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Influence of Kaolin in Fly Ash Based Geopolymer Concrete: Destructive and Non-Destructive Testing

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Abstract. Development of geopolymer concrete is mainly to reduce the production of ordinary Portland cement (OPC) that adverse the natural effect. Fly ash is a by-product collected from electrical generating power plant which resulted from burning pulverized coal. Since fly ash is waste materials, it can be recycled for future advantages particularly as pozzolanic materials in construction industry. This study focused on the feasibility of fly ash based geopolymer concrete to which kaolin has been added. The main constituents of geopolymer production for this study were class F fly ash, sodium silicate and sodium hydroxide (NaOH) solution. The concentration of NaOH solution was fixed at 12 Molar, ratio of fly ash/alkaline activator and sodium silicate/NaOH fixed at 1.5 and 2.5, respectively. Kaolin was added in range 5% to 15% from the mass of fly ash and all the samples were cured at room temperature. Destructive and non-destructive test were performed on geopolymer concrete to evaluate the best mix proportions that yield the highest strength as well as the quality of the concrete. Compressive strength, flexural strength, rebound hammer and ultrasonic pulse velocity (UPV) result have been obtained. It shown that 5% replacement of kaolin contributed to maximum compressive strength and flexural strength of 40.4 MPa and 12.35 MPa at 28 days. These result was supported by non-destructive test for the same mix proportion.

1. Introduction

An increasing demand in more environmental friendly construction materials has been driving force for researchers to develop innovative solution such as utilizing waste materials from coal industries and decomposition of natural resources [1]. Geopolymer was initially created by Joseph Davidovits, which comprises of SiO_4 and AlO_4 tetrahedral systems. Alumina-silicate responsive materials break up in solid antacid arrangements and free SiO_4 and AlO_4 tetrahedral structure shapes.

Geopolymer material has caught the enthusiasm of numerous researchers from various fields since it exhibits amazing properties such as high compressive strength, resistance towards fire, corrosion, thermal and many more. Besides, it is an environment friendly because of the way that it can be set up from industrial wastes and natural resources. The main materials use to produce geopolymer are

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alumina-silicate materials such as fly ash, kaolin, slag, and rice husk ash, palm ashas well as alkaline activators [2-4].

For past thirty years, through a variety of new and better processes, many clay-based products have been produced with high quality. The increased in fabrication of clay-based product has further expanded conventional applications by bringing more innovation of modern applications. Kaolin is white or close white shading with pseudo-hexagonal precious stone alongside plates, some bigger books and vermicular stacks. However, utilization of kaolin as geopolymer source material produce weak structure due to low reactivity where it required certain period of times in order to increase the geopolymerisation rate for strength gain [5].The low reactivity of kaolin as source material for geopolymer due particle shape of the kaolin itself where the small surface area allowed minimum dissolution of Si and Al by alkaline activator hence lower strength [6]. When kaolin was activated by alkaline activator, the reaction starts from the surface and edge where it slowly penetrates inside the structure hence cause the low strength performance for kaolin based geopolymer [7]. Nevertheless, kaolin based geopolymer has favourable properties in which it has good volume stability in the water. In addition, kaolin also has good compressive strength and can be supplemented with increased ageing. However, the development of strength is very slow and it is supposed due to limitation of kaolinite structure that has low surface area and limited replacement of other element during the reaction.

Van Jaarsveld et al. [8] found that the inclusion of high kaolin content (41 wt.%) in fly ash based geopolymer reduced the strength of final product. This result was obtained because not all kaolin involved in geopolymerisation process to form the geopolymer network. Meanwhile, Okoye et al. [9] investigated the replacement of fly ash with kaolin up to 50% in fly ash based geopolymer concrete where it was found that, compressive strength increased with higher replacement of kaolin.

The current study, fly ash is replaced with different content of kaolin (0% until 15%) where the destructive and non-destructive test was conducted on fly ash-kaolin geopolymer concrete to identify the homogeneity of these two materials.

2. Materials and methods

2.1. Materials

Class F fly ash was obtained from CIMA, Perlis, Malaysia and kaolin was supplied by Associated Kaolin Industries Sdn. Bhd., Malaysia. The chemical composition for both materials is tabulated in Table 1 which obtained from X-ray Fluorescence (XRF) analysis. Figure 1a and 1b showed the morphology of fly ash and kaolin particles.

Table 1. Chemical composition of fly ash and kaolin.

Constituents	Mass (%)	
	Fly Ash	**Kaolin**
SiO_2	55.9	54.0
Al_2O_3	27.8	31.7
CaO	3.95	-
Fe_2O_3	7.09	4.89
TiO_2	2.25	1.41
K_2O	1.55	6.05
SrO	0.37	-

Sodium hydroxide (NaOH) pellets were dissolved using distilled water to produce 12 Molar concentration [10]. The NaOH solution was prepared 24 hours prior the mixing process of geopolymer samples. Technical grade of sodium silicate solution was mixed with NaOH solution to produce alkaline activator. Crushed stone with size in ranges from 6mm to 8mm were used as coarse aggregates and river sand was used as fine aggregates.

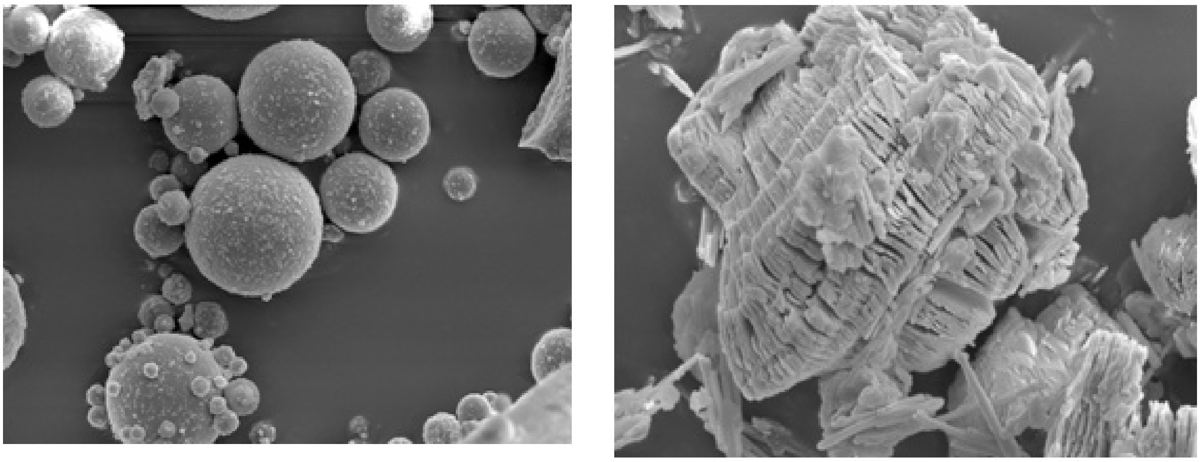

Figure 1. (a) Morphology of fly ash (b) morphology of kaolin observed using FESEM.

2.2. Mix design and mixing process

Sodium silicate and sodium hydroxide (NaOH) solution was mixed together to produce alkaline activator with ratio of 2.5 [2, 11-13]. NaOH pellets were dissolved with distilled water to obtain 12 Molar concentration [10]. Meanwhile, the ratio of fly ash/alkaline activator was fixed at 1.5 with respect to the workability of geopolymer concrete. The combination of coarse aggregate and sand contributed 70% to the total weight of geopolymer mixture. Fly ash was replaced by kaolin start from 0% (control samples), 5%, 10% and 15%. The details of mix design were presented in Table 2.

In preparation of geopolymer mixture, the fly ash was mixed together with kaolin and alkaline activator for about 3 minutes in the mixer to ensure homogeneity of the mixture. Then, sand and coarse aggregate were added and mixed for another 5 minutes. The geopolymer concrete were poured into 100mm x 100mm x 100mm mould and vibrated on the vibrating table for 5 minutes to release entrapped air. The samples were left at room temperature for 3, 7 and 28 days. For flexural strength only the optimum design was used to produce geopolymer samples. The geopolymer mixture were casted in prism mould 100mm x 100mm x 500mm and tested for 3, 7 and 28 days.

Table 2. Mix proportion of geopolymer concrete.

Mix	**Quantity of Ingredients (kg/m^3)**					
	Fly Ash	**Kaolin**	**Sand**	**Coarse Aggregates**	**NaOH solution**	**Sodium silicate**
FAK0 (control)	432	0	672	1008	82	206
FAK5	410.4	21.6	672	1008	82	206
FAK10	388.8	43.2	672	1008	82	206
FAK15	367.2	64.8	672	1008	82	206

2.3. Testing

The performance of fly ash-kaolin based geopolymer concrete were prepared for destructive (DT) and non-destructive test (NDT). The NDT consists of Ultrasonic Pulse Velocity (UPV) and rebound hammer while the DT comprising both compressive strength and flexural strength.

2.3.1. Ultrasonic Pulse Velocity. UPV test was conducted on samples at the age of 3, 7 and 28 days in order to examined the impact of partially replacing fly ash with kaolin on geopolymer concrete uniformity. A set of geopolymer concrete comprising of three cubes were tested by measuring the velocity of ultrasonic pulses for three times by direct, semi-direct and indirect transmission which means nine reading for each cube. Then, the result for each transmission was compared with the guidelines as in Table 3.

Table 3. General guideline for concrete quality based on UPV [14].

Pulse Velocity (m/s)	Concrete Quality
> 4500	Very good to excellent
3600 - 4500	Good to very good but slight porosity may exist
3000 - 3500	Satisfactory but loss of integrity is suspected
2100 - 3000	Poor and loss of integrity exist
< 2100	Very poor

2.3.2. Rebound Hammer. The rebound hammer test was conducted on the geopolymer concrete with smooth, clean and dry surface. For some rough surfaces present on the concrete, the surface was rubbed with grinding wheel or stone. The point of impacting the hammer should be 20mm far from the edge and discontinuity shapes. The rebound hammer should be kept perpendicular to the surface of the concrete.

On each surface, numbers of observations are taken and the average of these observations gives the strength of concrete. The rebound hammer test was carried out on specimens at the age of 3, 7 and 28 days in order to determine the effect of partially replacing fly ash with kaolin on geopolymer surface hardness and penetration resistant. Table 4 shows the guidelines values for rebound hammer test.

Table 4. General guideline for concrete quality based on rebound number [15].

Rebound no.	Quality of Concrete
> 40	Very good
30 - 40	Good
20 - 30	Fair
< 20	Poor

2.3.3. Compressive strength. Compressive test was conducted on cube samples at day 3, 7 and 28 where the samples were tested by using hydraulic test machine. Compressive strength test was carried out on three cube samples for each mix design. Samples were tested by using hydraulic test machine governed by BS. 1881: Part116: 1983. Load was applied gradually without shock at a nominal rate of 0.3 $N/(mm^2.s)$.

*2.3.4. Flexural strength.*In order to quantify flexural strength of geopolymer concrete containing diverse rates of kaolin as fractional substitution of fly ash, ASTM C293 was chosen as code of practice. Prisms samples were tested with pace rate of 0.06 $N/(mm^2.s)$.

The modulus of rupture resolved by centre-point loading is higher than third-point loading. For this study, the test conducted based on ASTM C293 (centre point loading), which sample had undergone the curing process for 3,7 and 28 days.

3. Results and discussions

3.1. Ultrasonic Pulse Velocity

Figure 2 illustrates the result of UPVforgeopolymer concrete with inclusion of kaolin on direct transmission mode. For this transmission mode, FAK5 shows excellent result where contributed the highest pulse velocity throughout the test days with values of 3770 m/s, 3280 m/s and 4180 m/s on 3rd, 7th and 28th days respectively. The pulse velocities of the samples decreased on 7th day of testing and increase again on 28th day. The quality of concrete is varying from satisfactory to good.

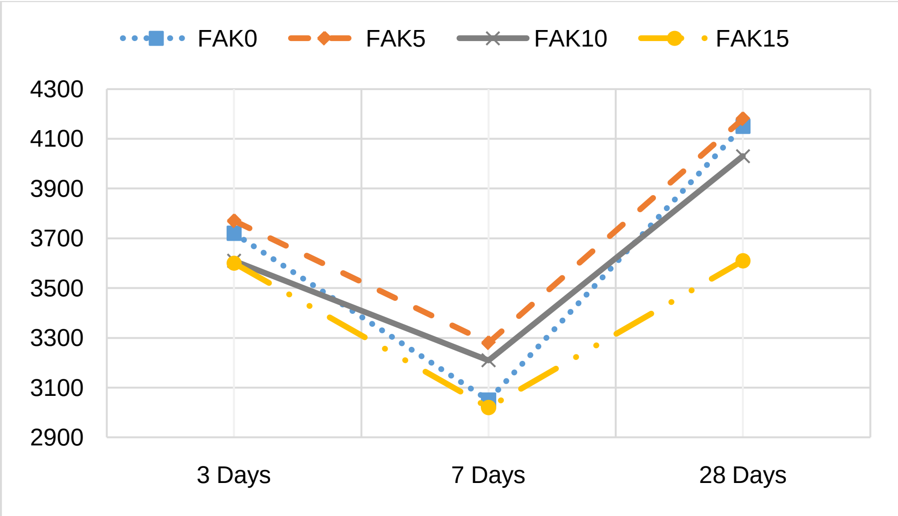

Figure 2. Ultrasonic pulse velocity result for direct transmission.

According to Benaicha et al. [16], pulse velocity usually increases very rapidly at early age of concrete and gets flatten in later age. However, from Figure 2 to Figure 4the pulse velocity decrease at early age and increase again in later age regardless any of the transmission mode. Addition of kaolin in geopolymer concrete obviously reduce the velocity of ultrasonic pulses and its homogeneity.

Figure 3 showed that on day 3, FAK15 has the closest pulse velocity to the control mix (FAK0) which has the value of 5430 m/s while FAK5 has the lowest pulse velocity of 5050 m/s. On 7th day, pulse velocity of FAK10 dropped dramatically from 5200 m/s to 3650 m/s while other mixes are slightly decreasing. The following test day, the pulse velocity of all the mixes are increasing and FAK5 has the highest pulse velocity of 5700 m/s surpassing FAK0, the concrete without kaolin. Meanwhile, on 28th day, FAK15 unexpectedly has the lowest pulse velocity of 5010 m/s. By referring to Table 4, most of the geopolymer concretes have excellent quality except for FAK10 and FAK15 on 7th day of testing, they showed good concrete quality but with slight porosity presence in them.

For indirect transmission mode, all geopolymer concretes show satisfactory to good quality of concrete as displayed on Figure 4. On the 3rd day of testing, FAK5 has the closest value of pulse velocity to FAK0 followed by FAK15 and FAK10 with pulse velocities of 3640 m/s, 3230 m/s, 3090 m/s and 2860 m/s respectively. Similar pattern with semi-direct transmission result, the pulse velocity dropped slightly on 7th day of testing and increase again on 28th day. However, pulse velocity of FAK10 slightly increase on 7th day. Again, on 28th day of testing, FAK5 has the highest pulse velocity of 3770 m/s while FAK15 has the lowest pulse velocity of 3450 m/s. However, on 28th day, FAK5 and FAK10 surpass the pulse velocity of control mix (FAK0).

From Figure 2 to Figure 4 the curing period does not have great influences on the value of ultrasonic pulse velocity. This is because addition of kaolin has caused the process of geopolymerisation take longer time to occur. Furthermore, the addition of kaolin allows the stiffness of concrete increase which resulting in enhancement of pulse velocity on 28 days.

According to the quality assessment of concrete by Saint-Pierre et al. [14], all the mixes can be classified as satisfactory to excellent quality concrete since their pulse velocity values ranging from the lowest of 2860 m/s to the highest of 5700 m/s.The increase of ultrasonic pulse velocity with age is a natural development due to the increase of stiffness by the geopolymerisation reaction. For the same concrete samples, the quality of concretes increase from day to day through geopolymerisation which makes it stronger and more durable concrete according to the time.

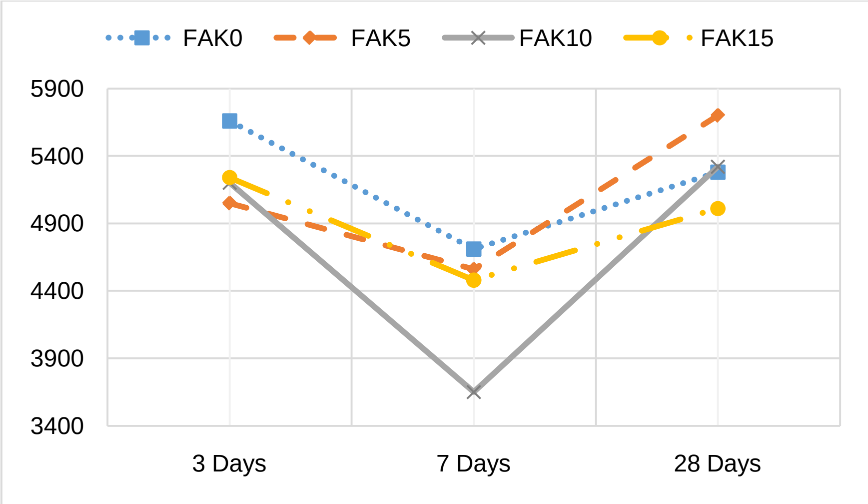

Figure 3. Ultrasonic pulse velocity result for semi-direct transmission.

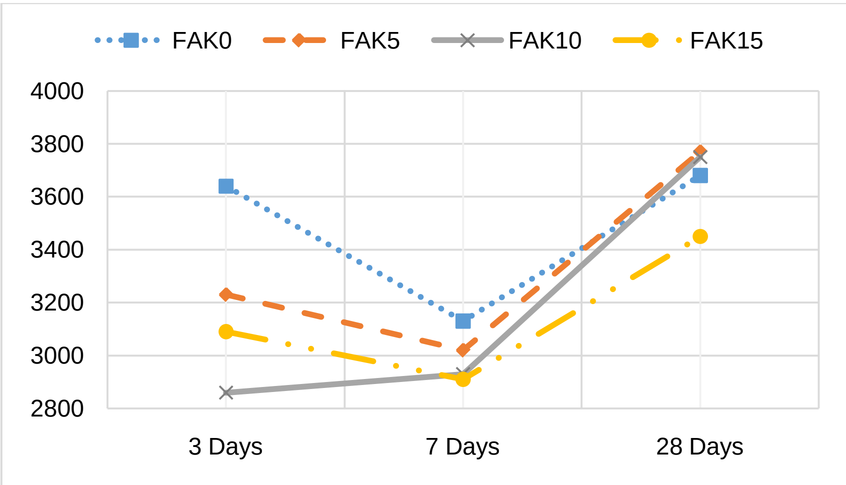

Figure 4. Ultrasonic pulse velocity result for indirect transmission.

3.2. Rebound Hammer

Based on the Table 5, all of the geopolymer concretes exhibit poor concrete quality when measured by Rebound Hammer. This result was contradictory compared to ultrasonic pulse velocity. The different result obtained between these two testing may be due to several factors that affecting the rebound hammer result. The condition of concrete surface influence the rebound number acquired during testing. Tests performed on a rough-surfaced concrete will commonly result in crushing of the surface paste, resulting in lower number and then again, tests performed on a similar concrete that has a hard, smooth surface will normally bring about a higher rebound number. Since fresh geopolymer paste has high viscosity and cohesive nature, it is important to place the concrete in the mould immediately after mixing as surrounding temperature of the geopolymer has direct connection with the mixing time. Increasing in temperature of geopolymerconcrete resulting from increase in mixing time would diminished the workability of geopolymer concrete [17]. Low workability of geopolymerconcrete make them hard to placed and thus, not achieving a smooth concrete surface. Hence, it is advised to grind the rough surface with Carborundum stone or any similar abrasive stone in order to achieve a uniform smoothness.

Table 5. Result for rebound hammer.

Mix No.	Rebound number					
	3-day	**Quality of concrete**	**7-day**	**Quality of concrete**	**28-day**	**Quality of concrete**
FAK0	12	Poor	12	Poor	16	Poor
FAK5	12	Poor	13	Poor	16	Poor
FAK10	13	Poor	14	Poor	17	Poor
FAK15	14	Poor	14	Poor	16	Poor

Agingperiod of concrete also affects the rebound number of the samples. Geopolymer concrete normally continues to develop strength with age due to geopolymerisation process. This is the reason behind the development of data relating rebound numbers to the compressive strength of the concrete mixture or cores from the structure. From Table 5, geopolymer concrete at age 3 days have low rebound numbers and for concrete at age 7 days, it has nearly similar rebound numbers. Alternately, the rebound numbers on concrete at the age of 28 days have slightly higher value but not significant as all of the concretes are in poor quality. It is not recommended to test the concrete less than 3 days old as the rebound numbers will be too low for an accurate reading. In this case, geopolymer concrete takes longer time to harden and this will affect the rebound numbers. Next, moisture content of the geopolymer samples**.** It has a profound effect on the test results in which dry concrete surfaces result in higher rebound numbers than wet surfaces. Geopolymer concrete at an early age are mostly not in fully dry condition especially concrete at age of 3 days. The concrete surfaces not dry enough due to low geopolymerisation rate between fly ash and kaolin especially with higher replacement of kaolin [19-20].

3.3. Compressive strength

Compressive strength of geopolymer concrete with various replacement of kaolin were performed and the results have been summarized in Figure 5. The compressive strength increases steadily at 3 days for FAK5 and FAK10. The compressive strength increase moderately with increasing curing ages. Nevertheless, FAK5 and FAK10 have noticeable changes in strength throughout the curing days. It is remarkable that the compressive strength developed rapidly before 7 days of curing. Corresponding to the previous research, early strength of geopolymer concrete increase rapidly.

For control sample (FAK0), compressive strength increases from 8.4 MPa at 3 days to 62.1 MPa at 28 days. Noticeable change in compressive strength of geopolymer with addition of different amounts of kaolin can be observed when compared to control sample (FAK0) at 3 days. Besides, addition of kaolin in fly ash geopolymer concrete improved the early compressive strength. Longer ageing times resulting in increasing compressive strength especially after 7 days when kaolin is added.

Addition of kaolin up to 5% increased the compressive strength of the concrete, but further increment of kaolin content resulted in slightly decrease in strength. Higher content of kaolin in fly ash based geopolymer concrete caused the mixture become stickier and takes longer time to harden.

FAK5 contributed to the highest compressive strength of 21.3 MPa and 32 MPa on both 3rd and 7th curing days while FAK0 gained the highest strength (62.1MPa) on 28th day. Nevertheless, on 28th day FAK5 displayed strength of 40.4 MPa. Meanwhile, both FAK10 and FAK15 also showed greater strength than FAK0 at the age of 3 days with strength of 15.5 MPa and 9.2 MPa respectively. This shows that inclusion of kaolin in fly ash based geopolymer concrete can enhance the early compressive strength of concrete. However, at the age of 7 days, FAK0 has slightly higher strength compared toFAK15. It can conclude that, inclusion of kaolin more than 10% slow down the hardening process of the concrete and did not contribute significantly in development of concrete strength.

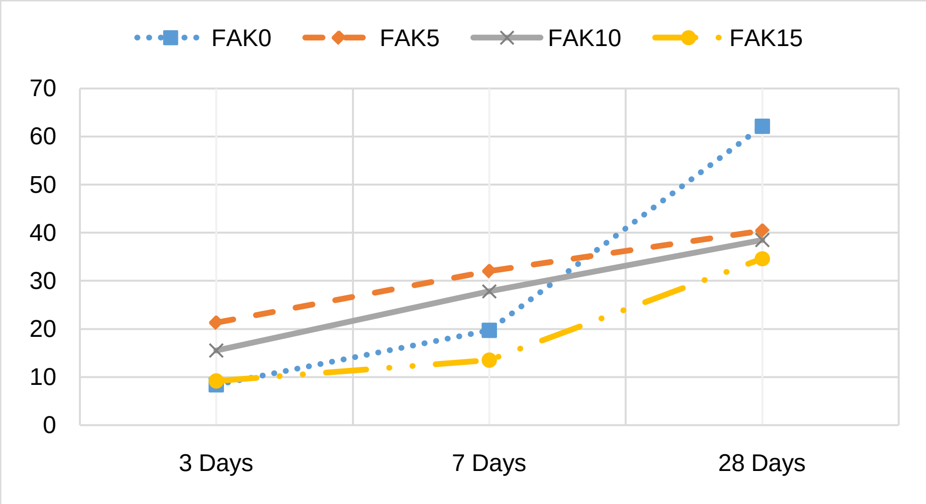

Figure 5. Compressive strength of geopolymer concrete at different aging periods.

3.4. Flexural strength

Flexural strength of geopolymer concrete with 5% of kaolin (FAK5) replacement have been summarised in Figure 6.The flexural strength at age of 3 days is 1.87 MPa and then increased dramatically from 5.74 MPa on 7th day to 12.35 MPa on 28th day. This samples displayed an increment in strength when the aging period increased which is similar pattern with compressive strength result. Based on research conducted by Nath and Sarker[18], it was found that the flexural strength of fly ash based geopolymer at 28 days is 4.89 MPa.

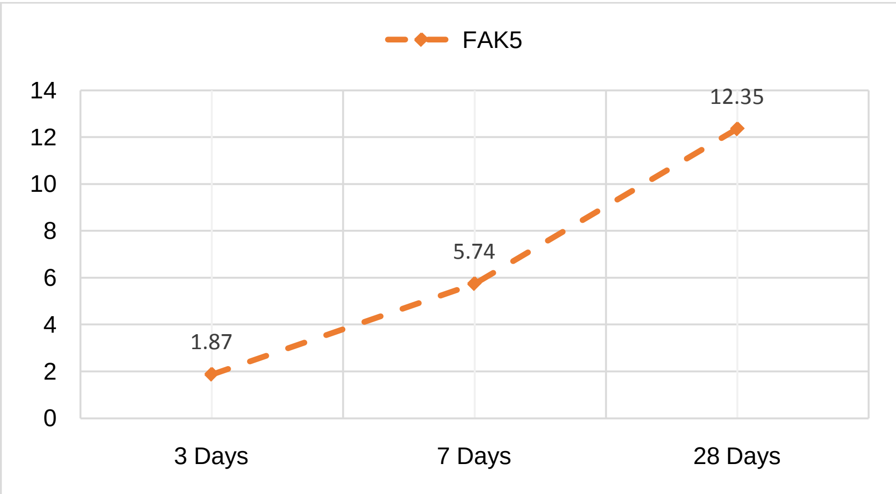

Figure 6. Flexural strength of geopolymer concrete with 5% inclusion of kaolin.

By comparing the flexural strength from previous study with this research, it is clearly showed that, the flexural strength of geopolymer concrete containing 5% kaolin is much higher. The addition of kaolin improved the long term strength of fly ash based geopolymer concrete.

4. Conclusions

The effect of partial replacement of kaolin in fly ash based geopolymer concrete were studied. Class F fly ash was blended with kaolin up to 15% replacement and the result of the study are summarized below:

i) The ultrasonic pulse velocity of all samples increased when respect to aging period. However, at the age of 7 days, the pulse velocity unexpectedly dropped for all samples. The geopolymer concrete generally have good quality concrete with slight porosity. It was noted that, the quality of the concrete is declining as the percentage of kaolin in the geopolymer mixture increase due to higher porosity and lower homogeneity.

ii) For rebound hammer, contradictory result from ultrasonic pulse velocity was obtained. All the geopolymer concretes were summarized as poor quality.

iii) FAK5 achieved the highest compressive strength at age of 3 and 7 days of 21.3 MPa and 32 MPa respectively, and second highest at age of 28 days with strength of 40.4 MPa. The control mix (FAK0) has compressive strength of 8.4 MPa, 19.7 MPa and 62.1 MPa at the age of 3, 7 and 28 days respectively.

iv) The flexural strength of FAK5 also showed increment when the aging period increased. The maximum strength recorded on 28th days is 12.35 MPa.

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