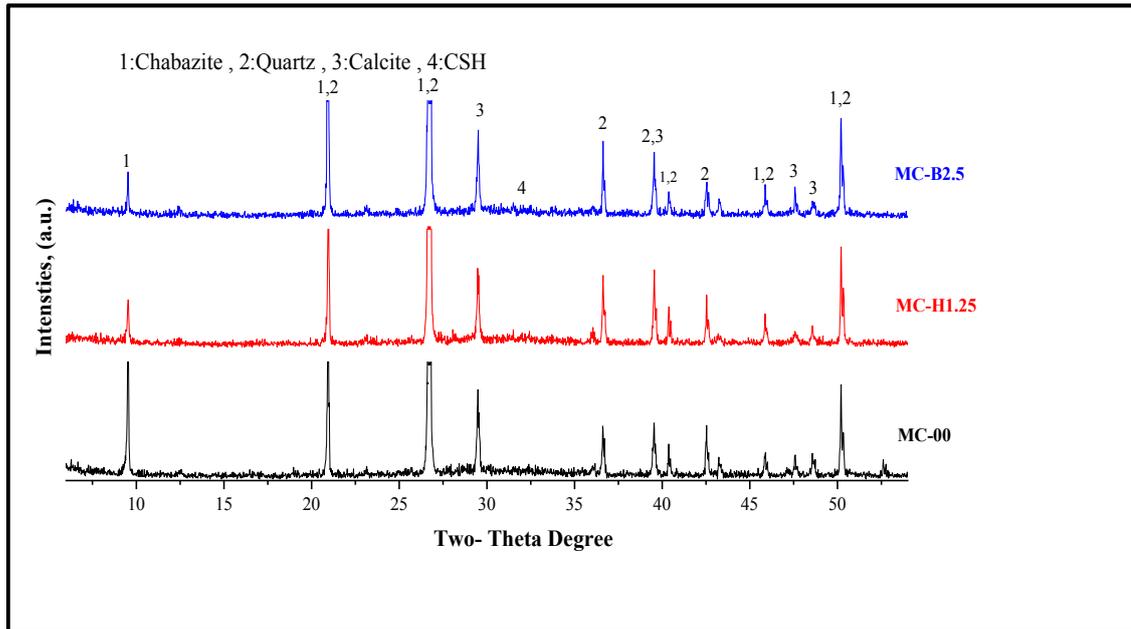


Fig. 5: FTIR of lightweight geopolymer samples prepared with/without foaming.

### 3.5. X-ray Diffraction (XRD)

Figure 6 shows the XRD of three selected samples: MC-00 (without a foaming agent), MC-H1.25 (mixed with  $\text{H}_2\text{O}_2$ ), and MC-B2.5 (mixed with  $\text{NaBO}_3 \cdot 4\text{H}_2\text{O}$ ) cured for 28 days at room temperature. All XRD patterns demonstrate the presence of the crystalline phase of quartz, and calcite in geopolymer and lightweight geopolymers. The crystalline quartz appeared due to the low dissolution of the crystalline quartz phase as the main component of silica sand flour in an alkaline solution [47, 48]. Calcite appeared in all XRD patterns as a result of calcium carbonate not fully reacting in the basic medium but remaining as filler material that increases the compressive strength of geopolymer [33, 49]. On the other hand, the calcium silicate hydrate ( $\text{CaO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ ) appeared due to the high percentage of calcium in ground granulated blast furnace slag (GGBFS) as

shown in XRF in table 1 (36.40%, CaO). The new crystalline chabazite phase ( $\text{CaAl}_2\text{Si}_4\text{O}_{12}\cdot 6\text{H}_2\text{O}$ ) appeared at ( $2\theta = 9$ ) due to slag reactions with an alkaline solution [39]. The intensity of the chabazite phase is different in the three geopolymer samples due to quick hardening and high rate of reaction of geopolymer in the control sample (MC-00) but the setting time is slow in case of adding foaming agent in (MC-H1.25, MC-B2.5) lightweight geopolymers sample. Except for this only difference of crystalline chabazite phase, there is a great deal of similarity in the phases of geopolymer (without a foaming agent) and lightweight geopolymers.



**Fig. 6: XRD patterns of lightweight geopolymer samples prepared with/without a foaming agent aged 28 days.**

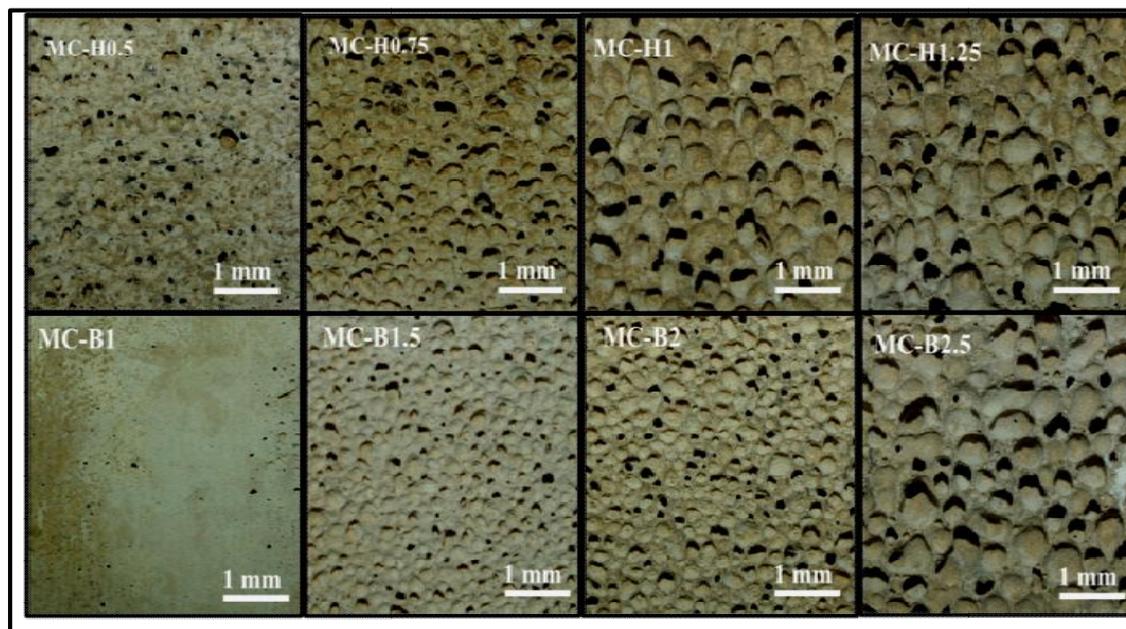
### 3.6. Morphology and Microstructure

The morphology of all lightweight geopolymer mixes can be investigated by taking photographic images at the scale of 1 millimeter as shown in figure 7. The images showed that the pore size distribution of samples (MC-H0.75, MC-H1, MC-B1.5, and MC-B2) exhibited uniform and homogeneous shapes compared with other mixes. From the photographic images, it can be seen that these pores formed by the foaming agents are mainly spherical closed pores. The increase of the foaming agent percentage in the geopolymer mixes is directly proportional to the pore sizes. At the same amounts of the foaming agent; hydrogen peroxide mixes showed a larger pore size compared with the corresponding sodium perborate mixes because of the high oxygen content in hydrogen peroxide decomposition.

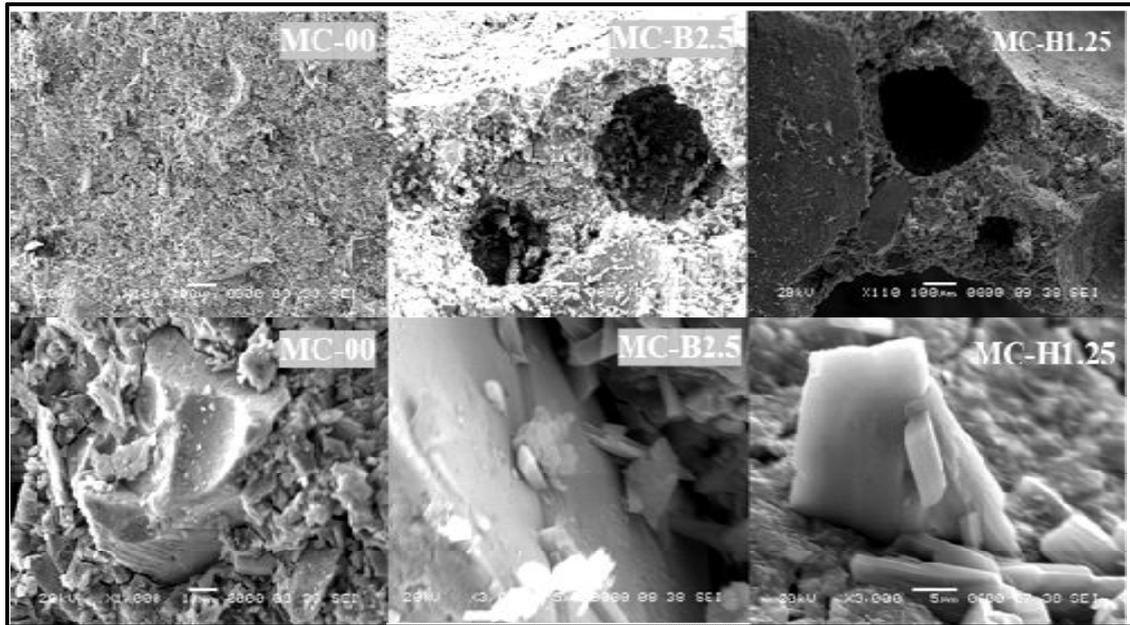
MC-B1 mix showed a non-porous shape with a high bulk density ( $1770 \text{ Kg/m}^3$ ) due to the low concentration of sodium perborate. With an increase in the sodium perborate content in other mixes (MC-B1.5 and MC-B2), the foams are formed in a uniform shape. MC-H1.25 and MC-B2.5 exhibited largely destroyed shapes.

Compared to the images taken in previous studies [29, 36], the foam distribution is excellent, perhaps because silica sand flour helps to homogeneity in the geopolymer mixture.

The microstructure of some selected samples MC-00, MC-B2.5, and MC-H1.25 is examined by SEM at two magnifications (X-100 and X-3000) with scales of 5, and 10  $\mu\text{m}$ , respectively in figure 8. The results show that the presence of unreacted slag, calcium carbonate, and silica sand flour in addition to appearing of the hydration product on the surface of slag and fly ash in the form of calcium silicate hydrate (C-S-H) and sodium aluminum silicate hydrate (N-A-S-H) [39]. SEM images exhibited high percentages of unreacted silica sand flour due to the high crystallinity of silica in the form of quartz phase and the high-substitution volume of GGBFS. These results are confirmed by XRD patterns for the same mixes [6]. SEM images at different magnification values revealed high compaction and uniform phases between geopolymer products and silica sand flour due to the high fineness of silica sand flour due to the absence of cracks at the interfacial transition zone [50].



**Fig. 7: Photographic images of lightweight geopolymer images for the different mixes taken at a 1-millimeter scale.**



**Fig. 8: SEM images for MC-00, MC-B2.5, and MC-H1.25 for three magnification values, X-100, and X-3000 at 28 days of hydration.**

#### 4. CONCLUSIONS

This paper studied mainly the preparation of low-cost lightweight geopolymers from The ground granulated blast furnace slag (GGBFS), silica sand flour, and calcium carbonate by chemical foaming methods without using surfactants or foam stabilizers and concluded some important points as follows:

1. Using insulation materials inside buildings is an important way to conserve energy and lightweight geopolymer materials, especially those based on foam, have an effective role in this.
2. Lightweight geopolymers mortars were successfully produced at room temperature from the mixture containing silica sand flour, and calcium carbonate as a filler (60%) in presence of hydrogen peroxide and sodium perborate tetrahydrate as foaming agents without using surfactants or foam stabilizers.
3. As expected, the addition of hydrogen peroxide or sodium perborate tetrahydrate increases the porosity and reduces compressive strength, and bulk density, as a result of its decomposition and release of oxygen bubbles that cause forming of voids.
4. The measured compressive strength of lightweight geopolymer samples aged 28 days at room temperature, ranged from 1.70 to 6 MPa and 3.30 to 17.60 MPa in the case of hydrogen peroxide and sodium perborate mixes, respectively.
5. According to ACI classification, the lightweight geopolymers produced in this work are used as structural lightweight concrete (Class I), structural and insulating lightweight concrete (Class II), and insulating lightweight concrete (Class III) according.

6. When compared to the prepared sample without foaming agents, all of the FTIR spectra of the prepared lightweight geopolymer samples have the same vibrational bands.
7. There is a great similarity in the geometrical structures for XRD phases of lightweight geopolymers with/without foaming agents, except for the difference in intensity of crystalline chabazite phases in selected mixes.
8. When a higher concentration of foaming agents is added, pores congregate, reducing their number while increasing their size.
9. The use of calcium carbonate, and silica sand flour in lightweight geopolymer, In addition to reducing the cost, improve the physical properties and increases the homogeneity of the geopolymer mixture.

## **5. REFERENCES**

- [1] UNFCCC, “CMA . 3 Glasgow Climate Pact II . Adaptation Advance unedited version Decision”, A report published by UN, vol. 2, pp. 1–11, 2021.
- [2] W. Shen, L. Cao, Q. Li, W. Zhang, G. Wang, and C. Li, “Quantifying CO<sub>2</sub> emissions from China’s cement industry”, *Renew. Sustain. Energy Rev.*, vol. 50, pp. 1004–1012, 2015, doi: 10.1016/j.rser.2015.05.031.
- [3] B. Singh, G. Ishwarya, M. Gupta, and S. K. Bhattacharyya, “Geopolymer concrete: A review of some recent developments”, *Constr. Build. Mater.*, vol. 85, pp. 78–90, 2015, doi: 10.1016/j.conbuildmat.2015.03.036.
- [4] M. Sambucci, A. Sibai, and M. Valente, “Recent Advances in Geopolymer Technology . A Potential Eco-Friendly Solution in the Construction Materials Industry: A Review”, *J. Compos. Sci.* vol. 5, no. 4, pp. 1-30; 2021. <https://doi.org/10.3390/jcs5040109>.
- [5] A.M. Mustafa Al Bakri, H. Kamarudin, M. Bnhussain, I. K. Nizar, and W. I. W. Mastura, “Mechanism and Chemical Reaction of Fly Ash Geopolymer Cement- A Review”, *J. Chem. Inf. Model.*, vol. 53, no. 5, pp. 1689–1699, 2013.
- [6] V. S. Le, V. V. Nguyen, A. Sharko, R. Ercoli, T. X. Nguyen, D. H. Tran, P. Ło’s, K. E. Buczkowska, S. Mitura, T. Špirek and P. Louda, “Fire Resistance of Geopolymer Foams Layered on Polystyrene Boards”, *Polymers*, 14, 1945, pp. 1-12, 2022. doi: [org/10.3390/polym14101945](https://doi.org/10.3390/polym14101945).
- [7] C. Bai and P. Colombo, “Processing, properties and applications of highly porous geopolymers: A review”, *Ceram. Int.*, vol. 44, no. 14, pp. 16103–16118, 2018, doi: 10.1016/j.ceramint.2018.05.219.
- [8] V. S. Le, M. M. Szczypinski, P. Hájková, V. Kovacic, and T. Bakalova, “Mechanical properties of geopolymer foam at high temperature”, *Sci Eng Compos Mater*; vol., 27, pp.129–138, 2020. doi: [org/10.1515/secm-2020-0013](https://doi.org/10.1515/secm-2020-0013)
- [9] M. E. Sultan, S. A. Abo-El-Enein, A. Z. Sayed, T. M. EL-Sokkary, and H. A. Hammad, “Incorporation of cement bypass flue dust in fly ash and blast furnace slag-based geopolymer”, *Case Stud. Constr. Mater.*, vol. 8, no. February, pp. 315–322, 2018, doi: 10.1016/j.cscm.2018.02.009.
- [10] N. A. Jaya, L. Yun-Ming, H. Cheng-Yong, M. M. A. B. Abdullah and K. Hussin, “Correlation between pore structure, compressive strength and thermal conductivity



- of porous metakaolin geopolymer”, *Constr. Build. Mater.*, vol. 247, Jun. 2020, doi: 10.1016/j.conbuildmat.2020.118641.
- [11] A. C. I. Committee, *Building Code Requirements for Structural Concrete and Commentary (ACI 318M-11)*, American Concrete Institute, First Printing September 2011.
- [12] H. M. Khater, “Development and characterization of sustainable lightweight geopolymer composites”, *Ceramica*, vol. 65, no. 373, pp. 153–161, 2019. doi: 10.1590/0366-69132019653732551
- [13] G. M. Tsaousi, I. Douni, and D. Panias, “Characterization of the properties of perlite geopolymer pastes”, *Materiales de Construcción*, vol. 66, no. 324, pp. 1–8, 2016. doi: 10.3989/mc.2016.10415
- [14] S. T. Erdoğan, “Inexpensive intumescent alkali-activated natural pozzolan pastes”, *J. Eur. Ceram. Soc.*, vol. 35, no. 9, pp. 2663–2670, 2015, doi: 10.1016/j.jeurceramsoc.2015.03.017.
- [15] A. Bhogayata, S. V. Dave, and N. K. Arora, “Utilization of expanded clay aggregates in sustainable lightweight geopolymer concrete”, *J. Mater. Cycles Waste Manag.*, vol. 22, no. 6, pp. 1780–1792, 2020, doi: 10.1007/s10163-020-01066-7.
- [16] P. Ro, M. Król, and W. Mozgawa, “Lightweight geopolymer-expanded glass composites for removal of methylene blue from aqueous solutions”, *Ceram. Int.*, no. April, pp. 0–1, 2020, doi: 10.1016/j.ceramint.2020.05.011.
- [17] V. Medri, E. Papa, M. Mazzocchi, L. Laghi, M. Morganti, J. Francisconi and E. Landia, “Production and characterization of lightweight vermiculite/ geopolymer based panels”, *Mater. Des.*, vol. 85, no. 15, pp. 266–274, 2015, doi: 10.1016/j.matdes.2015.06.145.
- [18] B. Singh, M. Gupta, M. Chauhan, and S. K. Bhattacharyya, “Lightweight Geopolymer Concrete with EPS Beads”, *Constr. Build. Mater.*, vol. 85, no. June, pp. 78–90, 2015.
- [19] Z. Abdollahnejad, F. Pacheco-Torgal, T. Félix, W. Tahri, and J. Barroso Aguiar, “Mix design, properties and cost analysis of fly ash-based geopolymer foam”, *Constr. Build. Mater.*, vol. 80, no. May 2010, pp. 18–30, 2015, doi: 10.1016/j.conbuildmat.2015.01.063.
- [20] R. M. Novais, R. C. Pullar, and J. A. Labrincha, “Geopolymer foams: An overview of recent advancements”, *Progress in Materials Science*, vol. 109, no. November 2019, pp. 1-76, 2020, doi: 10.1016/j.pmatsci.2019.100621.
- [21] H. Yu, M. Xu, and C. Chen, “A review on the porous geopolymer preparation for structural and functional materials applications”, no. December 2021, pp. 1793–1813, 2022, doi: 10.1111/ijac.14028.
- [22] M. Nodehi, “A comparative review on foam-based versus lightweight aggregate-based alkali-activated materials and geopolymer”, *Innov. Infrastruct. Solut.*, vol. 6, no. 4, pp. 1–20, 2021, doi: 10.1007/s41062-021-00595-w.
- [23] T. Yang and C. Chien, “A Study on Chemical Foaming Geopolymer Building Materials”, *Advanced Materials Research*, vol. 853, pp. 202–206, 2014, doi: 10.4028/www.scientific.net/AMR.853.202.
- [24] V. Medri, E. Papa, J. Dedecek, H. Jirglova, P. Benito, A. Vaccari and E. Landi., “Effect of metallic Si addition on polymerization degree of in situ foamed alkali-aluminosilicates”, *Ceram. Int.*, vol. 39, no. 7, pp. 7657–7668, 2013, doi: 10.1016/j.ceramint.2013.02.104.

- [25] R. Arellano Aguilar, O. Burciaga Díaz, and J. I. Escalante García, “Lightweight concretes of activated metakaolin-fly ash binders, with blast furnace slag aggregates”, *Constr. Build. Mater.*, vol. 24, no. 7, pp. 1166–1175, 2010, doi: 10.1016/j.conbuildmat.2009.12.024.
- [26] R. Szabó and G. Mucsi, “Generally About Geopolymer Foams”, Conference: MultiScience - XXIX. microCAD International Multidisciplinary Scientific Conference, pp. 1–6, 2018, doi: 10.26649/musci.2015.014.
- [27] J. Wu, Z. Zhang, Y. Zhang, and D. Li, “Preparation and characterization of ultra-lightweight foamed geopolymer ( UFG ) based on fly ash-metakaolin blends”, *Constr. Build. Mater.*, vol. 168, pp. 771–779, 2018, doi: 10.1016/j.conbuildmat.2018.02.097.
- [28] K. Korniejenko and J. Miku, “Thermal insulation and thermally resistant materials made of geopolymer foams”, *Procedia Engineering*, vol. 151, pp. 410–416, 2016, doi: 10.1016/j.proeng.2016.07.350.
- [29] S. Wattanarach, S. Supothina, and P. Thavorniti, “Preparation and properties of metakaolin- based porous geopolymer formed with sodium perborate”, *J. Asian Ceram. Soc.*, vol. 10,no.3, pp.1–8, 2022, doi: 10.1080/21870764.2022. 2088755.
- [30] N. Böke, G. D. Birch, S. M. Nyale, and L. F. Petrik, “New synthesis method for the production of coal fly ash-based foamed geopolymers”, *Constr. Build. Mater.*, vol. 75, pp. 189–199, 2015, doi: 10.1016/j.conbuildmat.2014.07.041.
- [31] V. Ducman and L. Korat, “Characterization of geopolymer fly-ash based foams obtained with the addition of Al powder or H<sub>2</sub>O<sub>2</sub> as foaming agents”, *Mater. Charact.*, vol. 113, pp. 207–213, 2016, doi: 10.1016/j.matchar.2016.01.019.
- [32] S. Petlitckaia and A. Poulesquen, “Design of lightweight metakaolin based geopolymer foamed with hydrogen peroxide”, *Ceram. Int.*,vol.45, 1, pp. 1322-1330, 2019, doi: 10.1016/j.ceramint.2018.10.021.
- [33] H. A. Abdel-Gawwad and S. A. Abo-El-Enein, “A novel method to produce dry geopolymer cement powder”, *HBRC J.*, vol. 12, no. 1, pp. 13–24, 2016, doi: 10.1016/j.hbrcj.2014.06.008.
- [34] A. Antoni, L. Chandra, and D. Hardjito, “The impact of using fly ash, silica fume and calcium carbonate on the workability and compressive strength of mortar” , *Procedia Eng.*, vol. 125, pp. 773–779, 2015, doi: 10.1016/j.proeng. 2015.11.132.
- [35] S. Bishnoi, S. Maity, A. Mallik, S. Joseph, and S. Krishnan, “Pilot scale manufacture of limestone calcined clay cement : The Indian experience”, *Indian Concr. J.*, vol. 88, no. 7, pp. 22–28, 2014.
- [36] W. Wongkeo, “Effect of calcium carbonate on compressive strength and physical properties of alkali-activated lightweight concrete”, *Key Eng. Mater.*, vol. 751 KEM, pp. 550–555, 2017, doi:10.4028/www.scientific.net/KEM. 751.550.
- [37] Y. M. Liew, H. Kamarudin, A. M. Mustafa Al Bakri, M. LuqmanaI.Khairul Nizar, C. M. Ruzaidi and C. Y. Heah, “Processing and characterization of calcined kaolin cement powder”, *Constr. Build. Mater.*, vol. 30, no. December, pp. 794–802, 2012, doi: 10.1016/j.conbuildmat.2011.12.079.
- [38] D. Falliano, D. De Domenico, G. Ricciardi, and E. Gugliandolo, “Experimental investigation on the compressive strength of foamed concrete: Effect of curing conditions, cement type, foaming agent and dry density”, *Constr. Build. Mater.*, vol. 165, pp. 735–749, 2018, doi: 10.1016/j.conbuildmat.2017.12.241.

- [39] M. F. Zawrah, R. A. Gado, and R. M. Khattab, "Optimization of Slag Content and Properties Improvement of Metakaolin-slag Geopolymer Mixes", vol. 12, pp. 40–57, 2018, doi: 10.2174/1874088X01812010040.
- [40] D. M. A. Huiskes, A. Keulen, Q. L. Yu, and H. J. H. Brouwers, "Design and performance evaluation of ultra-lightweight geopolymer concrete", *Mater. Des.*, vol. 89, pp. 516–526, 2016, doi: 10.1016/j.matdes.2015.09.167.
- [41] D.M.A. Huiskes, "Ultra-lightweight geopolymer concrete Towards a monolithic building concept", Master thesis, Eindhoven University of Technology, 2015.
- [42] F. R. Svingala, "Alkali activated aerogels", A thesis accepted for the Degree of Master of Science on June 18th, 2009.
- [43] M. M. Al Bakri Abdullah, K. Hussin, M. Bnhussain, K. N. Ismail, Z. Yahya, and R. A. Razak, "Fly ash-based geopolymer lightweight concrete using foaming agent", *Int. J. Mol. Sci.*, vol. 13, no. 6, pp. 7186–7198, 2012, doi: 10.3390/ijms13067186.
- [44] C. Finocchiaro, G. Barone, P. Mazzoleni, C. Leonelli, A. Gharzouni, and S. Rossignol, "FT-IR study of early stages of alkali activated materials based on pyroclastic deposits (Mt. Etna, Sicily, Italy) using two different alkaline solutions", *Constr. Build. Mater.*, vol. 262, p. 120095, 2020, doi: 10.1016/j.conbuildmat.2020.120095.
- [45] M. F. Zawrah, R. S. Farag, and M. H. Kohail, "Improvement of physical and mechanical properties of geopolymer through addition of zircon", *Mater. Chem. Phys.*, vol. 217, no., pp. 90–97, 2018, doi: 10.1016/j.matchemphys.2018.06.024.
- [46] P. Olein, F. Stabilizer, Q. Yu, X. Li, Z. Wang, and J. Xue, "Characterization and Performance Evaluation of Metakaolin-Based Geopolymer Foams Obtained by Adding Palm Olein as the Foam Stabilizer", *Materials (Basel)*, vol. 15, no.10, pp.3570, 2022. doi: 10.3390/ma15103570.
- [47] T. Pantongsuk, P.Kittisayarm, N. Muenglue, S. Benjawn, P. Thavorniti, C. Tippayasam, S. Nilpaira ch, G. Heness and D. Chaysuwan, "Effect of hydrogen peroxide and bagasse ash additions on thermal conductivity and thermal resistance of geopolymer foams", *Mater. Today Commun.*, vol. 26, 202149, 2021, doi: 10.1016/j.mtcomm.2021.102149.
- [48] L. Chen, Z. Wang, Y. Wang, and J. Feng, "Preparation and properties of alkali activated metakaolin-based geopolymer", *Materials (Basel)*, vol. 9, no. 767, pp. 1–12, 2016, doi: 10.3390/ma9090767.
- [49] D. Dutta and S. Ghosh, "Effect of Lime Stone Dust on Geopolymerisation and Geopolymeric Structure", *International Journal of Emerging Technology and Advanced Engineering*, vol. 2, no. 11, 2012.
- [50] C. L. Hwang, M. Damtie Yehualaw, D. H. Vo, and T. P. Huynh, "Development of high-strength alkali-activated pastes containing high volumes of waste brick and ceramic powders", *Constr. Build. Mater.*, vol. 218, pp. 519–529, 2019, doi: 10.1016/j.conbuildmat.2019.05.143.