



Preparation of Metakaolin Based Geopolymer Foam Using a Combination of Na and K Types of Alkali Activators

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HIGHLIGHTS

- Highlights could be presented by 3-5 bullets.
- points capturing the novelty of
- methods and/or results of the work.

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ABSTRACT

In this research, the OPC has been mixed with metakaolin, activator, hydrogen peroxide, and olive oil to synthesize hybrid geopolymer foam. Obtained results indicated internal heat release throughout OPC hydration in the combination. OPC has been employed as a calcium source in geopolymers (Geopolymer-Portland cement (HGPF)) to explore the curing process of geopolymers at ambient temperature. The functionality of geopolymer components and (HGPF) mixture, the elemental composition and proportion analyses have been compared. A principal aim of this research focuses on the development of geopolymer foam and conducting many tests such as physical tests that related to the surface area and pores size as well as compression of the foam to investigate the capacity of applying this foam in different applications that required good strength. Furthermore, microstructure tests using SEM and XRD techniques have been conducted to examine surface structure components. Overall, findings presented in this research shows that the materials selected to develop the geopolymer foam were compatible with each other giving high porosity with acceptable compression via optimizing the processing parameters by RSM.

1. Introduction

Geopolymers are initially developed in the 1970s by Joseph Davidovits. Geopolymers are inorganic polymeric materials having chemical compositions comparable to zeolites but amorphous or semi-crystalline structures. Structurally, Geopolymers are structured from a combination of aluminosilicate materials (with high Si and Al content) and an alkaline-activating solution (mainly Na⁺ or K⁺ silicates and hydroxides). Geopolymers were primarily used in civil construction after introducing fire-resistant alternatives to thermoset polymers [1]. Because of their superior mechanical qualities, endurance, and adhesion to iron and conventional concrete, they can replace concrete and comparable products containing Portland cement.

Geopolymers have been initially thought of as a fire-resistance material [2]. Nevertheless, since the early 1980, these materials have mostly been explored as a substitute for typical Portland cement, owing to their reduced CO₂ embodied [3], as well as performance benefits (for example, early compressive strength; stronger chemical and heat resistance) [4]. An additional benefit, especially in light of the new circular economy paradigm, is the utilization of diverse industrial waste streams as raw materials, including fly ash [5], metallurgical slags [6], and glass wastes [7].

Notwithstanding strenuous contributions, the full potential of geopolymers is still to be realized [8] that has been linked to legislative, societal, and technological hurdles [9]. Expanding the potential applications of geopolymers to places where Portland cement did not match the criteria is a fundamental motivator for their utilization. Porous geopolymers, which are purposely intended to include pores, have recently been investigated for high-added-value applications such as thermal [10] and acoustic insulation [11], fire resistance [12], adsorbents [13], and pH regulators [14]. Nevertheless, this method (for example, intentionally generating porosity in the samples) is still investigated in other industrially significant applications, including their usage as catalysts or catalytic supports [15]. The number of papers on geopolymer foams (lightweight solid materials) has risen dramatically in recent years, indicating a spike in interest.

The geopolymers' mechanical characteristics are linked closely to density so that low-density geopolymers will demonstrate unacceptably low strength [16]. Duxson et al. suggest a tendency towards enhanced mechanical intensity at higher SiO₂/Al₂O₃ proportions and increased porosity at greater H₂O/SiO₂ proportions [17]. Besides that, adequate mechanical

strength could also be attained with the regulated introduction of foaming agents where an optimal density and pore structure is obtained. Various foaming agents may be utilized to synthesize low-density geopolymers, and one of them is surfactants. Surfactants are liquid additives that can manufacture lightweight materials through entraining air throughout blending [16].

Geopolymers have long been researched as alternate cementitious materials. Setting time is faster and environmental impact during production is significantly lower as compared to ordinary Portland cement. For every ton produced, only 0.15-0.20 tons of CO₂ was created by geopolymer cement versus 1 ton for Portland cement [18]. They also outperform Portland cement in compressive strength [19].

There are many advantages for geopolymer including [20]: High compressive strength gain in some cases, Low CO₂ footprint, good resistance against corrosion, especially if mixed with filler of PTFE, Fire resistance (1200°C) and no liberation of toxic fumes if heated, Geopolymer cement was able to make strong bonds old and fresh substrates, steel, boro-silicate ceramics and glass at ambient temperature. Outstanding resistance against chloride chemical attacking, involving sulphate, different acids and seawater and bleeding free, high isolation and low permeability.

Foam concrete is a lightweight material made up of a cementitious binder, space, and sometimes fine particles. The binder is usually OPC-based, and it has blended with additional ingredients to give it diverse properties. Fly ash and GBFS are often used to replace the cement component. Adding granitic limestone and silica fines to foam concrete may occasionally lower the cost of manufacture. Controlling the size, distribution, and character of the voids is an important phase in foam concrete. Endogenous gas production or the introduction of a substantial volume proportion of air bubbles creates these gaps. Diverse sizes and distributions of bubbles will be disseminated in the mixture due to these various processes, affecting the characteristics of the geopolymer foam concrete. The thermal qualities of the concrete are one of these critical qualities [21].

The rise in popularity of foam concretes is primarily due to their excellent thermal insulation properties. Foam concretes containing geopolymers have been shown to have low thermal conductivity, meaning that the heat energy transferred to them is retained. This is significant in the world's cold climates since it enables buildings to retain heat, reduce the need for heating systems, and make buildings more energy-efficient [22]. This is yet another crucial aspect of addressing the climate crisis [23]. Buildings that use less energy and generate fewer emissions are becoming increasingly popular. Heat loss thru the walls of a building is a significant contributor to high energy consumption. Less heat is lost when concretes like geopolymer foam concretes are used, effectively addressing this issue [24, 25].

Previously published work [26 – 29] focused on synthesizing organic–geopolymer hybrid and composite binders to improve the geopolymer's mechanical and thermal characteristics. A co-reticulation process occurs between the organic component (epoxy or siloxane resin) and the inorganic matrix to produce these materials (geopolymer). These materials have exhibited a broad range of performance depending on reaction circumstances and chemical composition, indicating that they have substantial potential applications in various sectors, including structural, fire-resistant, and insulating systems.

Metakaolin and an alkali silicate solution have been combined with dialkyl siloxane oligomer mixtures (GHF-MK) to create novel organic-geopolymer-based hybrid foams [26]. In situ foaming agent SiO powder has been employed in various quantities to produce foams with densities ranging from 0.25 to 0.85 g/cm³. These new lightweight materials have low heat conductivity and high fire resistance. Furthermore, they demonstrated outstanding mechanical capabilities that outperformed pristine geopolymer foams described in the literature and commercially available AAC materials. [30].

In the current research, the ordinary Portland cement has been mixed with Metakaolin (MK-750) powder, sodium silicate, sodium hydroxide, silica gel, and hydrogen peroxide to prepare the geopolymer.

2. Experimental Work

2.1 Materials

Kaolin, sodium silicate, sodium hydroxide, potassium hydroxide, silica gel, hydrogen peroxide, and ordinary Portland cement have been utilized as starting materials to synthesis hybrid geopolymer foam. The materials source, pureness, and chemical formula utilized in this investigation are illustrated in Table 1. The wet chemical technique has been utilized to identify the chemical composition of kaolin. The Iraq geological and mining survey in Baghdad conducted this investigation. The findings of the kaolin's wet chemical analysis of kaolin are shown in Table 2. Furthermore, the XRD patterns of the Metakaolin (MK-750) powder used to prepare hybrid geopolymer foams and particle size distribution are shown in Figures 1 and 2, respectively.

Table 1: Chemical formula and purity of materials utilized in work.

Materials	Chemical formula	Purity (%)	Manufacturer
Sodium silicate	Na ₂ SiO ₃ .5H ₂ O	97.98	Thomas Baker, India
Sodium hydroxide	NaOH	97.99	Thomas Baker, India
Potassium hydroxide	KOH	97.99	Thomas Baker, India
Silica gel	SiO ₂	98.99	Thomas Baker, India
Hydrogen peroxide	H ₂ O ₂	30 w/v	Thomas Baker, India
Olive Oil	-	99	ZER, Turkey

Table 2: The kaolin wet chemical analysis results.

SiO ₂ %	Al ₂ O ₃ %	Na ₂ O %	K ₂ O %	CaO %	MgO %	Fe ₂ O ₃ %	TiO ₂ %	SO ₃ %	P ₂ O %	Cl %	LOI %
48.77	34.27	0.17	0.43	0.43	0.08	1.76	1.47	0.11	0.02	0.03	12.46

2.2 Preparation of Geopolymer Foam (GPF) Samples

The GPFs have been produced in the following way. Initially, the metakaolin (MK-750) and alkaline solution were mixed; geopolymer paste will be formed. After allowing the paste to sit for a bit, hydrogen peroxide (H_2O_2) has been introduced as a foaming agent, followed by olive oil as a stabilizing agent. The mixture was blended for approximately 2 minutes before it became a homogeneous slurry (see Figure 3). After that, the slurry has been forced into a cylindrical-shaped plastic mould with a diameter of 21 mm and a height of 42 mm, and cured for 24 hours at room temperature, and then demolded (see Figure 4). Eventually, the remaining olive oil was extracted by soaking it in hot water approximately (80 degrees Celsius) for 30 minutes and then repeating the process until it was clear.

In this investigation, the five monitored parameters have been N, H_2O (ml)/10.73 MK-750, OPC, foaming agent (wt%) and stabilizing agent (wt%) that have been utilized to prepare hybrid geopolymer foam, and their levels utilized have been shown in Table 3.

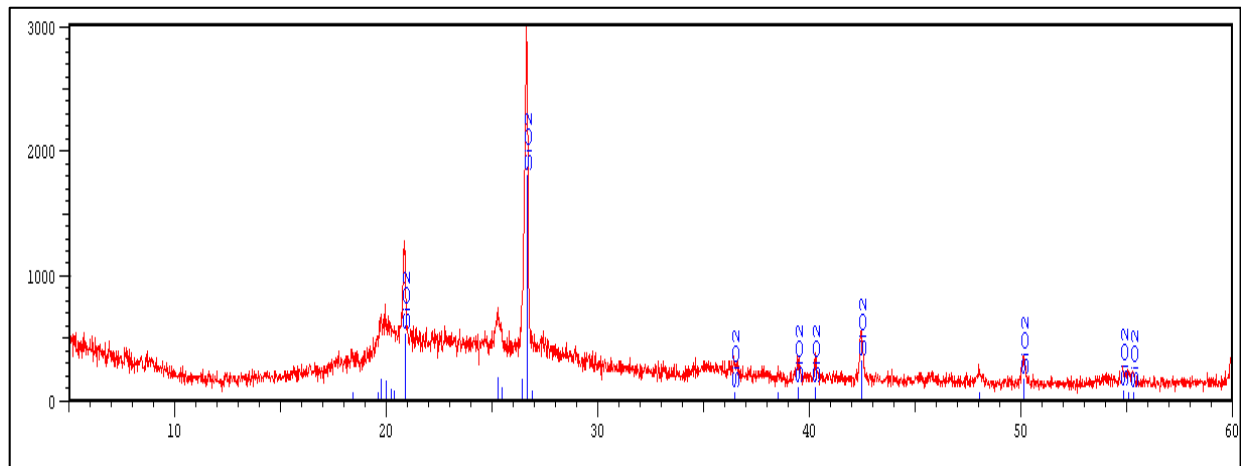


Figure 1: The XRD patterns of the Metakaolin (MK-750) powder

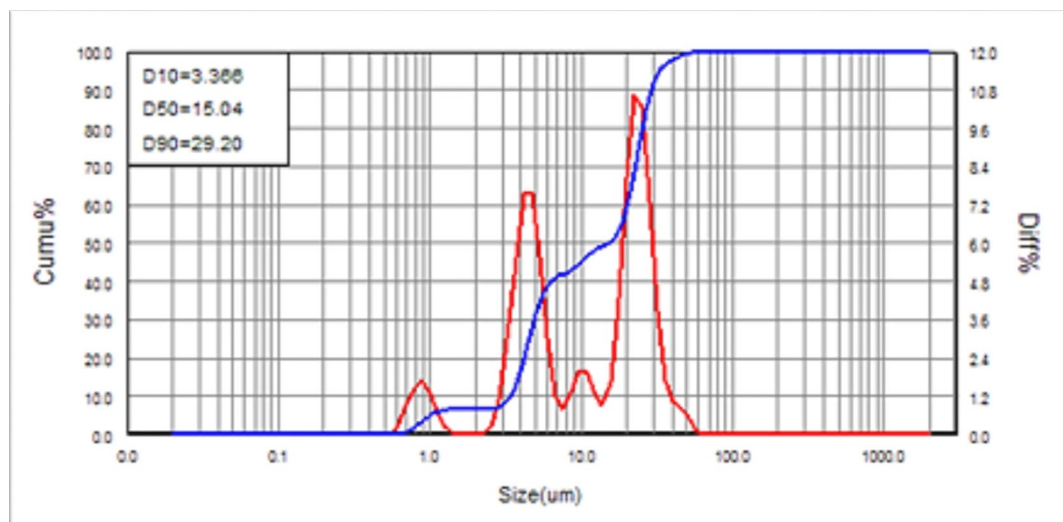


Figure 2: PZA chart of Metakaolin (MK-750) powder

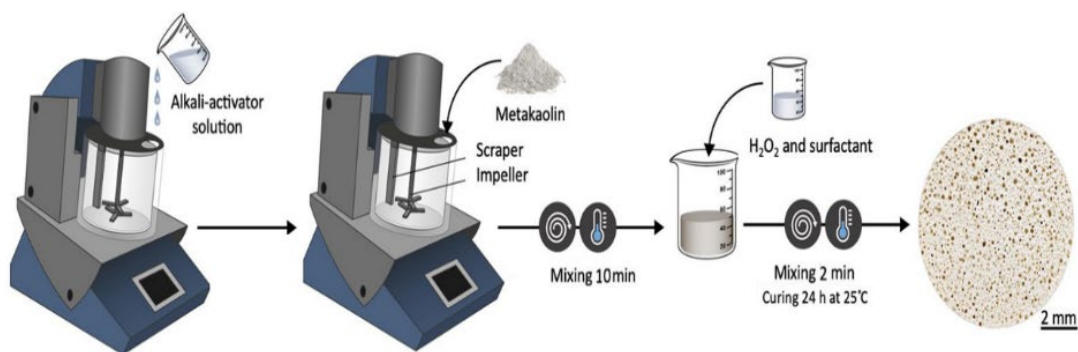


Figure 3: Scheme of the GPFs preparation procedure

Table 3: The affected parameters of the manufacture of the geopolymer foam.

N	H ₂ O (ml)/10.73 MK-750	OPC powder (wt%)	Foaming agent (wt%)	Stabilizing agent (wt%)
3.2	8	0	0	0
3.4	9	5	1	1
3.6	10	10	2	2
3.8	11	15	3	3
4.0	12	20	4	4


Figure 4: The specimen of geopolymer cement paste after curing 24 hr. at 25°C

3. Experiments Design

3.1 Physical analysis

Water absorption, apparent porosity, bulk density, apparent specific gravity (or true density), and total porosity of the hybrid geopolymer foam have been measured by Archimedes' method according to (ASTM C729-11) as follow:

- 1) The test samples have been dried to constant mass by heating in a furnace at 90 degrees Celsius for 6 h, cooled in desiccators to the normal temperature of a room. The dry mass, D , has been identified utilizing a four-digit electronic balance (Sartorius, Germany, Chemical Engineering Department/the University of Babylon, the volume has been calculated by the specimen dimensions, as well as by utilizing the four-digit high precision density tester GP-120S (Matsu Haku, Korea, College of the Materials Engineering/the University of Babylon).
- 2) The test specimens have been located in a distilled water pan and allowed to soak for an additional 24 h. then the mass identifies each specimen while suspended in water, S , has been identified.
- 3) The saturated mass, M , of each sample, has been calculated by gently blotting the surface with wetted cotton to eliminate excess water.
- 4) The following calculations have been made to find the physical characteristics of the cement nanocomposite samples, as well as the calculations carried out by utilizing the four-digit high precision density tester GP-120S (Matsu Haku, Korea, College of Materials Engineering/ University of Babylon):

$$\text{Water absorption (\%)} = \left(\frac{M-D}{D} \right) \times 100 \quad (1)$$

$$\text{Apparent porosity (\%)} = \left(\frac{M-D}{M-S} \right) \times 100 \quad (2)$$

$$\text{Bulk density } \left(\frac{g}{cm^3} \right) = \left(\frac{D}{M-S} \right) \quad (3)$$

$$\text{True density } \left(\frac{g}{cm^3} \right) = \left(\frac{D}{D-S} \right) \quad (4)$$

$$\text{Total porosity (\%)} = \left(1 - \frac{\text{bulk density}}{\text{true density}} \right) \times 100 \quad (5)$$

3.2 Microstructural and elemental composition analysis

Both fracture and polished surfaces of the set hybrid geopolymer foam samples have been analyzed. The samples have been coated with gold by Ion Sputtering (E-1010, UK, Iran Polymer and Petrochemical Institute/ Iran). Microstructural analysis has been performed with scanning electron microscopy, SEM (TESCAN, Chic, Iran Polymer and Petrochemical Institute/ Iran).

The SEM has been utilized to examine the features of the microstructure of the hybrid geopolymer foam; these features parallel to their long axes utilizing a Micro Computer Controlled Electronic Universal testing machine which a maximum load

of 50 KN (Time Group, INC, China, College of Materials Engineering include the shapes and sizes of the pores, micro-cracks, and crystal presence. The analysis has been performed in the presence of crystals. An Energy Dispersive X-ray (EDX) analysis, which has been equipped with SEM, has been utilized to define the chemical composition of the resulting products and reported in terms of weight percentage of each element and oxide compositions.

3.3 Mechanical Testing

The mechanical characteristics of the hybrid geopolymer foam samples have been defined by compression testing. The specimens utilized for the compression test must be cylindrical, 21 mm in diameter, and 42 mm high have been smoothed utilizing a polishing machine to ensure that the samples are levelled. Specimens have been compressed at 0.2mm/min along their longitudinal axis until failure is achieved. The compressive intensity has been calculated utilizing the full load. The compressive strength is a calculation of how much force this could endure per unit area.

All samples have been tested dry after 28 days. Before testing, the specimen's geometrical measurement has been accounted for. The hybrid geopolymer foam cylindrical samples have been loaded /the University of Babylon).

4. Results and Discussion

4.1 SEM-EDX Results

In the case of (GPF3), the SEM images and EDX results are shown in Figures 5 and 6 below. Generally, the microstructure of (GPF3) consists of more or less loosely packed GPs particles with high porosity and some grains of unreached material having granulated structure. K-ions have made GPs denser and more homogeneous when substituted with Na-ions at Si/Al=1.85. This is the most likely result of a greater degree of reaction of metakaolin and a higher degree of aluminum incorporation; since K⁺ is more basic, it allows a higher rate of solubilized polymeric ionization and dissolution, leading to a dense polycondensation reaction that provides greater overall network formation and an increase in the compressive strength of the matrix [19, 31]. Higher Si/Al ratio has relatively homogeneous structures. Figure 5 also shows that GPFs with a high Si/Al ratio have a smaller number of quite large pores when compared to samples with a lower Si/Al ratio. However, porosity in GPs is poorly understood because of different complications with experimental characterization of pores size distribution [32].

4.2 Parametric Analysis of Compressive Strength

Compressive strength is a significant property in geopolymer foam mechanical performance evaluation because of its vital influence on engineering applications. Table 4 illustrates the processing parameters' values to obtain the optimum compressive strength according to the trial experiments and previous studies [4]. The components of (HGP₃) are identical with the components of the (GP₃) sample with the addition of (20 wt%) OPC. The compressive strength of the batch of the geopolymer paste (GP₃) sample at 28 days was (112 MPa). This value is too high as compared with the values reported for the metakaolin-based geopolymer.

4.3 Physical Tests Results

Table 5 below shows the density, porosity, water absorption, and setting time of geopolymer paste (GP₃) samples. The results indicate that batch GP₃ has a high-density value and high porosity. This indicates that the presence of the K-ion in the geopolymer increases the true density of the geopolymer. This result explains the reason behind the result of the compressive strength. These results are agreed with the previous studies [33]. Also, adding Portland cement plays a significant role in increasing the density and decreasing the porosity and water absorption of geopolymer paste. It was found that the inclusion of OPC in geopolymer mixes reduced the workability and setting time but significantly increased the compressive strength due to the quick reaction of OPC with alkali activators. The calcium sources were found to play an important role to shorten the setting time, which agrees with previous studies [34].

Table 1: The values of the processing parameters of GP3

Sample code	SiO ₂	H ₂ O/ 10.73MK-750 (ml)	(SS+SP)/(SH+PH)	Mixing time
GP ₃	3.8	9	3.04	10

Table 2: The Results of Physical tests of GP3

Sample code	Density (g/cm ³)	Porosity (%)	Water Absorption (%)	IST (min.)	FST (min.)
GP ₃	1.6	11.2	7.16	105	205

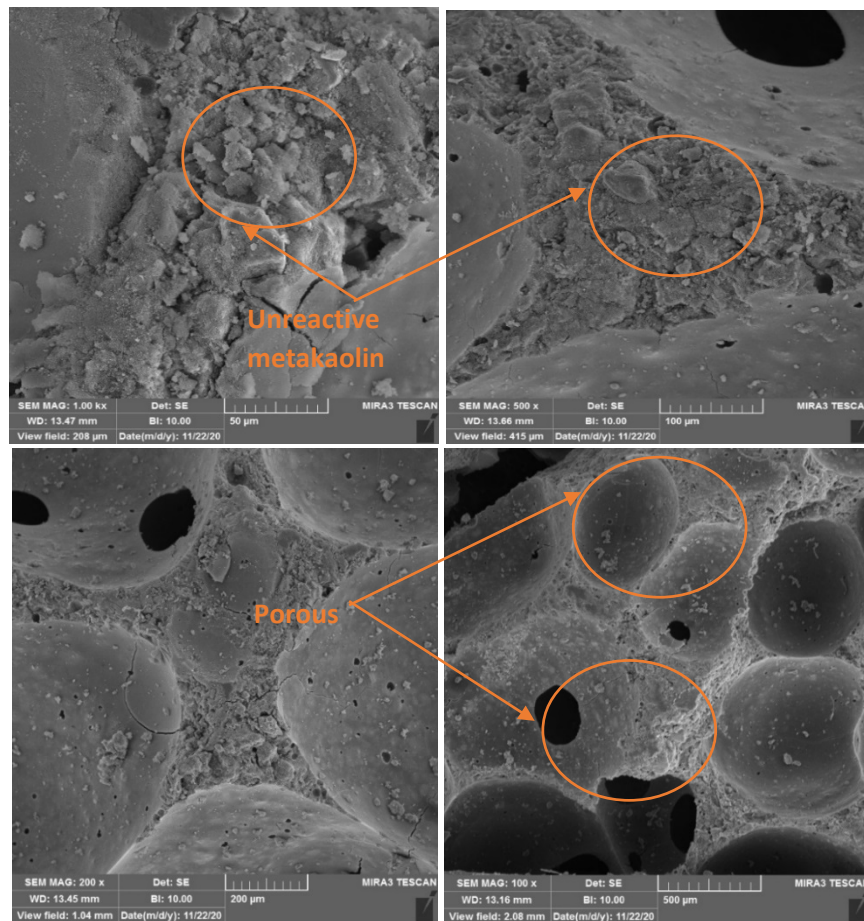


Figure 5: Surface SEM micrographs of GPF₃ sample at different magnifications

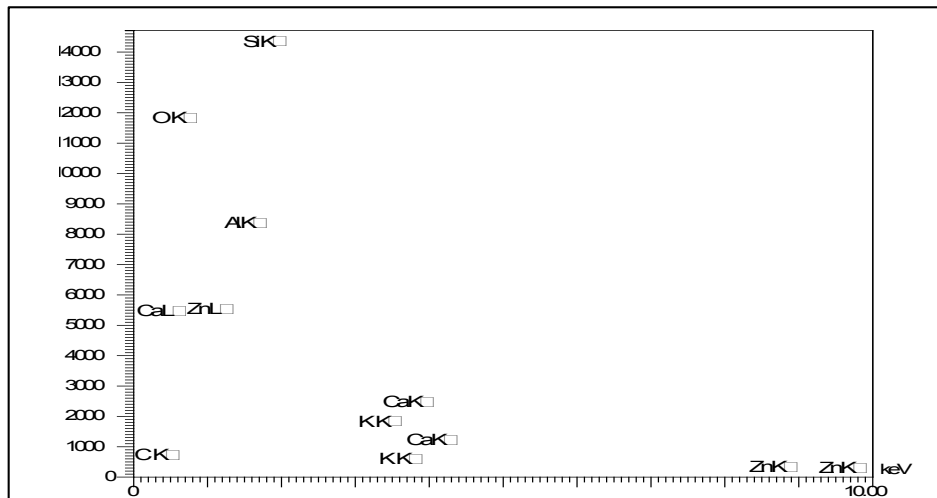


Figure 6: EDX Spectrum of GPF₃ sample with Si/Al= 1.85

5. Conclusions

During the execution of this research, it was concluded that some facts that may be useful to be mentioned and the most important are:

1. In general, the K-ions substitution of Na-ions improves the compressive strength and increases the porosity of the produced foam.
2. Regulating the content of silica, water, OPC, H₂O₂, and olive oil is necessary to produce hybrid geopolymer foam with the highest compressive strength and highest porosity and tailor the characteristics of the pore such as the amount and the size of the pores.

Author contribution

All authors contributed equally to this work.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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