



Study Physicochemical and Thermal Properties of Eco-friendly Lightweight Geopolymers Incorporating Silica Sand Flour

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Abstract

Low-cost lightweight geopolymer mortars based on water-cooled slag, fly ash, and silica sand flour were prepared as a structural and thermally insulating material. The effect of chemical foaming agents such as hydrogen peroxide (H₂O₂) and sodium perborate tetrahydrate (NaBO₃·4H₂O) on thermal conductivity, bulk density, water absorption, 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 prepared lightweight geopolymers were given a compressive strength of 1.05 to 17 MPa. The compressive strength, bulk density, and thermal conductivity values decrease with increasing foaming agent content due to the decomposition of its chemical structure and releasing of oxygen bubbles. The results show that hydrogen peroxide mixes have better performance in the physio-chemical and thermal properties than sodium perborate mixes to achieve low thermal conductivity (0.21–0.24 W/mK) with compressive strength values (1.18–3.45 MPa) for MS-H1 and MS-H2 mixes, respectively. According to the results of bulk density (454–800 kg/m³), MS-H1, MS-H2, MS-B3, and MS-B4 mixes can be considered ultra-lightweight. Using silica sand flour in powder form improves the physicochemical and thermal properties of the lightweight geopolymer and decreases the production cost of the lightweight geopolymers.

Keywords Lightweight geopolymers · Hydrogen peroxide · Sodium perborate · Thermal insulators · Silica sand flour · Sodium aluminosilicate gel

1 Introduction

The world is currently witnessing a state of imbalance in climatic conditions with record rainfall, devastating floods, and forest fires as a result of global warming resulting from greenhouse gas emissions, primarily carbon dioxide (CO₂). Therefore, the signatories to the Glasgow climate change conference–October–November 2021 Pact (197 countries) agreed on several items, including the need to work in this critical decade to reduce carbon dioxide emissions by 45% by 2030 compared to the 2010 level [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 (CO₂) as a by-product of the combustion of limestone [3]. This production process not only emits large quantities of carbon dioxide (CO₂) but also needs a large amount of energy. In general terms, buildings consume 40% of global energy during the life cycle [4].

Brick is an important building material used with ordinary Portland cement (OPC) for a building wall. In addition to the consumption of large quantities of energy and the emission of harmful gases to humans and the environment, the production of traditional bricks from clays leads to a reduction in the area of agricultural land, for example, 1.5 million m³ of clay of agricultural land is bulldozed to fabricate one billion bricks [5].

Greater efforts have recently been undertaken to find an eco-friendly alternative to conventional bricks and ordinary Portland cement [6]. Geopolymers are one of the most

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suitable eco-friendly alternative 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 [7]. Lightweight geopolymers are low-density materials that have the following properties:

- (1) Good physical, chemical, and thermal stability
- (2) Low thermal conductivity and fire resistance [8]
- (3) Appropriate compressive strength, low toxicity, and low cost of transport
- (4) Low carbon dioxide (CO_2) emission and low energy consumed in the production process [9, 10].
- (5) Lower setting times, rapid hardening, and low shrinkage [11].

Due to their excellent properties, geopolymers are widely used in different fields besides building fields such as pH regulators, membrane separation, catalysts, adsorbent of metal ions and dyes, moisture management, sound absorption [12], evaporative cooling, and air purification [13, 14]. Using lightweight geopolymers as insulator decreases energy consumption and the cost of heating and cooling inside buildings [15].

According to the American Concrete Institute (ACI 318 M-11) classification, lightweight geopolymer concrete is grouped into three types based on the following properties: bulk density, compressive strength, and thermal conductivity. Type I is structural lightweight concrete with compressive strength of more than 17 MPa, thermal conductivity in the range of 0.4–0.7 W/mK, and bulk density of 1440–1840 kg/m^3 . While type II is structural and insulating lightweight concrete with a compressive strength of 3.4–17 MPa, thermal conductivity is in the range of 0.22–0.43 W/mK, and a bulk density is 800–1400 kg/m^3 . Type III is considered an insulating lightweight concrete with compressive strength values between 0.7 and 3.4 MPa, a thermal conductivity of 0.065–0.22 W/mK, and a density of 240–800 kg/m^3 [16, 17].

There are two methods for manufacturing lightweight geopolymers according to the method of reducing density [18].

- (1) Replacing part of solid raw materials with a lightweight aggregate such as

(a) Inorganic materials such as perlite, expanded clay aggregates, expanded glass aggregates, and Vermiculite [19–23].

(b) Organic materials such as cellulose, polystyrenes [24], polyethylene, polyurethane, and other polymers. Some researchers [24] have indicated that these organic materials (polystyrene and polyurethane) are flammable and toxic [4]. On the other hand, inorganic materials (perlite and Vermiculite) are expensive.

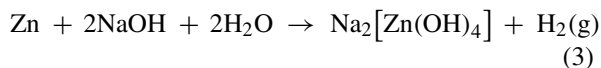
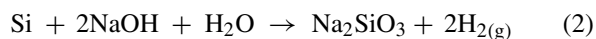
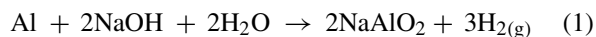
- (2) Addition of foaming agent into a paste (in this case geopolymers called foamed or aerated geopolymers).

Foamed geopolymers can be generated by some methods such as mechanical, chemical, thermal, and irradiation methods [9, 13, 14, 25, 26]. Prepared foamed geopolymers through irradiation method by microwave energy are obtained by putting the sample in a household microwave oven for 1 min at 850 W with a high concentration of NaOH, but the lowest density of produced geopolymers was 850 kg/m^3 which is considered one of the disadvantages of this method. However, this method has several advantages such as very quick intensity up time, and simple control [27].

S.T. Erdogan [20] succeeded in obtaining the pours of geopolymers by exposing perlite to high temperatures of 300 and 500 °C. Using a high concentration of NaOH as an alkaline activator for the prepared geopolymer given porous material has a bulk density of 450 Kg/m^3 . Mechanical foaming was taken place by introducing a huge volume of air bubbles through an air-pressure foam generator or by a foam-generating tank [12, 28–30].

A chemical foaming agent generates gases, such as (oxygen and hydrogen), into geopolymers paste and produces pores structure with low bulk density by two methods:

- (1) Reaction between metallic powders (chemical foaming agents) with an alkaline activator such as Al, Si, and Zn powders [9, 31–33].



- (2) Decomposition of some chemical foaming agents such as hydrogen peroxide [34–36], sodium perborate [4, 37], and sodium hypochlorite [9, 38, 39].

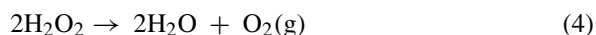
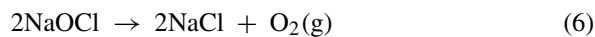
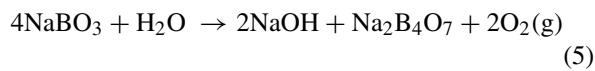


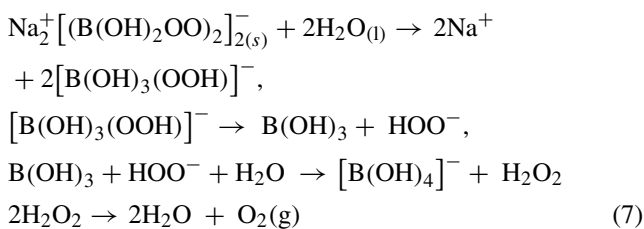
Table 1 Chemical compositions of aluminosilicate material (MSA) and silica sand flour (SSF)

Oxides, wt %	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	TiO ₂	MnO	SO ₃	L.O.I
ASM	38.00	21.10	2.19	26.60	6.19	0.26	0.62	2.21	0.38	1.31	0.78
SSF	99.16	0.69	0.013	0.11	–	0.01	0.003	–	–	–	0



According to V. Ducman and L. Korat [40] using hydrogen peroxide is better than AL powder because it gives a uniform distribution of pores, so we will study it in our work. In general, metallic particles formed inhomogeneous foams due to the local production of the gas from a consistent source term until its exhaustion [41].

Sodium perborate tetrahydrate which is used in our work is soluble in water and releases hydrogen peroxide then decomposes and releases oxygen gas [42].



Abdollahnejad et al. [4] studied the effect of two types of chemical foaming agents, hydrogen peroxide (H₂O₂) and sodium perborate (NaBO₃) with (1, 2, 3%) weight ratio with fly ash as precursors on compressive strength, bulk density, and thermal conductivity. The results showed that the bulk density, compressive strength, and thermal conductivity ranged from (700–1400) Kg/m³, (0.2–5)MPa, (0.1–0.2) W/mK, respectively, in the case of hydrogen peroxide mixes while ranged from (1200–1300) Kg/m³, (3–5)MPa, (0.05–0.2)W/mK, respectively, in case of sodium perborate mixes.

Viengsai Phavongkham et al. [43] used sodium perborate as a foaming agent in the presence of a mixture of fly ash and ground granulated blast furnace slag as precursors; the achieved foamed geopolymers have a density of 1100 kg/m³, compressive strength, and thermal conductivity ranged from (4.21–4.82) MPa, (0.27–0.32) W/mK, respectively. The previous studies worked on aluminum silicate material without using high proportions of sand, as well as using surfactants or foam stabilizers [35, 44, 45]. Many scientific researchers studied the utilization of silica sand in granular form to produce geopolymer mortars [4, 47]. But, the final products are

unsuitable for lightweight geopolymer applications as insulating materials due to the high thermal conductivity and bulk density of granular sand. To decrease the thermal conductivity and bulk density of the prepared geopolymer, using very fine and grounded silica sand (silica sand flour) with a low bulk density of 0.85 kg/m³ is considered a suitable solution compared with the normal granular silica sand which has a high bulk density 2.3–2.4 kg/m³. The aim of the work is using silica sand in powder form which is commercially called silica sand flour as filler with the same weight ratio of aluminosilicate material. Two different chemical foaming agents such as sodium perborate and hydrogen peroxide were used without using surfactants or foam stabilizers to achieve low-cost lightweight geopolymers with good mechanical and thermal properties.

2 Experimental Work

2.1 Materials

The aluminosilicate material succor Geo.101 used in this work was supplied by the succor company for chemicals, Cairo, Egypt. This binder is a mixture of Ground Granulated Blast Furnace Slag, Fly ash, and aluminosilicate additive with a weight ratio (75:20:5) % in all mixes. The silica sand flour was provided by an engineering company for mining (KNOUZ), El-Sadat City, Egypt. The chemical composition by XRF and XRD patterns of aluminosilicate binder and silica sand flour are shown in Table 1 and Fig. 1, respectively.

The used alkaline activator prepared a mixture between sodium hydroxide and liquid sodium silicate. Sodium hydroxide in the form of flakes is obtained from Alkout industrial projects company, Kuwait, 97.5% purity. Liquid sodium silicate (Na₂O = 13.9%, SiO₂ = 32.5%, Solid content 46.4%, density = 1.55 g/mL at 20 °C) is 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 is obtained 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 Tetra hydrate 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.

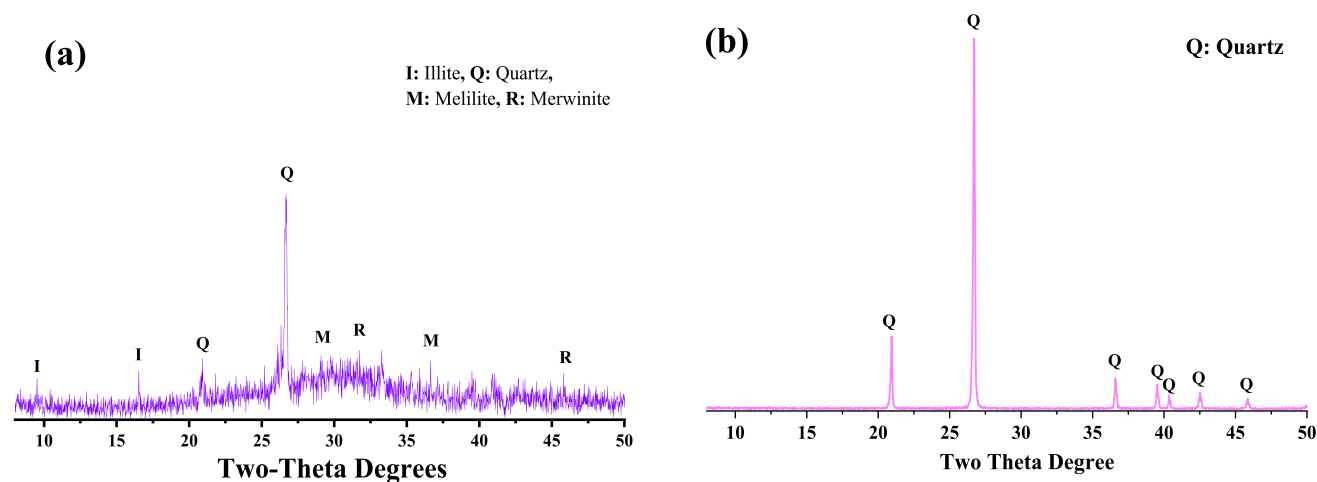


Fig. 1 XRD patterns of the raw materials: **a** aluminosilicate material (ASM), **b** silica sand flour (SSF)

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 g 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 and then kept for 24 h at room temperature before use to allow for equilibration [46].

2.2.2 Preparing Lightweight Geopolymers

In the Case of Hydrogen Peroxide (H₂O₂) Mixes The aluminosilicate materials were mixed with silica sand flour in a porcelain ball mill for 15 min with a (1:1) weight ratio as a solid part of the mixture. The alkaline solution was separately mixed with different concentrations of hydrogen peroxide (1, 2, 3, 4 wt.%). The solid part was gradually added to the liquid and then mixed for 5 min to complete the homogeneity of the mortar.

In the Case of Sodium Perborate Tetrahydrate (NaBO₃·4H₂O) Mixes The aluminosilicate materials and silica sand flour with a (1:1) weight ratio were mixed with different percentages of sodium perborate tetrahydrate (1, 2, 3, 4 wt.%) by a porcelain ball mill for 15 min. The alkaline activator was placed in the mixer then the solid part was gradually added then mixed for 5 min.

2.3 Mix Design

The mixes design is illustrated in Table 2. The fresh mortars were cast in polystyrene mold with dimeters 150*150*100 mm and then kept to set at room temperature for 48 h 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. For thermal conductivity measurement, the fresh mortar was cast in plywood molds with diameters 30*30*5 cm and allowed to sit at room temperature (20–25 °C) for 48 h. Before being removed from the molds after 28 days of curing, the samples were oven dried at 70 °C for 24 h before testing.

3 Methods

The chemical composition of aluminosilicate material and silica sand flour can be determined using X-ray fluorescence (XRF), model Philips PW/1404. The test was run using Rh- α (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. X-Ray diffraction equipment model X'pert PRO with a secondary monochromator, Cu-radiation ($\lambda = 1.542 \text{ \AA}$) at 45 k.v., 35 M.A. A continuous mode was used for collecting data in the 2θ range from 5 to 50° and a scanning speed of 0.04°/sec before testing, samples were prepared by fine grinding to 25 μm using a HERZOG grinder (Herzog

Table 2 Mix design of lightweight geopolymers samples

Mix Code	Aluminosilicate material, (g)	Silica sand flour, (g)	Alkaline Activator, (g)	Sodium perborate, (g)	Hydrogen peroxide, (g)
MS-00	50	50	50	0	0
MS-H1	50	50	50	0	1
MS-H2	50	50	50	0	2
MS-H3	50	50	50	0	3
MS-H4	50	50	50	0	4
MS-B1	50	50	50	1	0
MS-B2	50	50	50	2	0
MS-B3	50	50	50	3	0
MS-B4	50	50	50	4	0

Co., Germany). The functional groups and chemical bonds were identified using a Fourier-transform infrared spectroscopy (FTIR) spectrometer of type Nicolet 10, Thermo Fisher, USA. The reading of spectra was recorded in the 400–4000 cm^{-1} using a KBr binder with a resolution of 4 cm^{-1} at room temperature. FTIR-IR spectra were recorded in the range 400–4000 cm^{-1} using a KBr binder with a resolution of 4 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 h. at 105 °C in an electric oven according to the following Eq. (8):

$$d = m/V \quad (8)$$

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 water absorption and porosity were carried out using an Archimedes method according to ASTM C642 –13. The water absorption can be calculated by Eq. (9).

$$\text{Water absorption (\%)} = \frac{M_{\text{immersion}} - M_{\text{dry}}}{M_{\text{dry}}} \quad (9)$$

where $M_{\text{immersion}}$ is the mass of water-immersion specimen (g), and M_{dry} is the mass of dry specimen (g). The porosity of lightweight geopolymers is obtained by Eq. (10) [47, 48].

$$\text{Porosity (\%)} = \left(1 - \frac{\text{Bulk density}}{\text{apparent density}} \right) \times 100\% \quad (10)$$

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 cm^3) were dried for 48 h. 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. A Laser Comp instrument was used to measure the thermal conductivity of the prepared samples following the steady-state method as per ASTM C518-21, Standard Test Method for Steady-State Thermal Transmission Properties using the Heat Flow Meter Apparatus on a 30*30*5 cm slab after 28 days of curing, the samples were oven dried at 100 °C for 24 h before testing.

4 Results and Discussion

4.1 Starting Material Characteristics

The chemical composition of aluminosilicate material (ASM) and silica sand flour (SSF) measured by X-ray fluorescence (XRF) is illustrated in Table 1. The major oxides of aluminosilicate material are silica (SiO_2), alumina (Al_2O_3), calcium oxide (CaO), and magnesium oxide (MgO). Silica sand flour (SSF) consists of over 99 wt.% SiO_2 . The XRD patterns of aluminosilicate material and silica sand flour are shown in Fig. 1. It indicated that silica sand flour is mainly composed of crystalline quartz, while XRD of aluminosilicate material shows a halo hump between $2\theta = 20$ and 40 with some semi-crystalline phases such as merwinite and melilite; this refers to an amorphous phase of ground granulated blast furnace slag. Illite phases appeared at the aluminosilicate material XRD pattern which indicates the presence of fly ash in aluminosilicate material [49].

4.2 Bulk Density and Porosity

Figure 2 shows bulk density and porosity for lightweight geopolymers prepared with various weight ratios of hydrogen peroxide and sodium perborate tetrahydrate aged for

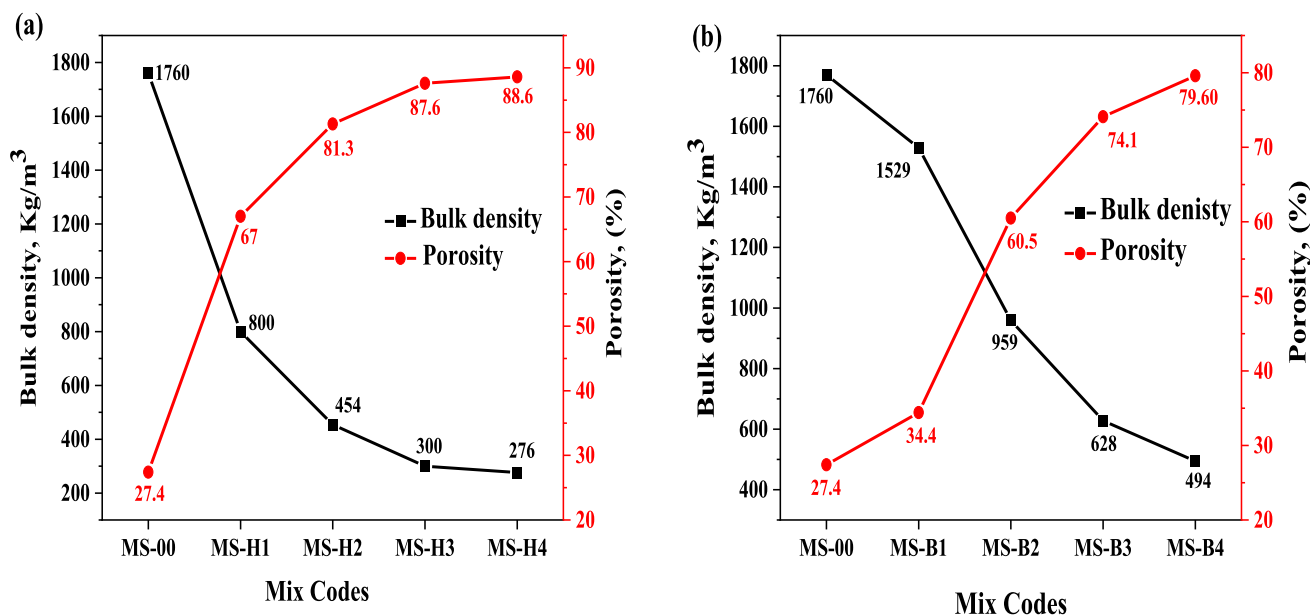


Fig. 2 a Bulk density and porosity of hydrogen peroxide mixes and control sample. b Bulk density and porosity of sodium perborate mixes and control sample

28 days at room temperature. In general terms, the increase in 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 Eqs. 4 and 7. The bulk density of control sample MS-00 is 1760 kg/m³, while the bulk density values of lightweight geopolymers with different values of H₂O₂ are 800, 454, 300, and 276 kg/m³ for MS-H1, MS-H2, MS-H3, and MS-H4, respectively, as shown at Fig. 2a.

MS-H4 mix gave the lowest bulk density and the highest total porosity values compared with other mixes (MS-H1, MS-H2, MS-H3) due to the increase in the hydrogen peroxide content in the geopolymeric matrix, while the bulk density values of lightweight geopolymers with different values of NaBO₃·4H₂O are 1529, 959, 628, and 494 kg/m³ for MS-B1, MS-B2, MS-B3, and MS-B4 mixes, respectively. The MS-B4 mix gave the lowest bulk density and the highest total porosity (79.60%), while the mixture of (MS-B1) showed the highest bulk density with porosity up to (34.40%) compared with other sodium perborate mixes.

The results indicate that using a lower percentage of hydrogen peroxide in the MS-H1 mix (1% H₂O₂) gave a bulk density of 800 kg/m³ and total porosity of around 67%, which is regarded as insulating material according to the American Concrete Institute (ACI 318 M-11) classification for lightweight geopolymers. To achieve the above results of the MS-H1 mix, it must be used a higher percentage of sodium perborate as the foaming agent such as 3% sodium perborate

in the MS-B3 mix. According to the obtained results of previous studies, MS-H1, MS-H2, and MS-B3 mixes exhibited good results in terms of bulk density and total porosity [16, 17, 26].

4.3 Water Absorption

Figure 3 shows water absorption by immersion for lightweight geopolymers prepared with various weight ratios of hydrogen peroxide and sodium perborate tetrahydrate aged for 28 days at room temperature. The water absorption of control sample MS-00 is 13%, while the water absorption of MS-H1, MS-H2, MS-H3, and MS-H4 is (19, 56.2, 91.9, and 94.2%), respectively. In the case of sodium perborate mixes, the water absorption ranged between 14.4 and 81.1%. As expected, the addition of hydrogen peroxide or sodium perborate tetrahydrate increases water absorption as a result of increased porosity. Figure 4 shows the relation between water absorption, bulk density, and porosity in hydrogen peroxide and sodium perborate mixes. From Fig. 4, it is possible to extract or predict a suitable lightweight geopolymer mix based on the final applications.

4.4 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 are plotted in Fig. 5. The compressive strength of the control sample MS-00 is 27.86 and 33.80 MPa at 7 and

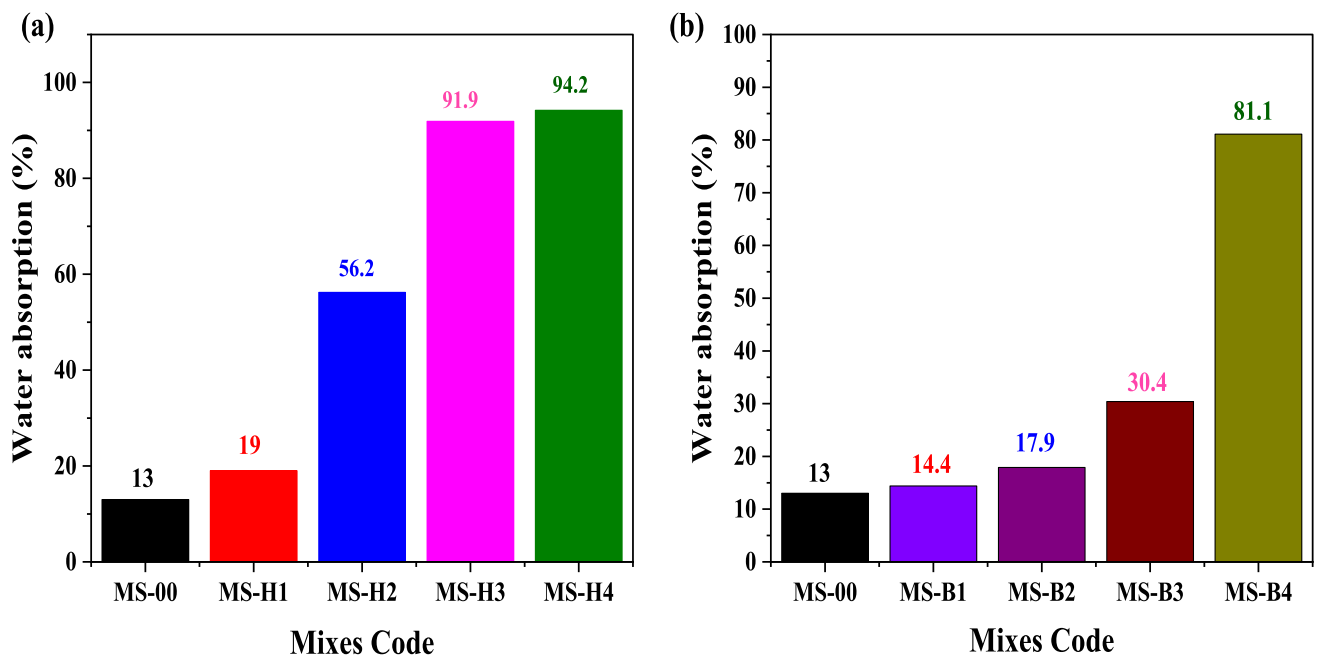


Fig. 3 a Water absorption of hydrogen peroxide mixes and control sample b Water absorption of sodium perborate mixes and control sample

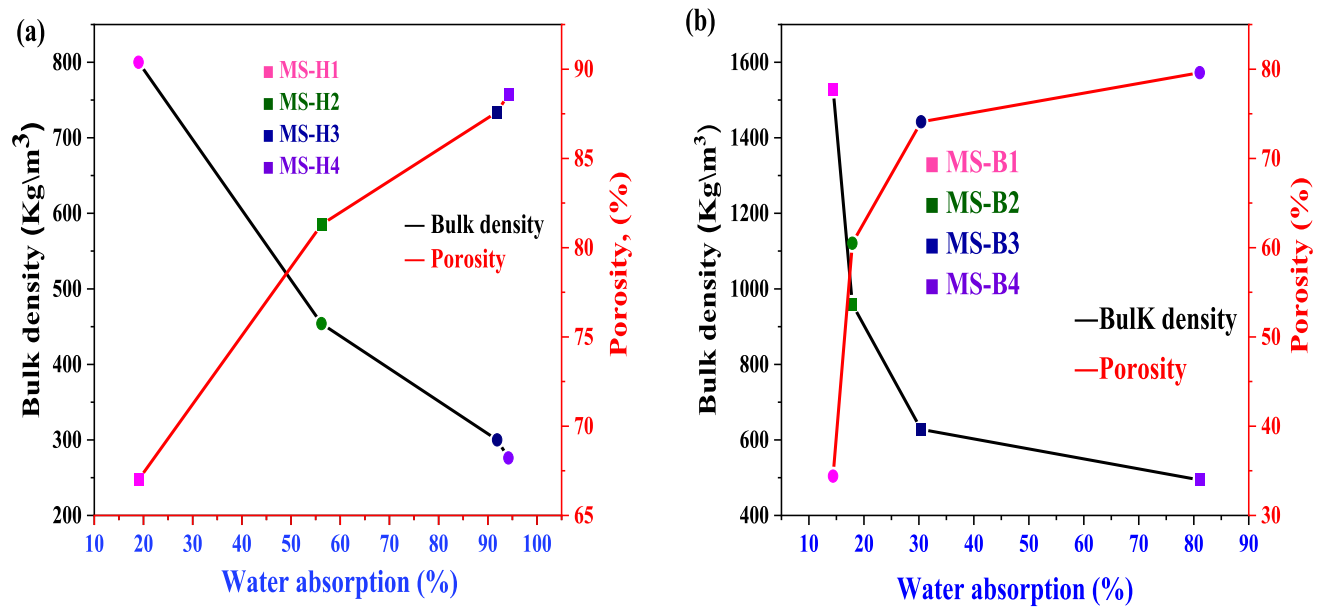


Fig. 4 Relation between water absorption, bulk density, and porosity in a hydrogen peroxide and b sodium perborate mixes

28 days, respectively. In general, the 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.

At a higher value of the foaming agent, the formed bubbles were fused leading to the formation of large voids and a decrease in the strength. In the case of hydrogen peroxide mixes (Fig. 5a), the compressive strength values for MS-H1 and MS-H2 were given 3.01 and 0.99 MPa at 7 days in addition to 3.45 and 1.18 MPa at 28 days which can be considered

accepted lightweight geopolymers according to the previous studies [26]. On the other hand, MS-H1 and MS-H2 mixes can be classified (Class-II, III) according to American Concrete Institute (ACI 318 M-11) classification for lightweight concrete based on compressive strength measurement [17]. Moreover, with the increase in the hydrogen peroxide content in the geopolymer matrix (MS-H3 and MS-H4) mixes, the compressive strength gave unacceptable values due to the formation of large voids.

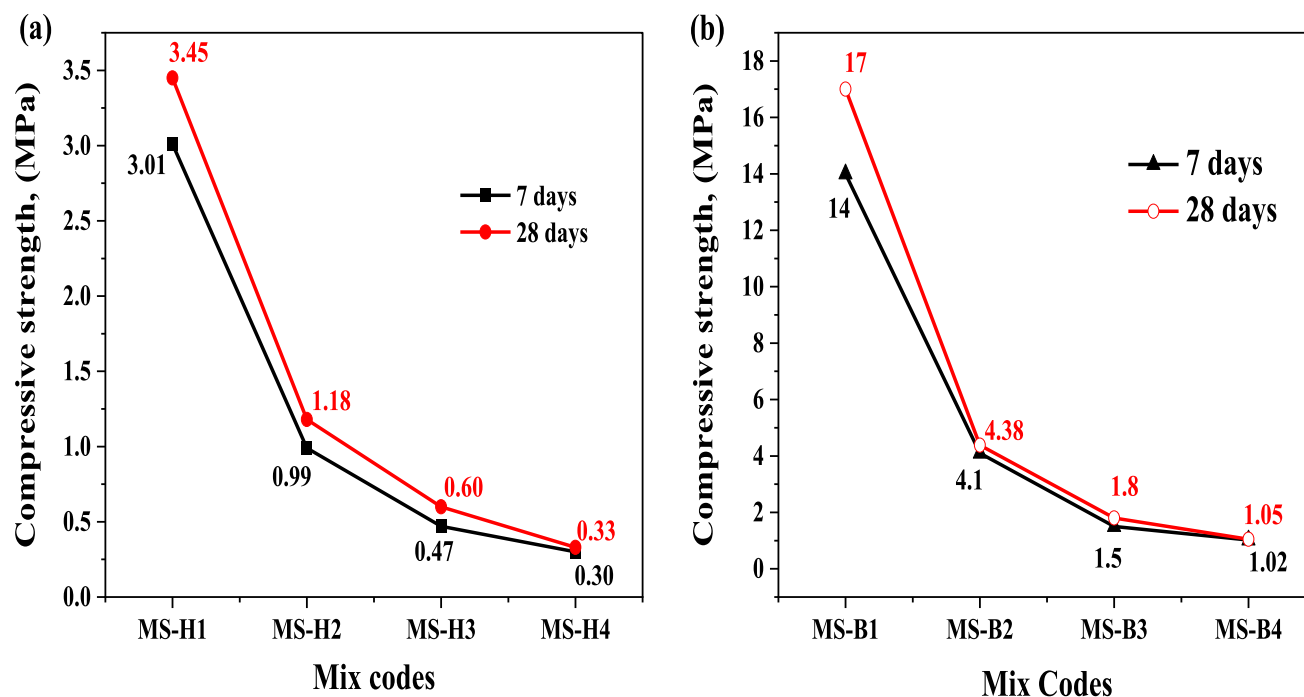


Fig. 5 a Compressive strength values of hydrogen peroxide light geopolymer mixes at 7 and 28 curing days. b Compressive strength values of sodium perborate light geopolymer mixes at 7 and 28 days

In the case of sodium perborate mixes, the compressive strength values were 14.00, 4.10, 1.50, and 1.02 MPa at 7 days of curing and 17.00, 4.38, 1.80, and 1.05 MPa at 28 days for MS-B1, MS-B2, MS-B3, and MS-B4, respectively. The results indicate that all samples have given acceptable compressive strength values according to the ACI 318 M-11 classification.

The MS-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). Here, the MS-B2 mix can be classified as structural and insulating lightweight concrete (Class-II). Finally, MS-B3 and MS-B4 mix can be considered insulating lightweight concrete because the compressive strength values lied between 3.4 and 0.7 MPa. The relation between the compressive strength, bulk density, and porosity for hydrogen peroxide and sodium perborate mixes is shown in Fig. 6a, b. By comparison, it can be concluded that the sodium perborate mixes were given higher compressive strength and bulk density values with lower porosity values compared with hydrogen peroxide mixes at the same foaming agent content.

4.5 FTIR Analysis

The FTIR spectra of three selected samples MS-00, MS-H1, and MS-B3 are presented in Fig. 7 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 cm^{-1} due to the strong vibration bending of (Si–O–Si and O–Si–O) bonds. Symmetric stretching vibration bands located at 693 and 778 for (Si–O–Si and Al–O–Si) bonds appeared at the same wave number and intensity [50, 51]. The weak bands located between 1450 and 1480 cm^{-1} and 1873 cm^{-1} corresponding to the stretching vibration bands of C–O bonds in the carbonate that formed by the reaction of carbon dioxide gas from the air with un-hydrated sodium in the geopolymers appeared [4, 46, 52].

The broadband between 2300 and 3500 cm^{-1} is referred to as stretching vibration O–H bonds in the hydrated water in the geopolymers matrix. On the other hand, the weak characteristic band at 1648 cm^{-1} can be attributed to the bending vibration of O–H from water molecules and hydroxyl groups of sodium hydroxide and sodium silicate [53]. From FTIR spectra, the absence of new vibration bands with foaming agent samples (MS-H1 and MS-B3), which refer to all by-products from the decomposition of sodium perborate to sodium metaborate, was encapsulated in geopolymerization reaction in addition to the lower concentration of foaming agent compared with the raw materials of geopolymers [44].

4.5.1 X-ray Diffraction (XRD)

XRD of three selected samples: MS-00 (without a foaming agent), MS-H1 (mixed with H_2O_2), and MS-B3 (mixed

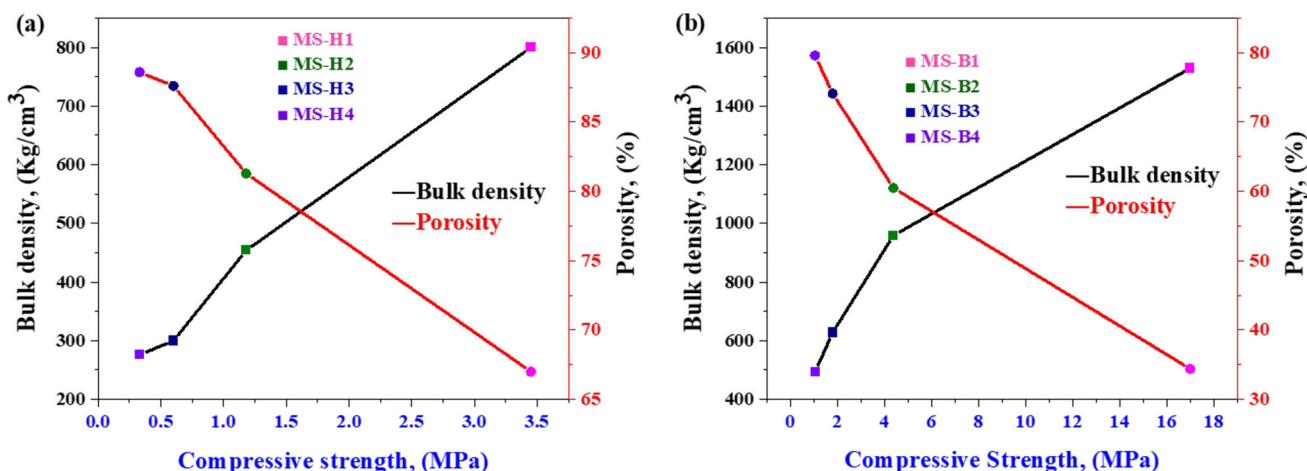


Fig. 6 a Relation between the compressive strength, bulk density, and porosity for hydrogen peroxide mixes. b Sodium perborate mixes

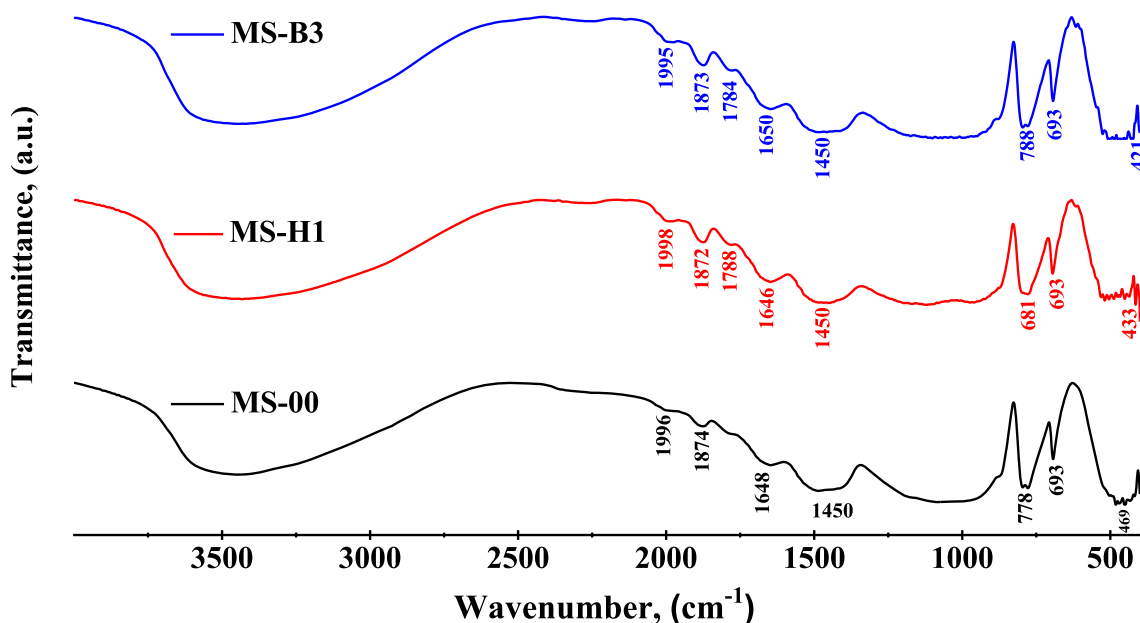


Fig. 7 FTIR of lightweight geopolymer samples prepared with/without foaming agents

with NaBO₃·4H₂O) cured for 28 days at room temperature as shown in Fig. 8. All XRD patterns demonstrate the presence of the crystalline phase of quartz in geopolymer and lightweight geopolymers due to the low dissolution of the crystalline quartz phase as the main component of silica sand flour in an alkaline solution [54, 55].

On the other hand, the amorphous phase of calcium silicate hydrate (CaO·SiO₂·H₂O) appeared due to the high percentage of calcium in aluminosilicate materials as shown in XRF in Table 1. (26.60%, CaO). The amorphous phase of sodium aluminosilicate gel (N–A–S–H) gives a clear indication that the geopolymerization reaction has taken place. (N–A–S–H) is the main factor in determining the mechanical properties of the geopolymer [3, 56, 57].

In addition, there is a great similarity in the phases of lightweight geopolymers with/without foaming agents. The appearance of the weak phase of crystalline Illite in MS-H1 and MS-B3 mixes may be related to the accelerating of the setting time in the case of adding hydrogen peroxide and sodium perborate.

4.6 Morphology and Microstructure

The morphology of all lightweight geopolymer mixes can be investigated by taking photographic images as shown in Fig. 9. The images showed that the pore size distribution of the acceptable samples exhibited uniform and homogeneous shapes without using surfactants or foam stabilizing agents. From the photographic images, it can be seen that these

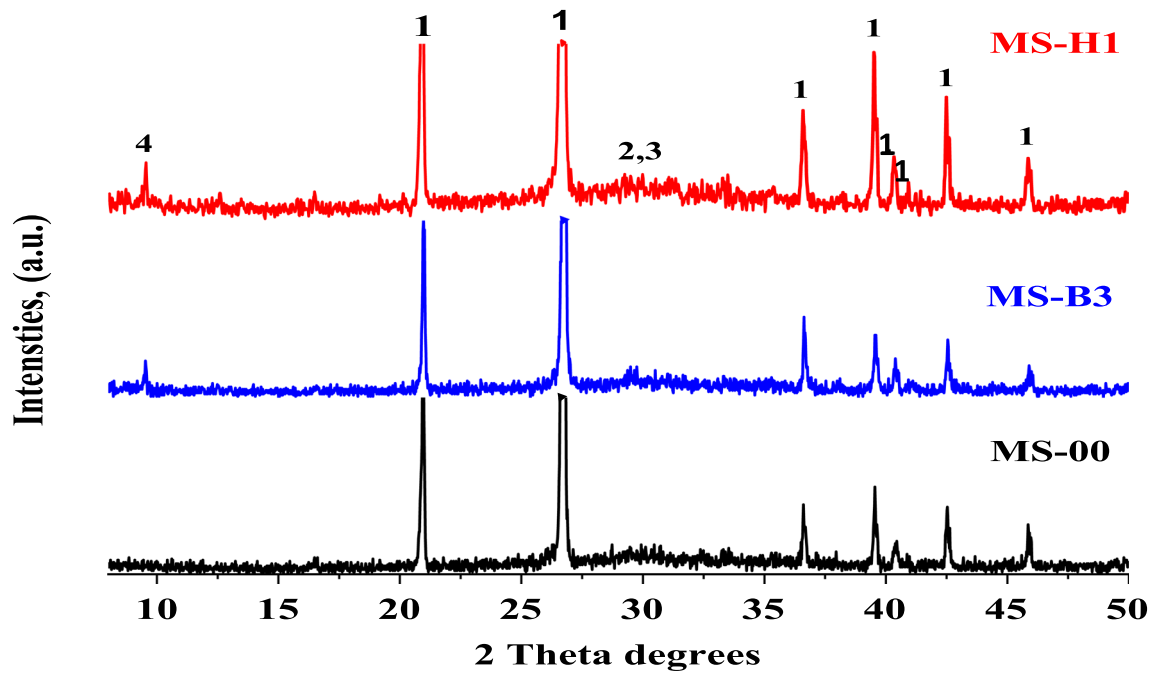


Fig. 8 XRD patterns of lightweight geopolymer samples prepared with/without a foaming agent aged 28 days (1: quartz; 2: calcium silicate hydrate; 3: sodium aluminosilicate, and 4: Illite)

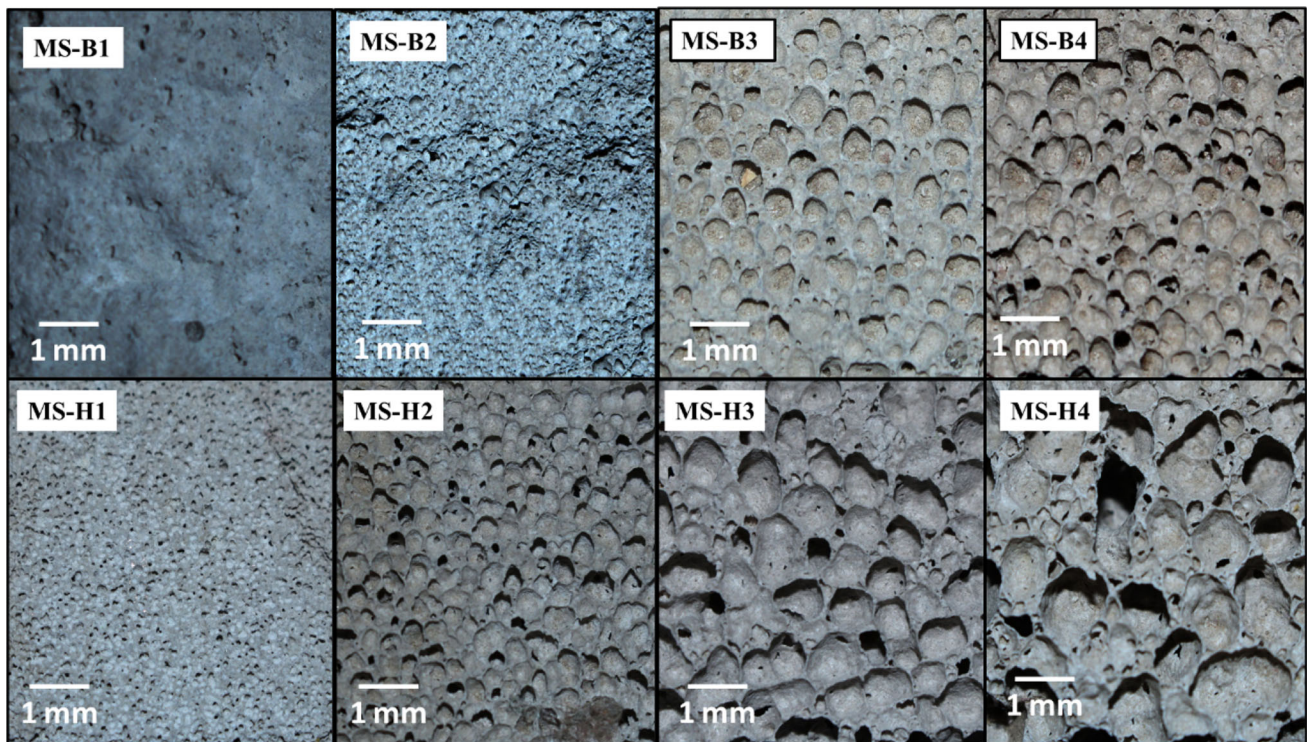


Fig. 9 Photographic images of lightweight geopolymer images for the different mixes taken at a 1-mm scale

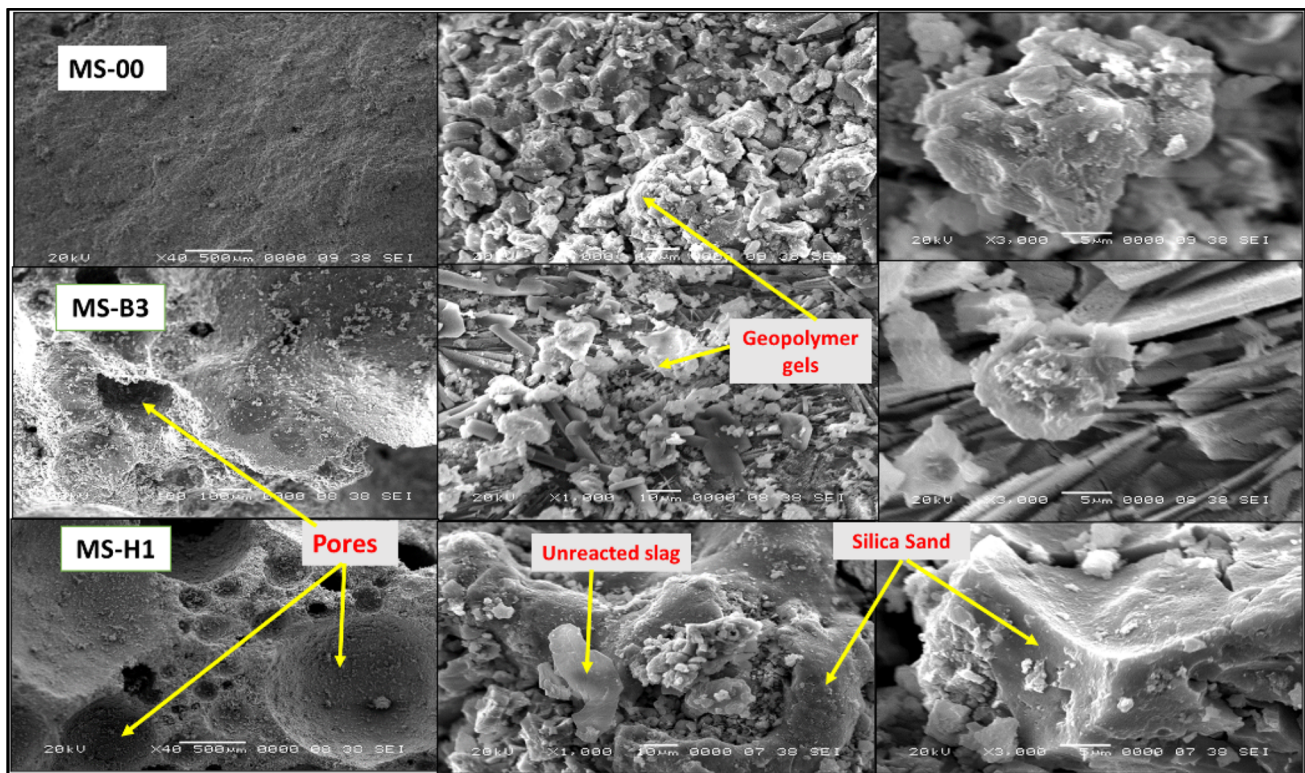


Fig. 10 SEM images for MS-00, MS-B3, and MS-H1 for three magnification values X-40, X-1000, and X-3000 at 28 days of hydration

pores formed by the foaming agents are mainly spherical closed pores. The increase in 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. MS-H3 and MS-H4 exhibited largely destroyed shapes; therefore, these mixes gave unacceptable results in compressive strength.

The microstructure of some selected samples MS-00, MS-B3, and MS-H1 is examined by SEM at three magnifications (X40, X-1000, and X-3000) with scales of 500, 10, and 5 μm , respectively in Fig. 10. The results show the presence of unreacted slag, fly ash, 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) [58]. SEM images exhibited high percentages of unreacted silica sand flour due to the high crystallinity of silica in the form of quartz phase. These results are confirmed by XRD patterns for the same mixes [11]. 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 [59].

4.7 Thermal Conductivity

The wide use of lightweight geopolymers is due to their excellent insulating properties as thermal conductivity is considered one of the most important parameters affecting thermal insulation [60].

In general, geopolymers are good energy conservers due to having a lower thermal conductivity if compared to traditional Portland cement. The amorphous phase of sodium aluminosilicate (N-A-S-H) in geopolymers reduces heat transfer [61]. Although silica sand flour has a high thermal conductivity of about (6–11 W/mK) [16], in addition, to use large quantities in the prepared lightweight geopolymers, the obtained results of the thermal conductivity for MS-H1, MS-H2, MS-B2, MS-B3 mixes give good results compared to previous studies [37].

The decomposition of foaming agents leads to releasing of oxygen bubbles that cause forming of voids leading to increase in porosity and decrease in the thermal conductivity of the sample. Figure 11 shows the thermal conductivity of MS-00, MS-H1, MS-B2, and MS-B3 mixes aged for 28 days at room temperature. When the hydrogen peroxide content was added to the control sample MS-00 with two different doses 1 and 2% in MS-H1 and MS-H2 mixes, the thermal conductivity of lightweight geopolymer mortars decreases from 1.06 to 0.24 and 0.21 W/mK, respectively. On the

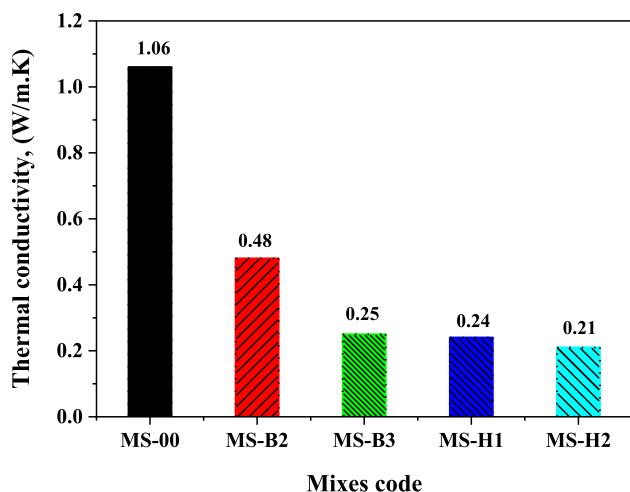


Fig. 11 The thermal conductivity of five selected samples aged for 28 days at room temperature

other hand, when the sodium perborate tetrahydrate content increased from 0% in the control sample (MS-00) to 2 and 3% in MS-B2 and MS-B3 mixes, the thermal conductivity of lightweight geopolymer mortars decreases from 1.06 to 0.48 and 0.25 W/mK, respectively. Hydrogen peroxide mixes are more efficient than sodium perborate mixes in terms of decreasing the thermal conductivity to use as good thermal

insulating materials. The relation between the thermal conductivity and compressive strength is illustrated in Fig. 12, which shows that (MS-H1) sample has an excellent compressive strength (3.45 MPa) with a low thermal conductivity of 0.24 W/mK. Figure 13 illustrates the relationship between the thermal conductivity with bulk density and porosity for selected samples.

5 Conclusions

This paper studied mainly the preparation of low-cost lightweight geopolymers used as insulators from waste materials and silica sand flour by chemical foaming methods without using surfactants or foam stabilizers and concluded some important points as follows:

1. Using insulation 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 were successfully produced at room temperature from the mixture containing silica sand flour as a filler by such ratio of aluminosilicate material in presence of hydrogen peroxide and sodium perborate

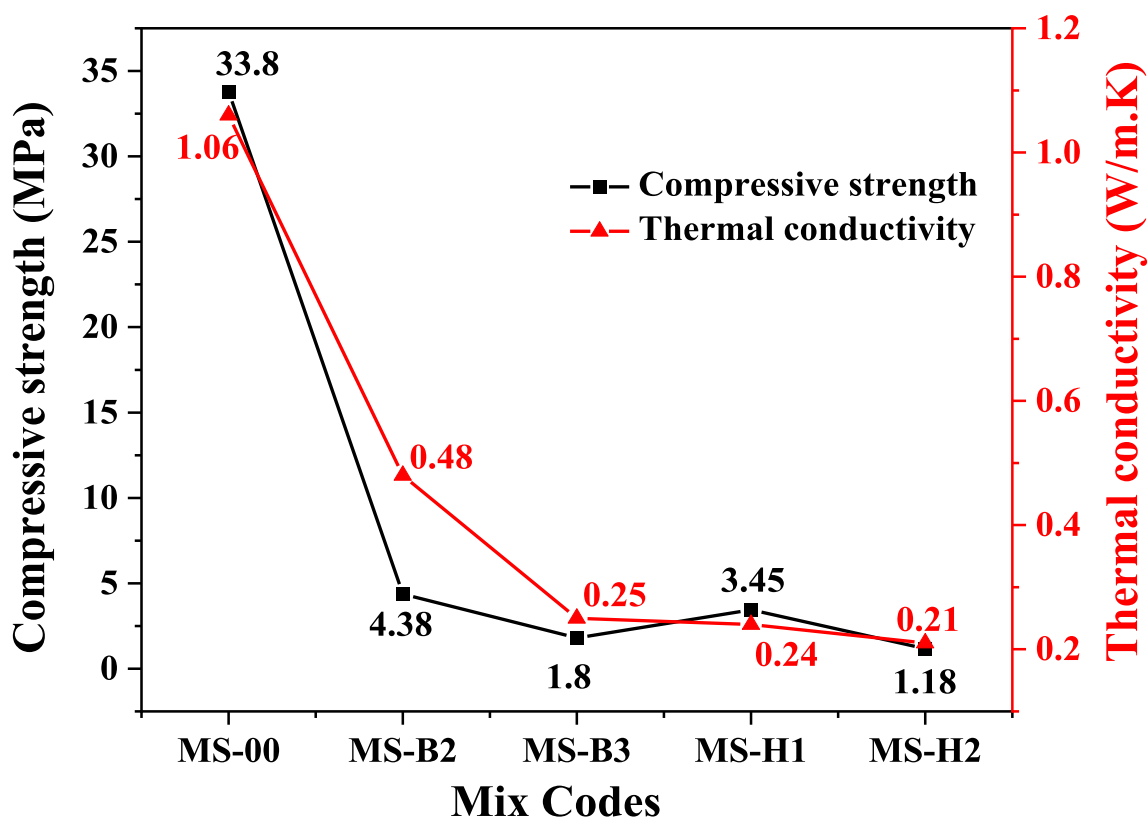


Fig. 12 The relation between thermal conductivity and compressive strength for selected samples

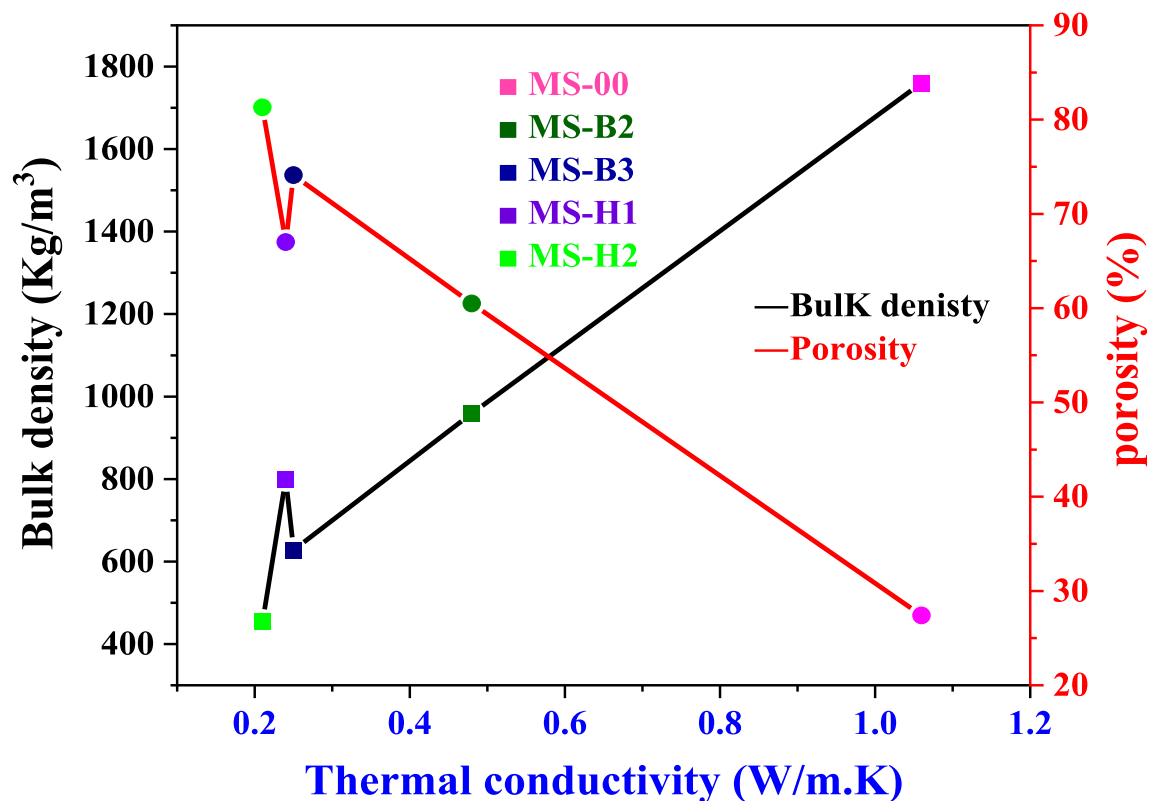


Fig. 13 The relation between thermal conductivity with bulk density, and porosity for selected samples

- tetrahydrate as foaming agents without using surfactants or foam stabilizers.
- As expected, the addition of hydrogen peroxide or sodium perborate tetrahydrate increases the porosity and water absorption, reducing compressive strength, bulk density, and thermal conductivity for all measured samples as a result of its decomposition and release of oxygen bubbles that cause forming of voids.
 - The measured compressive strength of lightweight geopolymer samples aged 28 days at room temperature, ranged from 0.33 to 3.45 MPa and 1.05 to 17 MPa in the case of hydrogen peroxide, and sodium perborate mixes, respectively.
 - Low thermal conductivity (0.21 W/mK) could be achieved, especially in the case of hydrogen peroxide mixes, which show higher efficiency than sodium perborate mixes in reducing thermal conductivity.
 - The produced lightweight geopolymers 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 to (ACI) classification.
 - The FTIR spectra of all prepared lightweight geopolymer samples have the same vibrational bands compared with the prepared sample without foaming agents.
 - There is a great similarity in the geometrical structures for XRD phases of lightweight geopolymers with/without foaming agents, except for the appearance of the weak phase of crystalline Illite in MS-H1, and MS-B3 mixes.
 - From SEM and high-resolution camera images, the distribution of pores through most samples was uniform. When an additional higher amount of foaming agents, pores gather together, so their number decreases and their size increases, and in all cases, their distribution is uniform.

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