RSM Optimizing the Characteristics of Metakaolin based Geopolymer Foam

Mohammed S. Radhi *1, Ahmed M. H. Al-Ghaban2 & Imad A. D. Al-Hydary3

1 University of Technology, Materials Engineering Department, Baghdad, Iraq.
2 University of Technology, Materials Engineering Department, Baghdad, Iraq.
3 Department of Ceramics Engineering and Building Materials, Faculty of Materials Engineering, University of Babylon, Babylon, Iraq.

* Corresponding author: eng.mohammedsattar@gmail.com

Abstract. The present study reports on the development of cost-effective hybrid geopolymer foam from metakaolin via a direct foaming mechanism to produce pores differ in amount and size to be suitable for wide range of applications such as filtration, catalyst support, acoustic and thermal insulation. The main goal of this research project has been to investigate the possibility of producing geopolymer foam with high compressive strength and high porosity at the same time. The complexity of the structure, the variety of the process parameters, and the impure nature of the starting materials make it hard to optimize the preparation process of HGPF and to evaluate the influence of each processing parameter on the final conducted characteristics. Response Surface Methodology (RSM) has been utilized to examine the influence of the main process parameters, on the compressive strength, and the physical characteristics of the samples with final aim to optimize the preparation process. The study involved investigation of batch (0.2 of K2O). The studied parameters were (silica content, water amount, OPC%, H2O2 %, and Olive oil %).

1. Introduction
Adsorption at different interfaces has piqued scientists' interest since the turn of the century, prompting them to research the process. Adsorption's significance in terms of technology, climate, and biology should never be overstated. It has a lot of realistic uses in manufacturing and environmental conservation [1]. This process is utilized, for instance, in water purification to eliminate any harmful components or toxins so that the filtered water is healthy for other applications, such as drinking. Adsorbents are divided into two categories: inorganic and organic compounds. Inorganic materials include zeolite, silica, and alumina, while organic materials include petroleum, polymers, and biomass. [2].

Several researchers and engineers are interested in geopolymers these days because they are simple to produce, affordable, and environmentally safe. Geopolymers are modern substance forms at the intersection of glass and products with traditional inorganic bonds (linkage). Geopolymers can prepare inorganic bonds and construction materials from waste such as kaolin, fly ash, and slag substances, among others. Since geopolymers are formulated, a high-burning temp method is not required throughout the processing of lime or cement. [3]. The manufacture of construction materials manufactured from geopolymers may not seem to affect the atmosphere in terms of Carbon dioxide
pollution; that is why these materials provide the prospect of manufacturing building materials without Carbon dioxide emissions. [4].

Davidovits invented the term geopolymer in 1978 [5] to identify a class of mineral-based materials with a chemical structure close to zeolite and a blended microstructure (from amorphous to semi-crystalline) [6]. Geopolymers are three-dimensional aluminosilicate binder compounds that are amorphous [7]. They are inorganic binders made from aluminosilicate interacting with alkaline media to form inorganic polymers. The prefix "geo" has been chosen to reflect the binders' constitutional connection to geological resources like minerals and natural stone [8].

By partially replacing the sintering method with alkaline activation and consolidation, geopolymer-based porous ceramics could be made at lower temperatures than traditional ceramic sintering [9]. Geopolymers (GP) are brittle, strong products with characteristics similar to conventional engineering ceramics and cementitious materials. Their superior mechanical characteristics to Portland cement more interest in large-scale production, although their capacity to process at ambient temperatures enables applications that conventional fired ceramics cannot [5]. Geopolymer products have strong early power, a longer lifespan, and virtually no alkali-aggregate interaction. The alkali amount of the triggering solution influences the microstructure production of geopolymers [10]. In a strongly alkaline medium, the alumina (Al2O3) and silica (SiO2) substances produced from raw materials reaction, organizing themselves in a continuous 3D framework by exchanging oxygen atoms, creating bonds including Si-O-Al-O-Si-O-Si-O, or Si-O-Al-O, Si-O-Al-O-Si-O [6].

The geopolymers' mechanical characteristics are linked closely to density so that low-density geopolymers will demonstrate unacceptably low strength [11]. Duxson et al. suggest a tendency towards enhanced mechanical intensity at higher SiO2/Al3O2 proportions and increased porosity at greater H2O/SiO2 proportions [12]. 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 light-weight materials through entraining air throughout blending[11].

A direct foaming technique has been utilized to manufacture highly porous open-cell structures from geopolymer byproducts in the availability of suitable surfactants to enhance geopolymer products' characteristics, including their low flexural strength and brittle behavior that typically restrict their comprehensive uses as a structural material. Porosity is one of the most significant microstructural variables for geopolymers, but there has been no success in monitoring or tailoring it. If geopolymer replaces current cementitious products, reducing porosity is critical for mechanical reasons, whereas growing geopolymer porosity opens up new products, including thermal insulation and filters. Although some advancement [13] toward porosity regulation has been also produced by changing different synthesis variables, major feature improvements have remained elusive, necessitating more study. Chemical additives can be useful for geopolymers as well, as shown by the significant changes in Portland cement characteristics obtained by simply using different organic materials, including high-water reductions ranges.

Given that Iraq has a sufficient supply of kaolin and has undertaken many studies into turning kaolin in great purity metakaolin, the nation continues to import large quantities of porous media for a range of uses. The present investigation supposed an innovative and cheap way of synthesizing geopolymer foam from Iraqi kaolin, which is the direct foaming technique. This study's success will bring more value to Iraqi kaolin and open a window of industrializing the synthesized metakaolin-based geopolymer foam for many applications.

Response surface methodology (RSM) is a set of statistical and mathematical tools for developing empirical models. The aim of attentive experiment design is to improve an output variable (response) that is impacted by a number of independent factors (input variables). An experiment is a sequence of tests, known as runs, in which input variables are changed to identify the causes of changes in the output response. The design of experiments [14], sometimes abbreviated as DoE, is a key part of RSM. These methodologies were created to fit models for physical investigations, but they may also
be utilized for numerical experiments. The goal of DoE is to identify the moments at which the response should be assessed, [15]. Response surface methodology (RSM) is a type of advanced experimental design technique. Using RSM can enhance the preparation strategy of a hybrid geopolymer foam, which has broken the constraint of one-dimensional linear model. The interaction and the influence of quadratic term are introduced to the model, so the test accuracy is improved, which ensures the scientific nature of the research. Moreover, multi-response analysis method is utilized in the analysis of each component's influence on strength and porosity.

2. Experimental Work

2.1. Materials

Kaolin, sodium hydroxide, sodium silicate, potassium hydroxide, silica gel, hydrogen peroxide, and ordinary Portland cement were 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.

| 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            |

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 are shown in Table (5).

2.2. Preparation of Hybrid Geopolymer Foams (HGPFS)

The metakaolin utilized in this investigation has been made by calcining kaolin clay from the surrounding region (Dwaikha, Western Iraqi Desert). The kaolin has been calcined in atmospheric air at 750°C for three hours at a heating rate of 5°C/min. The symbol (MK-750) in the work will be utilized to identify the metakaolin synthesized in this investigation.

The HGPFS have been produced in the following way. Initially, the metakaolin (MK-750) and (0, 5, 10, 15, and 20 wt%) ordinary Portland cement (OPC) were mixed. Hybrid geopolymer paste will be formed as a result of mixing the powders with the cold alkaline solution. After allowing the paste to set for a bit, hydrogen peroxide (H2O2) has been introduced as a foaming agent, followed by olive oil as a stabilizing agent. The mixture has been blended for approximately 2 minutes before it became a homogeneous slurry. After that, the slurry has been poured into a cylindrical-shaped plastic mold with a diameter of 21 mm and a height of 42 mm, and cured at room temp. Eventually, the remaining olive oil has been extracted by soaking it in hot water approximately (80 degree Celsius) for 30 minutes and then repeating the process until it has been clear.

The governate equation to design the geopolymer formulations according to the composition of the prepared geopolymer is equation (1), as describe below:

\[
m \text{K}_2\text{O}. (1-m) \text{Na}_2\text{O}. \text{Al}_2\text{O}_3. n \text{SiO}_2. x \text{H}_2\text{O}
\]

Whereas: (m) is the number of moles of K2O in the geopolymer formula and (n) is the number of moles of SiO2 in the geopolymers' formula.
This work investigates the characteristics of the hybrid geopolymer foam batch that has (m) equals to (0.2), the mix of geopolymer has been selected as indicated in the Table (2).

Table 2. shows batch of hybrid geopolymer foam.

| Symbols | Mix formula |
|---------|-------------|
| HGPF2   | 0.2 K₂O. 0.8 Na₂O. Al₂O₃. n SiO₂. w H₂O |

For the above mix formula, the influences of (n) and (w) were studied in addition to the OPC percent (C%), the foaming agent amount (F%), and the stabilizing agent content (S%) using the Response Surface method to design the experiments.

These five parameters have been chosen depending on prior research and the findings of various trials, including those that have a significant impact on the production of hybrid geopolymer foam. The upper and lower bounds of each parameter have been chosen depending on past research and several primary experiments. The following criteria were utilized to identify the values of these limits: the geopolymer paste should be easy to mix, the setting time should not be too short or too long, the volume expansion is adequate, the wet foam is stable and does not collapse, the pores distribution is uniform along the sample, and the resultant porous geopolymer body should be free of macro-cracks. The magnitudes of the parameters are shown in Table 3.

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

| N     | H₂O (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                      |

2.3. Experiments Design

The experiments design using the full factorial method. The above set of process parameters requires a large number of samples, up to 3125 samples (LP= 55), and the efforts, time, and high cost. Response Surface Methodology (RSM) has been utilized to overcome these drawbacks. RSM suggested 52 experiments for geopolymer mix (HGPF2). The conditions of each experiment are shown in Table 4.

Table 4. The conditions of the experiments produced by RSM.

| NO. | N   | W  | C  | F  | S  |
|-----|-----|----|----|----|----|
| 1.  | 3.2 | 10 | 10 | 2  | 2  |
| 2.  | 3.4 | 9  | 5  | 1  | 1  |
| 3.  | 3.4 | 9  | 5  | 1  | 3  |
| 4.  | 3.4 | 9  | 5  | 3  | 1  |
| 5.  | 3.4 | 9  | 5  | 3  | 3  |
| 6.  | 3.4 | 9  | 15 | 1  | 1  |
| 7.  | 3.4 | 9  | 15 | 1  | 3  |
| 8.  | 3.4 | 9  | 15 | 3  | 1  |
| 9.  | 3.4 | 9  | 15 | 3  | 3  |
| 10. | 3.4 | 11 | 5  | 1  | 1  |
| 11. | 3.4 | 11 | 5  | 1  | 3  |
| 12. | 3.4 | 11 | 5  | 3  | 1  |
| 13. | 3.4 | 11 | 5  | 3  | 3  |
| 14. | 3.4 | 11 | 15 | 1  | 1  |
| 15. | 3.4 | 11 | 15 | 1  | 3  |
The glycerol produced by the saponification has been extracted with hot water prior to classification, swapping it every 30 minutes until it stayed clear, demonstrating wholly extracted. Because non-fully condensed geopolymer materials were also sensitive to water and swelling or destruction, this stage of glycerol extracted could also be utilized to verify the extent of the polymerization reactions.

2.4. Physical analysis
Water absorption, apparent porosity, bulk density, apparent specific gravity (or true density), and total porosity of the hybrid geopolymer foam were 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 degree Celsius for 6 h, cooled in desiccators to the normal temperature of a room. The dry mass, D, has been identified using 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 using 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 were located in a distilled water pan and allowed to soak for an additional 24 h. then the mass identifies of 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 any 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 using the four-digit high precision density tester GP-120S (Matsu Haku, Korea, College of Materials Engineering/University of Babylon):

\[
\text{Water absorption (\%) } = \left( \frac{M - D}{D} \right) \times 100
\]

\[
\text{Apparent porosity (\%) } = \left( \frac{M - D}{M - S} \right) \times 100
\]

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

\[
\text{True density (g/cm}^3\text{) } = \left( \frac{D}{D - S} \right)
\]

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

2.5. Mechanical Testing

The mechanical characteristics of the hybrid geopolymer foam samples were defined by compression testing according to (ASTM C-109). The specimens utilized for the compression test must be cylindrical, 21 mm in diameter, and 42 mm high were smoothed using a polishing machine to make sure that the samples are leveled. Specimens have been compressed at 0.2mm/min along their longitudinal axis until failure achieved. The compressive intensity has been calculated using the full load. The compressive strength of a substance is a calculation of how much force this could endure per unit area.

All samples were tested dry after 28 days. Before testing, the specimen's geometrical measurement the have been accounted. For compression testing, the hybrid geopolymer foam cylindrical samples were loaded (the University of Babylon).

3. Findings and discussion

3.1. Chemical Analysis Findings

The findings of the kaolin's wet chemical analysis are shown in Table 5. Because of the inertness of quartz, which helps make quartz an inert constituent during the synthesis of geopolymer, the quantity of SiO2 in the clay is greater than the stoichiometric quantity in the kaolinite, which is (48.77\%). This aids in determining the quantity of free quartz, roughly, that should be removed when the geopolymer composition is estimated.

| SiO₂  | Al₂O₃ | Na₂O | K₂O | CaO | MgO | Fe₂O₃ | TiO₂ | SO₃ | P₂O | 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 |
3.2 RSM for Data Analysis

RSM has been utilized to design and carry out the experiments. The building of response surface models and the selection of acceptable models were carried out using Minitab statistical software, version 17. For the response parameters, compressive strength, apparent porosity, water absorption, total porosity, bulk density, and true density, regression equations for the selected model were generated. These regression equations have been created using experimental data and shown to see how process factors affected specific response qualities (outputs). To statistically examine the findings, the ANOVA has been utilized.

3.2. Determining the Regression Model’s Degree

The first step is to identify the degree of regression model, which is done by comparing R2 and R2adj. values for linear, linear plus square, linear plus interaction and full quadratic, Table 6 of full quadratic model for (HGPF2) is utilized to summarize in table 6. Table 6, of the full quadratic model summarizes them for (HGPF2). The full quadratic model is found to be the best for all responses; for example, R2 = 95.88 % for compressive strength of the (HGPF2) implies that 95.88 % of all fluctuations in the output parameter are explained by factors or predictors in the model. Furthermore, R2adj. = 92.95 %, which is the number of factors in the model utilized to define the relationship's significance. As a result, the full quadratic must be considered for further analysis in this study.

Table 6. R2, R2adj., and R2pred test for responses full quadratic regression model of HGPF2.

| Response characteristic | R2 (%) | R2adj (%) | R2pred (%) |
|-------------------------|--------|-----------|------------|
| Compressive strength    | 95.88  | 92.95     | 82.49      |
| Apparent porosity       | 98.69  | 97.75     | 93.74      |
| Water absorption        | 98.22  | 96.95     | 93.39      |
| Total porosity          | 96.85  | 94.61     | 85.83      |
| Bulk density            | 98.35  | 97.18     | 92.94      |
| True density            | 95.69  | 92.64     | 82.37      |

Depending on the fitted (predicted) regression equation for each response characteristic (compressive strength, apparent porosity, water absorption, total porosity, bulk density, and true density) as a function of five processing parameters has been improved by experimental data as shown below:

C. S.2 (MPa) = 31.8 - 10.28 N + 2.133 W + 0.189 C - 0.621 F - 1.155 S + 1.373 NxN - 0.1485 WxW - 0.00098 CxC + 0.0227 FxF + 0.1288 SxS + 0.164 NxW - 0.0199 CxC + 0.1675 FxF + 0.077 NxS - 0.00379 WxC + 0.0099 WxF + 0.0132 WxS + 0.00046 CxF - 0.0082 CxS - 0.0037 FxF

A. P.2 (%) = 64.36 - 4.20 N + 3.412 W - 1.620 C + 7.814 F + 6.722 S + 2.842 NxN + 0.0925 WxW + 0.005700 CxC - 0.0975 FxF - 0.0120 SxS - 1.377 NxW + 0.3934 NxS - 1.938 NxF - 1.738 NxS + 0.00057 WxC - 0.0011 WxF - 0.0010 CxF - 0.00019 CxS - 0.0014 FxF

W. A.2 (%) = 107.8 - 16.87 N + 1.54 W - 0.823 C + 7.125 F + 0.097 S + 4.166 NxN + 0.1474 WxW + 0.00963 CxC + 0.0310 F + 0.0962 SxS - 1.207 NxW + 0.1541 NxS - 2.059 NxF - 0.115 NxS - 0.00319 WxC + 0.0801 WxF + 0.0583 WxS - 0.02236 CxF + 0.00512 CxS + 0.0239 FxF

T. P.2 (%) = 62.4 - 6.39 N + 2.842 W - 1.034 C + 5.456 F + 3.369 S + 2.223 NxN + 0.0211 WxW + 0.00321 CxC + 0.0305 FxF + 0.1625 SxS - 0.792 NxW + 0.2400 NxS - 1.365 NxF - 0.911 NxS + 0.00280 WxC - 0.0229 WxF - 0.0308 WxS + 0.00045 CxF - 0.00399 CxS + 0.0013 FxF

B. D.2 (g/cm³) = 0.1756 + 0.0708 N - 0.0088 W + 0.001052 C - 0.00500 F - 0.00355 S - 0.01137 NxN - 0.000005 WxW - 0.000025 CxC + 0.000408 FxF + 0.000180 SxS + 0.001834 NxW - 0.000079 NxC
The gained strength for HGPF2 obtained when the content of silica (3.4) with (11ml) water, the weight percent of OPC, H2O2, and increasing the silica content and decreasing by increasing the water. The above-mentioned models may be utilized as effective tools for exploring the design space inside the process parameters domain to get a thorough grasp of process features, as well as in the optimization stage to identify the best mix design for direct foaming conditions on metakaolin-based geopolymer.

3.3. Parametric analysis of compressive strength

Figures (1-3) show the main influence, response surface, and contour plots, respectively, of each variable on the compressive strength for the batch (HGPF2). Figure (1) demonstrates the main influence plot for the compressive strength of HGPF2-batch. In general, the strength increases by increasing the silica content and decreasing by increasing the water. The Run Order No. 38, has provided the highest achieved 28-day compressive strength has been about (32.02 MPa), which is obtained when the content of silica (3.4) with (11ml) water, the weight percent of OPC, H2O2, and olive oil were 15, 3, and 3wt%. As for the concentration of (SS+PS)/(SH+PH) has been fixed at 3.04. The gained strength for HGPF2-batch is greater than that of samples which are free of potassium [16]. This suggests that when sodium ions are partly replaced by potassium ions, the strength increases.

Smaller Na+ ions make coupling with the anion of silicate easier, resulting in smaller oligomers. While the bigger ions of K+ combine with the anion of silicate to form bigger oligomers. As a result, when comparing geopolymers depending on K+ to geopolymers depending on Na, geopolymers depending on K+ have a 25% higher compression strength. [17]. As well as, larger ions of K+ help to accelerate the geopolymers setting [18]. The findings contradict those of Liew Yun-Ming et al., who reported that when various kinds of ions are utilized, compressive strength diminishes. [19].

$$+ 0.000266 \text{NxF} - 0.000884 \text{NxS} + 0.000022 \text{WxC} - 0.000022 \text{WxF} + 0.000318 \text{WxS} - 0.000009 \text{CxF} + 0.000016 \text{CxS} + 0.000134 \text{FxS}$$

(11)

T.D.2 (g/cm³) = 5.318 - 1.473 N - 0.1324 W + 0.03748 C + 0.0390 F + 0.0957 S + 0.2081 NxN - 0.00025 WxW - 0.000187 CxC + 0.00301 FxF - 0.00038 SxS + 0.0348 NxW − 0.00951 NxC - 0.0388 NxF - 0.0373 NxS – 0.000063 WxC + 0.00269 WxF - 0.00294 WxS + 0.000924 CxF + 0.001766 CxS + 0.01387 FxS

(12)

Figure 1. Main Influence Plot for compressive strength of HGPF2.

Figure 2. Response Surface Plot of compressive strength for HGPF2.

Figure 3. Contour Plot of compressive strength for HGPF2.
3.4. Parametric analysis of physical characteristics

Figures (4-6), show the main influence, response surface, and contour plots of the apparent porosity for (HGPF2). Figures (7-9) show the main influence, response surface, and contour plots of the water absorption for (HGPF2).

Water content has been shown to be a key determinant in porosity and, hence, water absorption, as predicted. This is related to the creation of pores as a result of:
1. The excess water that is normally added to improve the paste's workability is removed.
2. The condensation polymerization produces water, which must be removed.

Furthermore, the most effective parameter on the physical characteristics is the foaming agent (H2O2), then the stabilizing agent, and at last OPC.

The bulk density of Na-geopolymer is affected by silica content more than that of potassium containing geopolymer. The bulk density of Na-based geopolymer is greater than that of K-containing geopolymer, according to this study. Lizcano, et.al. have been reported contrary finding in previous study [20].

The clear description for the physical characteristics for the samples that are included the foaming and stabilizing agents is as following:
Similar to the compressive strength, with the addition of hydrogen peroxide and olive oil, both bulk density and true density of the samples tended to decrease, but apparent porosity, water absorption, and total porosity tended to increase. However, owing to the creation of closed pores in the samples, there has been a fluctuation in bulk density and water absorption values. Total porosity has been shown to be connected to the trend of compressive strength value. The addition of OPC, on the other hand, had the opposite impact on the physical characteristics.

In general, the release of the trapped O2 from H2O2 in the sample, where H2O2 may decompose to H2O and O2, induced these physical property alterations. Increasing H2O2 levels, on the other hand, aids in the reduction of pore size with a narrow pore size distribution. Increasing the quantity of olive oil causes the samples to have a greater porosity surface. This is because olive oil may serve as a surfactant, preventing the gas bubbles from merging as they did in the non-olive oil sample.

To evaluate the influence of adding oil on the microstructure of the samples, we must examine the following factors: (i) the composition of the oil, (ii) the viscosity changes induced by the oils, and (iii) the emulsion stability. In fact, the oil forms several types of soap molecules (surfactants), and the degree of foaming is affected by the viscosity of the combination (while the rest of the processing parameters, such as mixing rate and amount of H2O2, are kept constant). Because the time scale for the process is often shorter than that for flocculation and coalescence in an oil/water emulsion, this complicated influence has a limited impact on emulsion stability.

The viscosity of the system has been greatly enhanced when oil has been added. It's also worth noting that the manufacturing of soap and glycerol might have an impact on the measurement. The use of olive oil resulted in small cells with a high total porosity, indicating that soap molecules generated in-situ during processing were more effective in stabilizing the liquid/air boundary in the foams. The small cells size of the samples account for the olive oil's limited influence on compression strength, despite the significant quantity of total porosity.

The rise in viscosity due by the evolution of geopolymerization in the mixes over time increased as the olive oil content increased. This is due to the consumption of OH− by acids in the oil, which rises as the oil volume fraction rises, reducing the geopolymerization process kinetics. The viscosity of the slurry increased somewhat when additional oil has been added, but not much, therefore this is not a sufficient basis to explain the observed trend [16].

Normally, the saponification process, which consists of the hydrolysis of triglycerides (found in oil or fats), plus glyceride, and glycerol (a water-soluble molecule), creates in situ carboxylate surfactants (soap molecules) when oil and a strong alkaline solution are reacted (a water-soluble molecule). This may result in the production of closed and interconnected pores in the samples, or the saponification of glycerol to build micro- and meso-pores.
The addition of H2O2 to the (HGP) samples resulted in mostly closed pores, but the combination of H2O2 and olive oil (at higher contents) resulted in the creation of interconnected pores in a matrix of samples [21].

**Figure 4.** Main Influence Plot for apparent porosity of HGPF2.

**Figure 5.** Response Surface Plot of apparent porosity for HGPF2.

**Figure 6.** Contour Plot of apparent porosity for HGPF2.

**Figure 7.** Main Influence Plot for water absorption of HGPF2.

**Figure 8.** Response Surface Plot of water absorption for HGPF2.

**Figure 9.** Contour Plot of water absorption for HGPF2.
3.5. On HGPF2, Parametric optimization of the foaming process

Depending on the developed quadratic mathematical responses (Equation 7-12), d7, d8, d9, d10, d11, and d12 are selected as the independent desirability functions for the C.S.2, A.P.2, W.A.2, T.P.2, B.D.2, and T.D.2, respectively, for HGPF2-batch. Moreover, the targets are placed on the C.S and porosity to be maximized. Table 7 illustrates optimum settings obtained of the processing parameters required to obtain the optimum outputs according to RSM analysis. Table 8 demonstrates the optimum output findings of the (HGPF-2) samples that were prepared using optimum processing parameters. The finding reveals that the suggested optimum mixes achieved compressive strength higher than that obtained from the experimental works, this confirms that RSM method has the ability to suggest the optimum conditions to get optimum findings.

Table 7. Optimum setting of the processing parameters to get optimum findings by RSM.

| Optimum setting | HGPF2-batch |
|-----------------|-------------|
| N               | 3.2         |
| W               | 12          |
| C               | 8.51        |
| F               | 3.84        |
| S               | 4           |

Table 8. The optimum output findings of the (HGPFs).

| Optimum output | HGPF2-batch |
|---------------|-------------|
| Compressive strength (MPa) | 32.00 |
| Apparent porosity (%) | 87.75 |
| Water absorption (%) | 96.67 |
| Total porosity (%) | 76.11 |
| Bulk density (g/cm³) | 0.24 |
| True density (g/cm³) | 2.39 |

Confirmation experiments are the most important, last, and necessary aspect of any optimization effort. The actual compressive strength (C.S.), apparent porosity (A.P.), water absorption (W.A.), total porosity (T.P.), bulk density (B.D.), and true density (T.D.) were compared to those as ideal responses obtained by the desirability technique in a verification experiment. The optimization findings, as well as experimentally observed responses and their percentage of relative verification errors, are summarized in Table (9). It's worth noting that the errors were computed as [22]:

\[
\text{Prediction error} \% = \left[\frac{\text{Experiment result} - \text{Predicted result}}{\text{Experiment result}}\right] \times 100
\]  

(13)

Being can be seen, the errors values are acceptable for engineering purposes, with -4.34 percent as the worst case in estimating the bulk density of the material (HGPF2- batch). Predictability ensures the method's practicality and efficiency.

Table 9. Experimental confirmation of multiple-response optimum points.

| HGPF2-Batch | Optimum input setting | C.S. | A.P. | W.A. | T.P. | B.D. | T.D. |
|------------|-----------------------|------|------|------|------|------|------|
| Responses  | Predicted             | 32.00| 87.75| 96.67| 76.11| 0.24 | 2.39 |
| Experiment |                      | 31.87| 87.88| 95.89| 76.92| 0.23 | 2.38 |
| Relative error (%) |                | -0.4 | 0.14 | -0.81| 1.05 | -4.34| -0.42|
4. Conclusion
During the process of this investigation, it has been discovered that there are a few facts worth mentioning, the most noteworthy of which are:

- Response Surface Methodology is a useful technique to experiments design and optimize the process of hybrid geopolymer foam synthesis.
- In general, the K-ions substitution of Na-ions improves the compressive strength and increases the porosity of the produced foam.
- Regulating the content of silica, water, OPC, H2O2, and olive oil is necessary to produce hybrid geopolymer foam with highest compressive strength and highest porosity, as well as to tailor the pores characteristics such as the amount and the pores size.
- The direct foaming combined method is suitable strategy to produce hybrid geopolymer foams having pores within micro and macro sizes and this feature makes the produced foams are suitable for many applications.

References
[1] Duxson P, Fernández-Jiménez A, Provis J L, Lukey G C, Palomo A and van Deventer J S J 2007 Geopolymer technology: the current state of the art J Mater Sci 42 2917–33
[2] Knaebel K S 2011 Adsorbent selection Accessed on 6
[3] LOPEZ G F J 2014 Study of Geopolymer Adsorbents Prepared from Metakaolin and Rice Husk Silica for Targeting to Heavy Metal Capture
[4] Abdul Halim N F 2015 Development of Porous Geopolymer Materials from Meta-kaolin and Fly Ash (IRC)
[5] Davidovits J 1991 Geopolymers: inorganic polymeric new materials J Therm Anal Calorim 37 1633–56
[6] Cilla M S, Colombo P and Morelli M R 2014 Geopolymer foams by gelcasting Ceram Int 40 5723–30
[7] Prud’homme E, Michaud P, Joussein E, Peyratout C, Smith A, Arrii-Clacens S, Clacens J-M and Rossignol S 2010 Silica fume as porogent agent in geo-materials at low temperature J Eur Ceram Soc 30 1641–8
[8] Buchwald A, Hohmann M, Posern K and Brendler E 2009 The suitability of thermally activated ilite/smectite clay as raw material for geopolymer binders Appl Clay Sci 46 300–4
[9] Landi E, Medri V, Papa E, Dedecek J, Klein P, Benito P and Vaccari A 2013 Alkali-bonded ceramics with hierarchical tailored porosity Appl Clay Sci 73 56–64
[10] Dutta D, Thokchom S, Ghosh P and Ghosh S 2010 Effect of silica fume additions on porosity of fly ash geopolymers J Eng Appl Sci 5 74–9
[11] Masi G, Rickard W D A, Vickers L, Bignozzi M C and Van Riessen A 2014 A comparison between different foaming methods for the synthesis of light weight geopolymers Ceram Int 40 13891–902
[12] Okada K, Ooyama A, Isobe T, Kameshima Y, Nakajima A and MacKenzie K J D 2009 Water retention properties of porous geopolymers for use in cooling applications J Eur Ceram Soc 29 1917–23
[13] Bell J L and Kriven W M 2009 Preparation of ceramic foams from metakaolin-based geopolymer gels Ceram Eng Sci Proc vol 29 pp 97–112
[14] Box G E P and Draper N R 1987 Empirical model-building and response surfaces vol 424 (Wiley New York)
[15] Dwivedi S P and Dwivedi G 2019 Utilization of RHA in development of hybrid composite by electromagnetic stir casting technique using RSM J Met Mater Miner 29
[16] Bai C, Franchin G, Elsayed H, Conte A and Colombo P 2016 High strength metakaolin-based geopolymer foams with variable macroporous structure J Eur Ceram Soc 36 4243–9
[17] Cioffi R, Maffucci L and Santoro L 2003 Optimization of geopolymer synthesis by calcination
and polycondensation of a kaolinitic residue *Resour Conserv Recycl* **40** 27–38

[18] Phair J W and Van Deventer J S J 2001 Effect of silicate activator pH on the leaching and material characteristics of waste-based inorganic polymers *Miner Eng* **14** 289–304

[19] Liew Y M, Heah C Y, Mohd Mustafa A B and Kamarudin H 2016 Structure and properties of clay-based geopolymer cements: A review *Prog Mater Sci* **83** 595–629

[20] Lizcano M, Kim H S, Basu S and Radovic M 2012 Mechanical properties of sodium and potassium activated metakaolin-based geopolymers *J Mater Sci* **47** 2607–16

[21] Lertcumfu N, Kaewapai K, Jaita P, Tunkasiri T, Sirisootthorn S and Rujijanagul G 2020 Effects of olive oil on physical and mechanical properties of ceramic waste-based geopolymer foam *J Reinf Plast Compos* **39** 111–8

[22] Assarzadeh S and Ghoreishi M 2013 Statistical modeling and optimization of the EDM parameters on WC-6% Co composite through a hybrid response surface methodology-desirability function approach *Int J Eng Sci Technol* **5** 1279