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Workability and Compressive Strength Properties of Fly Ash-Metakaolin based Flowable Geopolymer Mortar

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Abstract

Reactive Powder Concrete is a high-strength concrete with outstanding technical qualities. Cement production is seen as an environmentally unsustainable process. As a result, it is necessary to substitute cement in RPC manufacturing with an environmentally acceptable binder. Geopolymer seems to be a novel binder that can completely replace cement. The properties of constituents and their percentages in the mix significantly affect the behavior of geopolymer concrete or mortar. This research aims to produce Geopolymer RPC (GRPC) and verify the impact of the ratios of fly ash/pozzolanic materials (FA/P), sand/pozzolanic materials(S/P), finer sand/fine aggregate (S2/S1), and alkaline solution/pozzolanic materials (A/P) on its mechanical and durability properties. The results of the current works demonstrate that increase in alkaline solution to binder ratio increase the compressive strength of the mortars from 62.28 to 70.01 MPa at 62.50% to 100% alkaline/binder ratio, respectively. As well as for the same alkaline/binder ratio the workability subsequently improves from 15 to 17.3mm

Keywords

RPC (Reactive Powder Concrete), Geopolymer concrete, Flow diameter, Compressive strength

1. Introduction

The Geopolymer Concrete (GPC) technique includes the creation of waste material-based concrete that is friendlier to the environment and might be a potential alternative for traditional concrete (Al-Husseinawi et al., 2022; Al-Khafaji et al., 2018; Zeini et al., 2023). GP binder is produced through a chemical reaction called geo-polymerization. Geopolymerization is when the alumino-silicate source is chemically reacted with alkali and calcium poly-silicates to produce Si-O-Al bonds (Davidovits, 1994). Nevertheless, standard GPC calls for a curing process at a high temp to generate acceptable early strength qualities. This poses a significant challenge for applications using cast-in-place concrete because of its severe constraint. Most of the research that have been done in the past on GPC have focused on the qualities of concretes that have been pre-hardened using heat curing or by harsh chemical treatment (for example, alkali activation utilizing concentrated sodium hydroxide (NaOH)). (Azarsa and Gupta, 2020).

Fly ash from coal combustion is one of the various types of aluminosilicates that have been investigated for their potential to undergo geo-polymerization. Fly ash is a by-product of the burning of coal, and it is divided into two categories, low-calcium fly ashes (fly ashes with a calcium concentration of less than 10 weight percent) and high-calcium fly ashes (fly ashes with a calcium content of more than 10 weight percent) (ASTM Class F and Class C fly ashes, respectively) (Al-Faluji et al., 2021; Falah et al., 2021; Majdi et al., 2020; A.A. Shubbar et al., 2020; Ali Abdulhussein Shubbar et al., 2020). The characteristics of fly ash play a key part in the geo-polymerization process; the exact nature of this involvement varies based on the kinds of coal used and the combustion method (Noor-ul-Amin, 2014; Sun, 2020).

Utilizing calcined clays as supplemental cementitious materials offers the possibility of greatly lessening the carbon load that the cement industry is responsible for. Clays that have been calcined may be utilized as a precursor in the production of GP cement. Metakaolin (MK) is a kind of calcined clay that has the benefits of being chemically consistent, having high thermal reactivity, and having low permeability. It is also a type of calcined clay. The large specific surface area of the metakaolin particles results in an increase in both the amount of mixing water required and the yield stress. Additional impacts include a reduction in durability, an increase in efflorescence, and possible harm to the pore structure (Hanein et al., 2021). There has been much more study conducted on the reactions, binders, and microstructure of each system. When re-designing mixes, utilizing fly ash as a substitute for metakaolin has the possibility to be economically beneficial and has good technical qualities. (Xia and Sanjayan, 2018; Zhang et al., 2014).

The category of ultra-high-performance concrete includes the material known as reactive powder concrete, or RPC (Al-Baghdadi et al., 2021; Al-Khafaji et al., 2021; Al-Khafaji and Falah, 2020; Ali et al., 2022; Falah and Al-khafaji, 2022; Falah et al., 2020; Majdi et al., 2020; Marshdi et al., 2021). The reason behind the name "reactive powder" is that all of the powder components that make up RPC were chemically reactive to one another (Tuama et al., 2020). Researchers have shown that ultra-high-performance concrete (UHPC) does not qualify as concrete since it does not include coarse aggregate (Mayhoub et al., 2021). Nonetheless, the word "concrete" rather than "mortar" is used to characterize UHPC because it contains fine steel fibers that increase the material's ductility (Falah et al., 2022). On the other hand, RPC may be made without the incorporation of fibers at all. Methods of microstructural modification have been employed to create RPC by adjusting its characteristics like its excellent durability, high compressive strength, and exceptional toughness. These improvements have been made possible thanks to the employment of RPC. The term RPC stands for reactive powder concrete, which is a unique kind of concrete that has exceptional qualities, most notably high compressive strength. On the other hand, one of the drawbacks of RPC is that it has a high concentration of cement, which may exceed 1000 kg/m3 in certain cases. Researchers have resorted to studying alternatives to cement, like supplemental cementitious materials, due to the high expense of creating cement as well as the greenhouse gas emissions involved with its creation.

Although RPC is an extremely valuable type of concrete that may attain extremely high strengths, it behaves poorly in the event of a fire under explosive spalling conditions (Falah and Al-khafaji, 2022; Falah et al., 2022). In addition, RPC has a negative environmental impact, particularly because of the high cement content used in the mixing matrix, which contributes to a higher carbon footprint. However, GPs have lesser strengths but better fire resistance than RPC. GP and RPC have been the subject of several investigations, but the number of studies examining the effects of GP and RPC in combination is far smaller (Ju et al., 2011; Peng et al., 2012; Zheng et al., 2013). This research aims to develop an alkaline-activated reactive powder geopolymer concrete (RPGCC) based on a combination of FA and MK. The current investigation aims to create new trends of reactive powder concrete that are produced by environmentally friendly concrete (geopolymer concrete).

2. Experimental Works

2.1 Materials

The test mortars were produced using the following materials:

- Metakaolin (MK) is a de-hydroxylated form regarding clay mineral kaolinite that conforms to ASTM C618-19 (ASTM C618 - 19, 2019). At temperatures between 100 and 200 °C, kaolinite loses most of its adsorbed water, and then it de-hydroxylates at temperatures between 500 and 700 °C. The procedure that changes kaolinite to metakaolin is an endothermic procedure because it requires a large amount of energy to eliminate the hydroxyl's chemically bonded ions, breaking the crystal structure down and creating alumina and amorphous silica phases that have a high surface area. Different reports give optimal activation temperatures ranging from 550 to 850 °C; however, 650-750 °C is most mentioned (Snellings et al., 2012). The recommended method for preparing metakaolin has been utilized in this investigation. Table 1 shows the chemical compositions of MK.
- Based on the ASTM C 618-19 (ASTM C618 - 19, 2019), fly ash (FA) Class (F) replaces 0.25% of metakaolin weight used in this research. Table 1 demonstrates the chemical analysis of FA.

Table 1. The chemical analysis of MK and FA

Compounds	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	Cao	K_2O	Na_2O	LOI
MK	51.59	38.11	1.82	0.14	0.23	0.45	0.43	0.11	6.12
FA	65.65	17.69	5.32	0.22	0.85	0.97	2.32	1.35	2.77

Table 2. Details of utilized textile.

Weight	160 g/m²
Slot mesh	4 mm * 4 mm
Slot thickness	0.52 mm
Tensile strength	2000 N/mm²

- Alkaline compounds include the sodium silicate (Na2SiO3) solution as well as the (8 and 12) molar solutions of sodium hydroxide. (NaOH) was decided upon as the alkaline liquid since it is available in pellet form, has a commercial grade, and has a purity level of 98%. Different amounts of molar concentricity may be accomplished by adjusting the proportion of caustic soda flakes to water in a solution. Na2SiO3 is a viscous liquid that is sticky, transparent to off-white in color, and has a faint odor. The proportions of sodium oxide to silicon dioxide and water determine the amount of sodium silicate, also known as Na2SiO3. The percentage of water that Na2SiO3 contains, measured by mass, approximately 55%.
- Quartz sand with maximum size 60 µm around and crushed quartz powder (approximately 5 – 20 µm).
- High range Kind F-water-decreasing, high range superplasticizer admixture for concrete according to ASTM C494 / C494M – 19 (ASTM C494 / C494M - 19, 2019).
- Commercial high-strength and alkali resistant fiberglass woven meshes with an equal quantity of fiber roving in two orthogonal directions (0°/90°) were used. Other details of this textile are listed in table 2.

2.2 Mortar mixing design and procedure.

A mortar with high flowability and early strength was required to fill the whole form and penetrate the mesh slots. Fresh GPM may benefit from fly ash that is composed of spherical silt-sized particles. When water is added to the geopolymer mortar, the workability increases, but the strength is slightly reduced. Additional workability may be gained with the use of a superplasticizer (Al-Khafaji et al., 2021; Marshdi et al., 2021; Falah et al., 2020). These factors were considered in designing the mixed proportions for the GPM. Table 3 shows the mixing ratios of geopolymer mortars.

In addition, it was necessary to combine the components to generate the GPM matrix to generate an appropriate mix. It was a two-part mix consisting of geopolymer-based materials (metakaolin and fly ash) and alkaline activators (sodium silicate and sodium hydroxide), and it was created in a proportion of geopolymer-based materials to activator. GPM stood for geopolymer-based materials and alkaline activators (1:1). According to (ASTM E200 - 16, 2016), the high 98% purity NaOH flakes dissolved with water to produce the NaOH solution with two concentrations (8 molar and 12 molar). The prepared NaOH solution was left for 24 hours and then mixed with Na2SiO3 solution to get the alkaline solution activator. The mixing ratio of the alkaline solution activator (Na2SiO3/NaOH) was 2.5. The alkaline solution activator should be left for 30 minutes before mixing with other components. The increased water requirement of mortar mixes was discovered by using a flow diameter of 110 mm ± 5 mm, as stated by ASTM C230 (ASTM C230 / C230M - 21, 2021). In the lab, a Hobart mixer was used to combine dry mixed basic ingredients and sand for a period of three minutes. After combining the alkaline solution, the additional water, and the superplasticizer for a period of five minutes, the liquid solution was thereafter slowly added to the dry mixture. The room temperature for combining was 23 degrees Celsius.

Table 3. Composition of geopolymer mortar matrix Metakaolin

Metakaolin MK	Fly ash FA	Quartz sand>0.6mm	Quartz sand<0.6mm	Activator (Na_2SiO_3+NaOH)	Water\(MK+FA)	SP by weight of (MK+FA)
0.75	0.25	2	0.2	1	0.28	0.02

2.3 Mixing Design

The current concrete was prepared by using two kinds of sands (A and B), the concrete mixture is considered reactive powder concrete since the absence of coarse aggregates. In all mixes the water proportion and SP ratio were fixed (0.19 and 0.02), respectively. while the other materials that were used in the mixture were varying as demonstrated in Tables 4 and 5. However, the study was mainly consisting of four groups: the first one has three samples with changeable Alkaline \binder ratio; the second group has four samples with changeable FA/Binder ratio; the third group has three samples with changeable sand/binder ratio and final group has three samples with changeable sand B/sand A ratio.

Table 4. The mixing proportion for the used materials

Mix	Binder		FA/Binder	Sand > 0.6mm (A)	Sand < 0.6mm (B)	Sand / binder	Sand (B/A)	NaOH	Na_2SiO_3	Alkaline / binder
	Fly Ash	Metakaolin								
GPM1	0	1	0.00	1.85	0.15	2	0.08	0.18	0.45	0.6
GPM2	0	1	0.00	1.85	0.15	2	0.08	0.23	0.57	0.8
GPM3	0	1	0.00	1.85	0.15	2	0.08	0.28	0.72	1.0
GPM4	0.05	0.95	0.05	1.85	0.15	2	0.08	0.28	0.72	1.0
GPM5	0.1	0.9	0.10	1.85	0.15	2	0.08	0.28	0.72	1.0
GPM6	0.2	0.8	0.20	1.85	0.15	2	0.08	0.28	0.72	1.0
GPM7	0.2	0.8	0.20	1.38	0.12	1.5	0.08	0.28	0.72	1.0
GPM8	0.2	0.8	0.20	2.31	0.19	2.5	0.08	0.28	0.72	1.0
GPM9	0.2	0.8	0.20	1.80	0.20	2	0.11	0.28	0.72	1.0
GPM10	0.2	0.8	0.20	1.88	0.12	2	0.06	0.28	0.72	1.0

Table 5. The mixing proportion percentage for the used materials

Mix	FA/Binder	sand/binder	sand B/sand A	alkaline\binder
GPM1	0%	200%	8%	62.50%
GPM2	0%	200%	8%	80.00%
GPM3	0%	200%	8%	100.00%
GPM4	5%	200%	8%	100.00%
GPM5	10%	200%	8%	100.00%
GPM6	20%	200%	8%	100.00%
GPM7	20%	150%	8%	100.00%
GPM8	20%	250%	8%	100.00%
GPM9	20%	200%	11%	100.00%
GPM10	20%	200%	6%	100.00%

3. Testing Methodology

3.1 Flow test

The ability of concrete to be worked on is a complex characteristic that has a direct impact on the material's strength, quality, and appearance. In addition to this, it determines the ease with which freshly mixed concrete may be poured, compacted, and polished with very little to no loss of homogeneity. As previously said, it is a complicated quality that is dependent on several parameters to produce excellent workable concrete. The concrete workability was measured in this test by studying the flowing quality of concrete, as the name indicates. The concrete quality is also identified by the flow table test in terms of segregation proneness, cohesion, and uniformity. There are two ways for determining the flow value of concrete, one of which is obsolete. We'll go through the new flow table test approach. The BS 1881 standard covers this new flow table test (Standard, 2009).

The flow diameter has been employed in line with the ASTM C1437 standard to determine the workability of fresh geopolymer mixes (Standard, 2013). The spread diameter was determined over all four perpendicular edges of the flow table. The results that were given have become a means of these measurements. Every combination was tested twice immediately after combining all the elements.

3.2 Compressive Strength

In order to evaluate the behavior of Geopolymer RPC, a compressive strength test was carried out in accordance with ASTM C109 (ASTM, 2008), on cubic specimens of 50 mm on each side and 50 mm in total height. Utilizing testing equipment (Controls, Milan, Italy) with a loading rate of 0.33 MPa and in accordance with ASTM C109, the compressive strength of Reactive powder concrete-based geopolymer mortar specimens was measured at the ages of 3, 7, and 28 days after their first mixing (ASTM, 2013). For every combination, a set of three specimens was examined and each specimen represented an age.

4. Results And Discussion

4.1 Flow test

Table 6 demonstrates the findings of flow test for different samples. Figure 1. show the relationship between the alkaline\binder and flow diameter (mm), where the minimal flow diameter has been obtained in sample GPM1 with 15 mm, and the diameter increased with increasing alkaline\binder ratio from 62.50% to 80 and 100%. And Figure 2. show the relationship between the FA/Binder and Flow Diameter (mm), where the minimal flow diameter has been obtained in sample GPM3 with 17.3 mm, and the diameter improved with increasing FA/Binder proportion from 0% to 5%, 10%, 20% and 100%. As can be observed in figures 1 and 2, the flowability of one-part fly ash-based geopolymer binders is greatly impacted by the kind of alkaline activator that is used. Generally, combinations activated with just NaOH had the lowest flowability. This might be attributable to the increased heat created owing to the exothermic reaction of NaOH with water, which caused the NaOH to react with the water in an exothermic manner. The Na2SiO3-activated combination had the greatest beginning flow of the group, although it declined dramatically from 17.3 to 20.5 mm during the experiment (Yousefi Oderji et al., 2019). When the proportion of solutions to fly ash in the geopolymer mortar is increased, the resulting mix has an adequate flowability. On the other hand, when the proportion of solutions to binder is decreased, the mixture becomes more rigid. In a similar manner, the workability of the mixture decreases with an increase in the sodium hydroxide content at certain ratios of solutions to fly ash (Haruna et al., 2018).

Table 6. Results of Flow tests for all samples.

Mix	Flow Diameter mm
GPM1	15
GPM2	16.5
GPM3	17.3
GPM4	18.2
GPM5	19.3
GPM6	20.5
GPM7	19.7
GPM8	21.3
GPM9	19.8
GPM10	18.5

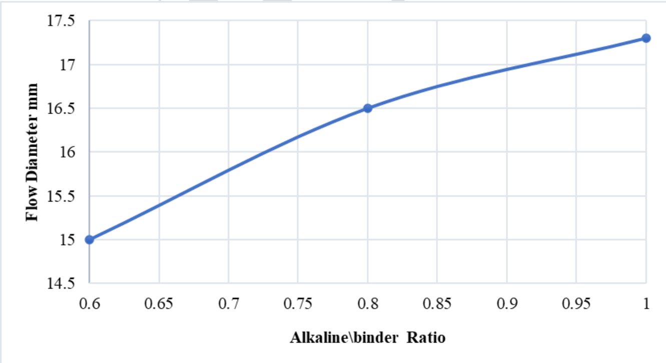

Figure 1. the influence of alkaline\binder on the geopolymer concrete Flow Diameter (mm).

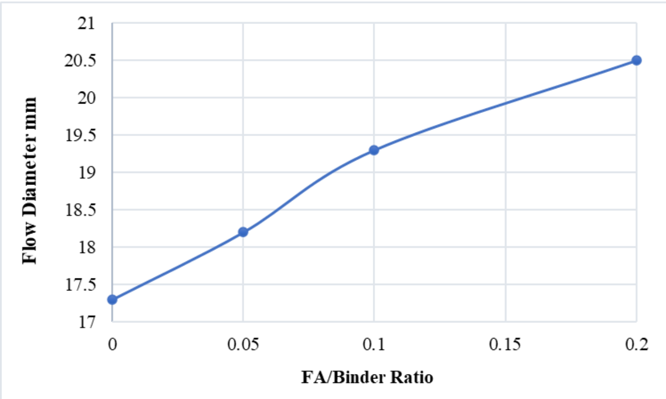

Figure 2. The influence of FA/Binder on the geopolymer concrete Flow Diameter (mm).

On the other hand, Figure 3 demonstrated the influence of sand/binder on the reactive powder concrete based-geopolymer Flow Diameter, where the ratio of the sand to binder has a fluctuating influence on the concrete flow diameter. Increasing the ratio of sand to binder from 100% to 150% lead to decrease the flow diameter approximately 25% comparison with sample GPM6, while increase the ratio of sand to binder from 100% to 200% lead to increase the flow diameter approximately 25% comparison with sample GPM8.

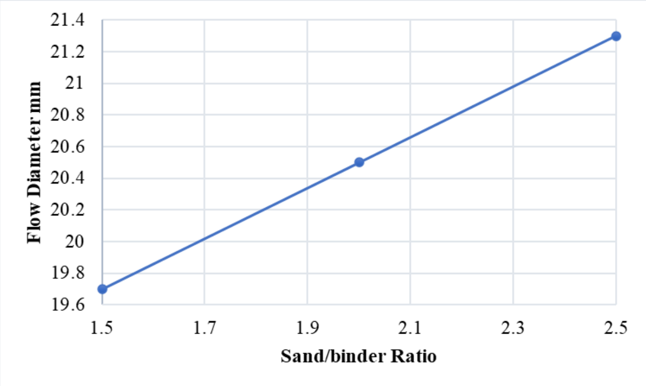

Figure 3. The influence of sand/binder on the geopolymer concrete Flow Diameter (mm).

As well as Figure 4 demonstrated the influence of different kind of sands ratios (sand B/sand A) on the reactive powder concrete based-geopolymer Flow Diameter, where the highest flow has been obtained in sample GPM6 with 8% (sand B/sand A) ratio, which refer to use higher amount of small particles sands with less than 0.6. And increasing or decreasing (sand B/sand A) ratios lead to reduce the flow diameter as shown in samples GPM9 and GPM10, where flowability of geopolymer increased with decreasing the particle size of sands (Gholampour et al., 2019).

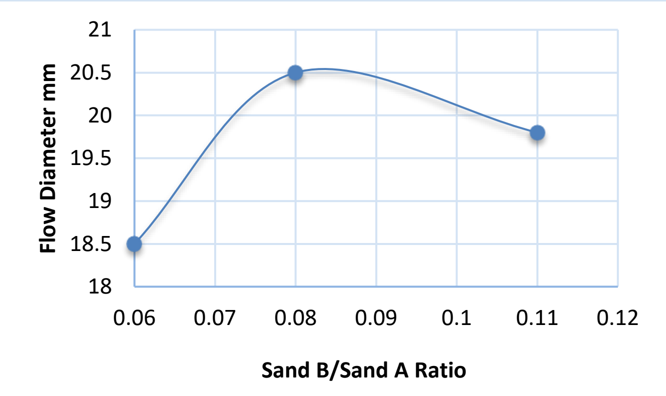

Figure 4. the influence of sand B/sand A on the geopolymer concrete Flow Diameter (mm).

4.2 Compressive Strength

After a total of twenty-eight days of curing, a variety of reactive powder concrete based-geopolymer mixtures were tested for their increasing compressive strength. The findings are shown in the form of a 3D response surface plot of the compressive strength of reactive powder concrete based-geopolymer samples after 28 days at a variety of mix conditions in figures 5 through 8. The findings displayed in Table 7 and Figures 5-8 represent the mean magnitude of the three specimens that were examined at the age that was being evaluated. It is important to note that all the geopolymer mortars had been able to attain greater than 15 MPa at a young age, and that at least 30 MPa was obtained for all the mixes after curing for a period of 28 days.

Table 7. Compressive strength results for 3, 7 and 28 curing days.

Mix	Comp. Strength MPa		
	Fcu 3	Fcu 7	Fcu 28
GPM1	38.72	53.13	62.28
GPM2	43.70	58.84	66.52
GPM3	46.42	61.74	70.01
GPM4	47.53	63.21	72.50
GPM5	49.66	66.54	76.42
GPM6	52.37	69.45	79.93
GPM7	50.56	66.93	76.25
GPM8	48.71	63.87	72.07
GPM9	50.96	67.76	77.26
GPM10	46.64	62.86	70.99

Figure 5 demonstrated the association between the alkaline\binder and compressive strength (MPa) at various curing ages (3, 7 and 28) days, where the minimal compressive strength has been obtained in sample GPM1 for all selected curing ages, and the Compressive strength improved with increasing alkaline\binder proportion from 62.50% to 80 and 100%. And the increasing in the Compressive strength during the time are compatible with the increasing alkaline\binder ratio.

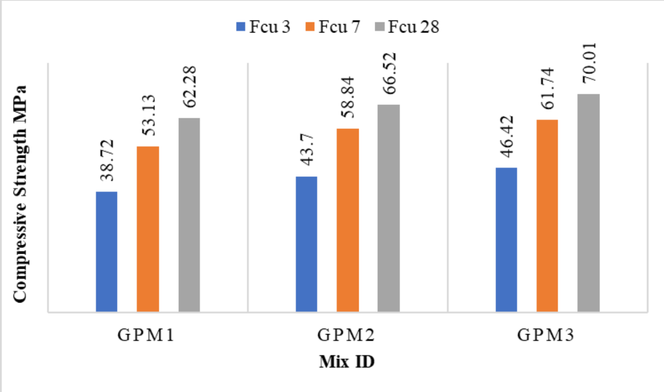

Figure 5. The influence of alkaline\binder on the geopolymer concrete compressive strength (MPa).

Figure 6. demonstrate the association between the FA/Binder and Compressive strength (MPa), where the minimal compressive strength has been obtained in sample GPM1 for all selected curing ages, and the Compressive strength improved with increasing FA/Binder proportion from 0% to 5%, 10%, 20% and 100%. And the improvement in the compressive strength during the time is compatible with the increasing FA/Binder ratio.

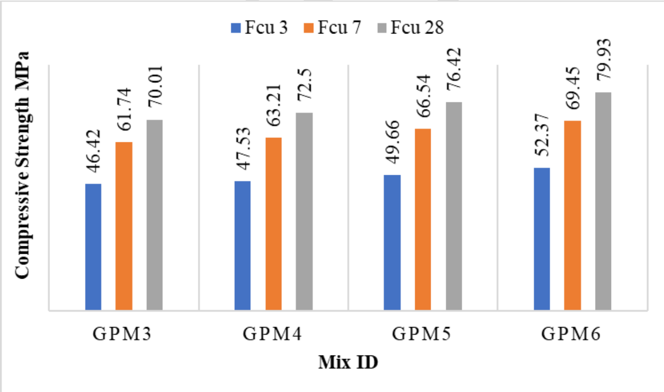

Figure 6. The influence of FA/Binder on the geopolymer concrete compressive strength (MPa).

Furthermore, to the reported behavior of geopolymer samples treated at various curing ages (3, 7, and 28 days), the experimental findings presented previously indicate that the strength development pattern in a customized hybrid binder might unavoidably be influenced by the nature of the source materials, the amount of alkaline solution, or the fine aggregates in a particular mixture. This is the case regardless of the reported behavior of geopolymer samples treated at various curing ages (3, 7, and 28 days). As a result, a comparison of the compressive strengths of geopolymer mortars with two various proportions of sand to binder is demonstrated in Figure 7. These findings are also connected to the experiment that was conducted in the past by (Khan et al., 2016), in which a various sand-binder proportion of 100%, 150%, and 200% was utilized. Despite this, increasing the amount of sand to binder resulted in a reduction in compressive strength after 3, 7, and 28 curing days.

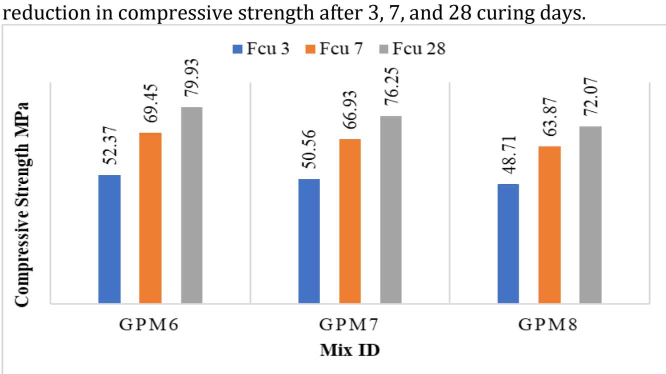

Figure 7. The influence of sand/binder on the geopolymer concrete compressive strength. (MPa).

Figure 8. represent the effect of changing sand B/sand A ratio on the compressive strength of geopolymer-reactive powder concrete for 3, 7 and 28 curing days. It has been observed that increasing or decreasing sand B/sand A ratio from (0.8) lead to decreasing compressive strength for all selected periods, therefore GPM6 records highest compressive strength value (Gholampour et al., 2019).

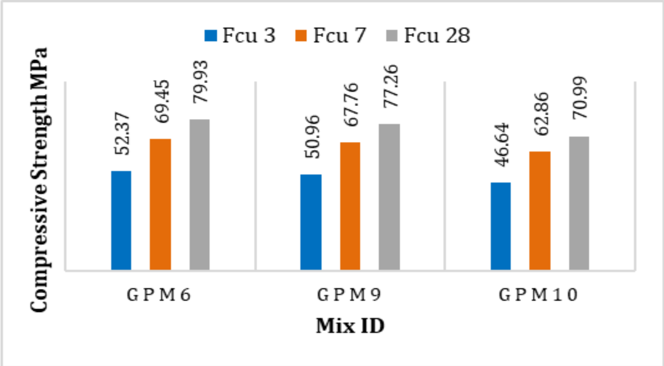

Figure 8. The influence of sand B/sand A on the geopolymer concrete compressive strength (MPa).

5. Conclusion

Based on the experimental finding that conducted on new trends of reactive powder concrete that produced with geopolymer cement, the following conclusion could be drawn down:

- Increasing alkaline\binder and FA/Binder ratios led to increasing the flowability of the created concrete, while changing in the (sand B/sand A) ratio 0.08 to (0.06 and 0.11) was reduced the flowability. And the higher flowability diameter was (GPM8=21.3mm), which was obtained when increasing sand/binder ratio to 250%.
- Increasing the binder to fly ash proportion decreases the strength of the geopolymer mortars and consequently improved the flowability of the mortars.
- Utilizing metakaolin alone had the lowest flow diameter value and compressive strength comparison with other selected mixtures.
- Higher amount of NaOH results in worsening the compressive strength findings at outdoor curing.
- Increasing alkaline\binder, sand/binder and FA/Binder ratios led to increasing the compressive strength of the created concrete, while changing in the (sand B/sand A) ratio 0.08 to (0.06 and 0.11) reduced the compressive strength. And the higher compressive strength was (GPM6=79.93 MPa), which was obtained when sand/binder ratio was 200% and any increase or decrease in this ratio led to decrease the compressive strength.

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