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Investigating the Microstructure and Mechanical Properties of Metakaolin-Based Polypropylene Fiber-Reinforced Geopolymer Concrete Using Different Monomer Ratios

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ABSTRACT

Researchers have focused on the fabrication and implementation of concrete that has optimal characteristics, logical price with minimum harmful impacts on the environment. For this purpose, the current project was conducted. Geopolymer cement, due to its high durability, insignificant energy consumption, least CO_2 emissions, acceptable investment cost, and specific characteristics, is accounted as a new class of mineral binders which is different from such binders as Portland cement. In this research, the geopolymer concrete was made using metakaolin precursor containing 0.3, 0.5, and 1% polypropylene fibers together with monomer with 2, 2.5, and 3 ratios. Next, a number of engineering properties such as the bending strength, compressive strength, electrical resistivity, water absorption, and microstructure were assessed at 90 days age using the electron microscopy scanning method.

Keywords: **metakaolin, concrete geopolymer, concrete durability, scanning electron microscopy test, polypropylene fibers, monomer ratios.**

1. INTRODUCTION

Concrete has widespread use in many types of a structures. This material degrades when it is exposed to a damaging environment, also is weak in tension. Thus engineers have sought to modify these weaknesses by increasing the tensile strength and consequently bending strength without affecting other properties. Replacing a portion of cement with waste materials is among the available methods for engineers. Among these materials, one could refer to metakaolin, fly ash, blast furnace slag, limestone powder, rice husk ash, etc., which are used with cement for the production of concrete [1,2]. When a portion of cement is replaced by pozzolans, a large amount of energy is saved, which otherwise was required for cement production. Also, they help with sustainable development by reducing the emission of greenhouse gases during clinker production. Furthermore, they could increase durability characteristics of concrete such as permeability and enhance the concrete mechanical properties like compressive strength at higher ages [3, 4]. Considering that the cement replacement materials are often not much expensive due to their natural existence or being by-products, when combined with chemical additives, could further improve the savings on concrete production cost [5]. In addition, some pozzolans such as fly ash or blast furnace slag perform the role of binder by using alkaline liquids. Therefore they could fully replace the used Portland cement in concrete. However, the activator alkalinity could vary from low to medium, where the major activated contents are calcium and silicon that are resulted from materials like blast furnace slag. C-

S-H gel is the name of a binder produced by the hydration process. Other components which are activated with a high alkali solution are silicon and aluminum derived from the materials like low calcium fly ash. Here the binder is produced by the polymerization process. This product was named geopolymer in 1978, and it was stated that binders could be produced using polymer synthesis, which is the result of activation by alkalis of geological origin or products such as fly ash and rice husk ash [6]. Davidovits has shown that feldspathic and zeolitic minerals could be produced by providing the synthetic conditions for synthesis of organic plastics (organic phenolic plastics). He concluded this idea based on observing various disastrous fires and the burning of organic plastics [6]. The geopolymer structure is constituted of a network of alumino-silicate bonds. The simplest form of geopolymer is poly (sialate) or silicon-oxo-aluminate. Geopolymers are, in fact, the result of a chemical reaction between alumino-silicate oxides and polysilicates in a concentrated alkaline environment. The sialate network is comprised of SiO_4 and AlO_4 in the form of tetrahedral, where they are connected one after the other sharing all the oxygen molecules. The presence of positive ions such as NH_4^+, Ba^{++}, Ca^{++}, Li^+, K^+, Na^+, and H_2O^+ in the pores of this network, is essential to balance the negative aluminum charge in the tetrahedral form. Polymers are made of large molecules that are the combination of a large number of monomers (repeating units). This single molecular structure enables the large molecules have control over the properties of the materials. The amorphous state is the one that has a solid structure but lacks a well-defined shape. Glass is an example of an amorphous solid. Geopolymers belong to the family of inorganic polymers. They possess a chain structure based on Al and Si ions. Geopolymers are amorphous materials (non-crystalline) with a chemical composition similar to natural silicate materials. The polymerization process is a very rapid chemical reaction with SI-AL minerals occurring in a very alkaline environment. The result is a 3D polymer chain with a ring structure comprised of Si-O-AL-O bond as shown [6]:

$$M_n\,[-(SiO_2)_z - AlO_2]\ n\ .\ wH_2O$$

In the above formula, M denotes any cation, for example, calcium, sodium, or potassium, n denotes poly-condensation degree, and z takes values of 1, 2, 3-32. The materials containing silicon (Si) and Aluminum (Al) in the amorphous form have the potential of producing geopolymers. Among the source materials, one could refer to calcined kaolin or metakaolin [6-8], fly ash [9, 10], the combination of calcined minerals and non-calcined materials [11], minerals containing natural aluminum and silica [11], combination of fly ash and metakaolin [10, 12], and combination of granulated furnace slag and metakaolin.The strength of geopolymer concrete is higher than cement concrete, although the former lacks Portland cement. The other advantage of geopolymer concrete is that it has a smaller reduction in volume and nearly produces no heat. The geopolymer cement production process produces 80% lower carbon dioxide compared with the amount produced in the Portland cement production process. Geopolymer cement production needs two stages of thermal activation and alkali activation. Furthermore, the raw materials are abundant all over the world, and any alumino-silicate source soluble in a highly alkaline environment is suitable as the source for this type of cement. Among the main characteristics of geopolymer cement, one could refer to its resistance to extreme erosion, high thermal resistance, high compressive strength, and good volume stability [13]. Some advantages of geopolymer cement over Portland cement used in concrete are: 1-low cost of production due to the abundance of alumino-silicate materials in the Earth's crust or production using the wastes from other industries. 2-low energy consumption because it hardens at very lower temperatures. 3-Some environmental advantages: a)-some wastes could be converted into usable materials through geo-polymerization. b) Co_2 greenhouse emission is reduced in the process of geopolymer production. c) Geopolymer composites also have applications in the stabilization of toxic and radioactive wastes [13]. Metakaolin has the advantages of having white color, high rate of separation within the reaction solution, and easy control over the Si/Al ratio [14], but it is expensive when used in large amounts in concrete production. The alkaline activator that is mostly used in geopolymer is a combination of potassium hydroxide (KOH), sodium hydroxide (NaOH), and sodium silicate or potassium silicate [6-11]. Some studies have also suggested the application of a single alkaline activator [8, 9]. Geopolymer concrete has been investigated in terms of its mechanical and durability properties [15-23]. Shehab et al. have investigated the mechanical properties of geopolymer concrete where fly ash was used as a complete replacement or partial replacement for Portland cement [16]. In this study, a total number of 18 concrete mix designs were prepared to identify the key parameters affecting the concrete behavior and its mechanical characteristics. The studied key parameters were the cement replacement ratio, binder content, and the ratio of activating solution in a fly ash-based geopolymer concrete. The study results exhibited enhanced mechanical properties of geopolymer concrete where fly ash was used as the replacement material. In addition, it was found that in the specimen with 50% replacement, the bending strength, compressive strength, and compressive strength were higher in comparison to the specimens with 0, 25, 75, and 100% replacement ratios. Singh et al. [23] studied the impact of the activator on the strength, interfacial transition zone of rocks and binders, and shrinkage of polymer concrete with fly ash and slag replacement. They investigated the mechanical properties, age hardening, and shrinkage of geopolymer concrete with fly ash/slag replacement and different activator concentrations. The optimal compressive strength belonged to the 14M specimen. As seen in the image by scanning electron microscopy, the presence of spongy amorphous geopolymer paste on the surface of the grains provides the optimal conditions at the interface between the paste and the aggregate. As is seen by the increase of the activator amount, Poissons' ratio is decreased, and modulus of elasticity, compressive strength, and impact resistance are increased. Implementing various types of steel, glass fibers, polypropylene, and occasionally carbon, one could produce a variety of composite concretes for different applications in industry. Composite concretes have a widespread application in the construction sector of developed countries. Fiber concrete also has appropriate characteristics such as high strength and ductility, energy absorption ability, and cracking stability, making it suitable for many applications. Although many studies have been done on the properties of the geopolymer pastes or mortars with small size specimens, full details of geopolymer paste mixtures have not been investigated yet.

2. MTERIALS AND METHODS

2.1. MATERIALS

Metakaolin which contains alumino-silicate, is produced by calcination of kaolin at 700-800 °C and is used as a raw material in the composition of geopolymer. Table 1 shows the chemical composition of metakaolin. The activating chemicals include 8M sodium hydroxide solution and sodium silicate with the chemical composition of Na2O = 15.4, SiO2 = 31.7, water = 52.9% by weight. The fine grain used is river bed sand having a maximum size of 2.36mm.

Table 1. Metakaolin chemical composition

Chemical analysis (%)	MK
SiO_2	52.1
Al_2O_3	43.8
Fe_2O_3	2.6
CaO	0.2
MgO	0.21
SO_3	0
K_2O	0.32
Na_2O	0.11
L.O.I	0.99
Surface area (m^2/g)	2.54
Specific gravity	2.6

2.2. MIX RATIOS

As shown in Table 2, in this research, 9 groups of concrete mixes with different ratios were prepared. The amount of used metakaolin was 400 kg per cubic meters with sodium silicate to sodium hydroxide ratios of 2, 2.5, and 3. Then polypropylene fibers at 0.3, 0.5, and 1 wt% of metakaolin were added to the mixtures. Also, 600kg sand and 1250kg sand and gravel were used for producing 1m3 concrete. In the mix designs nomenclature, the number next to R denotes the sodium silicate to sodium hydroxide ratio, and the number next to letters PP denotes the percentage of polypropylene fibers.

Table 2. Mix designs of geopolymer specimens

Design	Metakaolin	Polypropylene Fibers	Sodium Silicate	Sodium Hydroxide	Sand	Gravel	Slump (mm)
R2PP0.3	400	1.2	90	45	600	1250	61
R2PP0.5	400	2	90	45	600	1250	60
R2PP1	400	4	90	45	600	1250	55
R2.5PP0.3	400	1.2	115	45	600	1250	62
R2.5PP0.5	400	2	115	45	600	1250	61
R2.5PP1	400	4	115	45	600	1250	56
R3PP0.3	400	1.2	135	45	600	1250	65
R3PP0.5	400	2	135	45	600	1250	62
R3PP1	400	4	135	45	600	1250	60

2.3. PREPARATION OF SAMPLES

First, the fine and coarse grains were dry mixed for about 1-2 minutes. Then metakaolin was added to the mixer and mixed for another –3 minutes. Next, the alkaline activating solution was gradually added to metakaolin to form the required gel. Full mixing of the gel is required for about 2-3 minutes so that aluminate and silicate monomers are formed. Stirring should be continued till a completely homogeneous paste is formed. Then the geopolymer concretes were placed in proper molds before becoming hardened. After 24 hours, they were removed from the molds and processed at 70 ± 10% humidity and 20 ± 2 °C temperature.

3. RESULTS AND DISCUSSION

3.1. COMPRESSIVE STRENGTH

Fig.1 shows the results of the compressive strength test for specimens with polypropylene fibers and different monomer ratios. As seen, the addition of fibers has an insignificant impact on the compressive strength of geopolymer specimens. Whereas in the specimens having the monomer ratio of 3, by the addition of fibers, more considerable changes are observed. Also, applying higher monomer ratios results in increased compressive strength. Furthermore, it is observed that in specimens with the sodium silicate to sodium hydroxide ratio equal to 2 and 1% polypropylene fibers, the compressive strength is 50.9 MPa. When the abovementioned ratio is increased to 3, and the percentage of polypropylene fibers is kept equal to 1%, the compressive strength of the specimen reaches 55.4 MPa, and indicating 10% enhancement of the compressive strength.

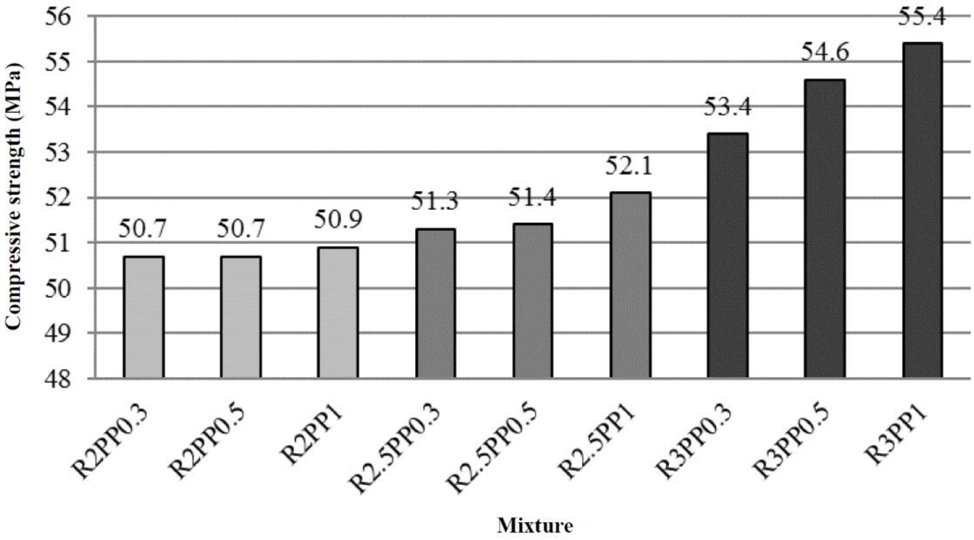

Figure 1. Compressive strength of the geopolymer concretes at 90 days age

3.2. BENDING STRENGTH

Fig.2 shows the results of the bending strength test for specimens with polypropylene fibers and different monomer ratios. It is observed from Fog.2 that polypropylene fibers have a considerable impact on the bending strength of geopolymer specimens. The specimens containing monomer ratios of 2, 2.5, 3, and 0.3% of polypropylene fibers had bending strength values of 7.03, 7.51, and 7.69, respectively. Increasing the percentage of polypropylene fibers to 1% resulted in bending strength values of 9.02, 9.36, and 9.87, indicating an increase of 28, 25, and 28%, respectively. As seen in the scanning electron microscopy image of specimen R3PP1 in Fig.3, one could conclude that polypropylene fibers can control the initiation of cracking and reduce the width and number of cracks and thus increase the bending strength. The desirable effect of fibers on the increase of the concrete bending strength can be explained as follows: Research shows that the addition of 0.9 kg/ m3 polypropylene fibers means applying one hundred million fiber strands per 1 m3 concrete. This large number of fibers within a short distance in the cement matrix prevents large cracks and propagation of them in the cement composite texture after applying the load, and only very small cracks (microscopic) would appear. These fibers resist the applied stresses and transfer them from one side of the crack to the other side, which eventually results in either detachment from the matrix, pulling out of it, or failure [24]. It should be noted that, however, increasing the sodium silicate to sodium hydroxide ratio in the specimens resulted in 10% improvement.

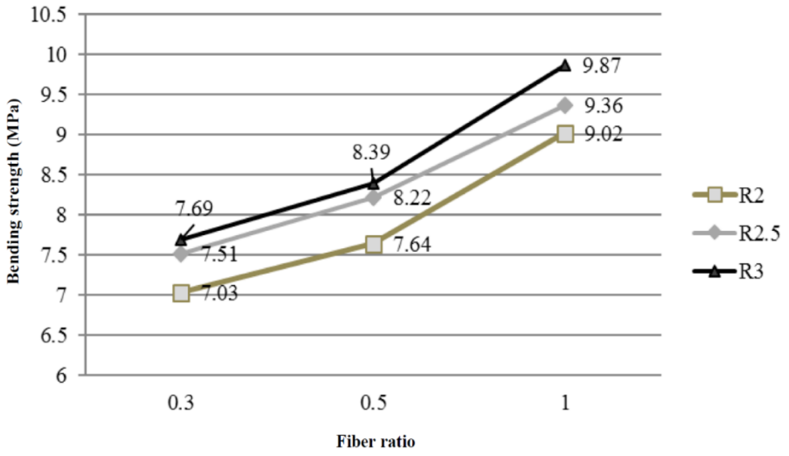

Figure 2. Bending strength of the geopolymer concrete specimens at 90 days age

Figure 3. Scanning electron microscopy image of R3PP1 specimen

3.3. WATER ABSORPTION

Fig.4 shows the amount of water absorption in the geopolymer specimens. As seen, polypropylene fibers have an insignificant impact on reducing water absorption; however, in the specimen with the monomer ratio equal to 2.5, the addition of fibers from .3 to .5, mainly due to inaccurate mix design, results in a small increase in the water absorption amount. Increasing the sodium silicate to sodium hydroxide ratio from 2 to 2.5 reduces the water absorption by 17.13 and 15%, respectively. But increasing the monomer ratio has no considerable effect on the water absorption amount. An overview of the water absorption values reveals that a monomer ratio equal to 3 in the geopolymer concrete reduced the water absorption rate. Also, specimen R3PP1 with 1% polypropylene fibers had the highest impact upon the water absorption rate in the concrete and reduced it from 4.7 to 3.87%. The water absorption rate is associated with saturated specimen porosity, and reduced water absorption could be attributed to reduced cavities in the specimens.

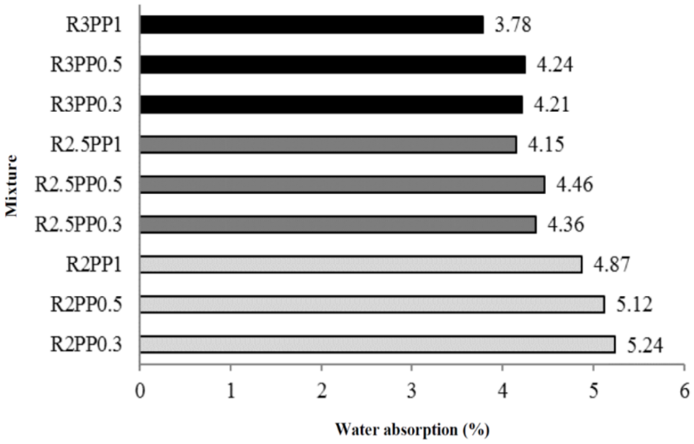

Figure 4. Water absorption of the geopolymer concretes at 90 days age

3.4. ELECTRICAL RESISTIVITY

Measurement of electrical resistivity is done simply using a special ohmmeter and by placing two copper or brass plates on the surfaces of the concrete specimen and reading the values. It should be noted that the specific electrical resistivity should be measured regardless of the specimen dimensions. In this way, a comparison of the values obtained from different specimens becomes possible. The used equation in this research is given below:

$$\rho_c = \frac{R.A}{L}$$

In the above equation, p represents the specific electrical resistivity of the specimen in ohm meter, R denotes the read value from the device, A denotes the surface area of the specimen, i.e., the contact surface of the brass plate with concrete, and L represents the specimen length, i.e., the distance between the two contact plates.Measurement of electrical resistivity in the geopolymer concretes containing polypropylene fibers was performed after 90 days. The higher resistance of the specimen to the current flow indicates a lower corrosion probability and rate. Fig.5 shows the results of the electrical resistivity test and the classification of the corresponding corrosion rates for the geopolymer concrete specimens

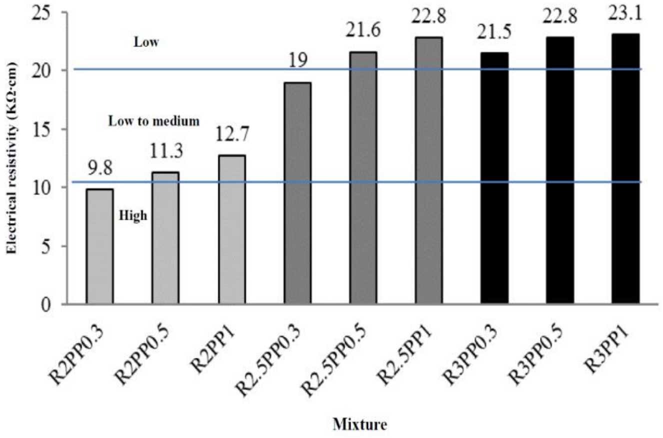

Figure 5. Electrical resistivity and corrosion rate of the geopolymer concretes at 90 days age

In the specimens with a monomer ratio of 2.5, containing 0.3, 0.5, and 1% polypropylene fibers, the electrical resistivity was 64, 91, and 80% higher with respect to the specimen having a monomer ratio of 2, respectively. In addition, the electrical resistivity of the specimens having the monomer ratio of 3 and containing 0.3, 0.5, and 1% polypropylene fibers increased by 119, 102, and 82%, respectively, with respect to the specimens with a monomer ratio equal to 2. The probability of corrosion in specimens with the monomer ratio equal to 2 ranged from low to medium. In these specimens, by the increase of the sodium silicate to sodium hydroxide ratio, they moved to the low range region. Figs. 6 and 7 show the images obtained from scanning electron microscopy for specimens R2PP1 and R3PP1, respectively. As seen in Fig.6, the cohesion disappears in the geopolymer paste resulting in reduced durability of concrete, whereas R3PP1 specimen possesses a homogeneous and uniform microstructure. It should be noted that there is very good compatibility between the results of the mechanical tests i.e. the compressive and bending strength values, with those of the scanning electron microscopy. This compatibility is even higher in the durability tests i.e. the water absorption and electrical resistivity tests. This issue could be explained in this way that improvement in the microstructure of geopolymer concrete specimens leads to enhanced mechanical and physical properties in them. It has been known that ionic concentration and porosity in the porous solution are two factors that could directly impact the electrical resistivity in cementitious materials [25]. Thus it is clear that by the increase of the sodium silicate to sodium hydroxide ratio, a significant change occurs in the specimens' strength. On the other hand, a higher monomer radio helps with reduced porosity and increased strength and causes the delay of chlorine ions penetration and increase of durability. Also increase in electrical resistivity reduces the movement of ions into the specimens and thus reduces the corrosion rate.

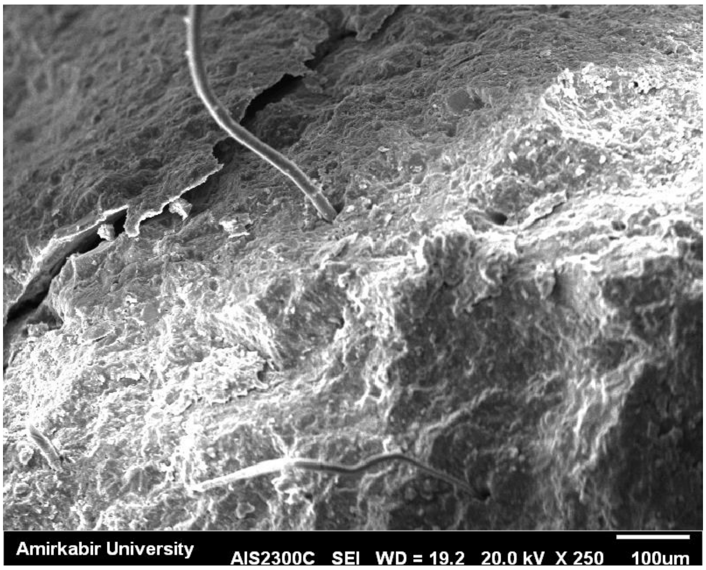

Figure 6. Scanning electron microscopy image of R2PP1 specimen

Figure 7. Scanning electron microscopy image of R3PP1 specimen

4. CONCLUSION

An increase of the sodium silicate to sodium hydroxide ratio from 2 to 3, causes improvement of the compressive strength in the specimens. The highest strength corresponded to the specimen containing 1% polypropylene fibers which showed 10% increase in the compressive strength. The specimens having monomer ratios equal to 2, 2.5, 3, and 0.3% polypropylene fibers had to bend strengths equal to 7.03, 7.51, and 7.69, respectively. In specimens with 1% polypropylene fibers, the bending strength increases to 9.02, 9.36, and 9.87, increasing 28, 25, and 28%, respectively. Increasing the sodium silicate to sodium hydroxide ratio from 2 to 2.5, caused a reduced water absorption rate of 17, 13, and 15%. By the increase of the monomer ratio, there was no significant change in the amount of water absorption. The electrical resistivity in specimens containing 0.3, 0.5, and 1% polypropylene fibers and a monomer ratio of 2.5 were 94, 91, and 80% higher than the specimens with a monomer ratio of 2, respectively. Furthermore, the electrical resistivity of the specimens with the same rates of polypropylene fibers but with a monomer ratio of 3 increased by 119, 102, and 82% with respect to the similar specimens with the moment ratio of 2, respectively. The probability of corrosion in specimens with the monomer ratio equal to 2 ranged from low to medium. Also, by the increase of the sodium silicate to sodium hydroxide ratio, they moved to the low range region. The results show that applying 10% polypropylene fibers with the sodium silicate to sodium hydroxide ratio equal to 3 yields good results in terms of increased mechanical and durability characteristics and improved geopolymer concrete microstructure. Observing the images taken by scanning electron microscopy from hardened geopolymer pastes reveals a considerable increase in density and uniformity of the polymer specimens having appropriate monomer ratios. The test results had good compatibility with the scanning electron microscopy images.

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AUTHORS CONTRIBUTION

This work was carried out in collaboration among all authors.

CONFLICT OF INTEREST

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