Strength and Setting Times of Low Calcium Fly

Ash-based Geopolymer Mortar

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Abstract

Geopolymer is a novel binding material produced from the reaction of fly ash with an alkaline solution. In Geopolymer mortar, Portland cement is not utilized at all. In this research, the influence of various parameters on the short term engineering properties of fresh and hardened low-calcium fly ash-based Geopolymer mortar were studied. Tests were carried out on 50 x 50 x 50mm cube Geopolymer mortar specimens. The test results revealed that as the concentration of alkaline activator increases, the compressive strength of Geopolymer mortar also increases. Specimens cured at temperature of $65^{o}C$ for 1 day showed the highest 28 days compressive strength. The mass ratio of activator/fly ash of 0.4 produced the highest 28 days compressive strength for the specimen. The obtained compressive strength was in the range of 1.6MPa – 20MPa.

Keywords: Geopolymer mortar, Compressive Strength, Setting times

1. Introduction

Portland cement concrete industry has grown astronomically in recent years. It will continue to grow as the result of continuous urban development. However, Portland cement concrete poses problems such as durability and carbon dioxide emission. Many concrete structures have shown serious deterioration, way before their intended service life, especially those constructed in a corrosive environment (Mehta 1997).

Carbon dioxide emission trading is likely to be a critical factor for the construction industry, in particular, the cement and concrete industry. The World Earth Summits in Rio de Janeiro, Brazil in 1992, and Kyoto, Japan in 1997 made it abundantly clear that for long-term sustainability, rate of emission of greenhouse gases to the atmosphere must be prevented from increasing. It has been speculated that one tonne of emission can have a trading value of US$10 (Malhotra 1999, Malhotra 2004). "Tradeable emissions" refers to the economic mechanisms that are expected to be developed to help countries worldwide meet the stringent emission reduction targets established by the 1997 Kyoto Protocol (Malhotra 1999). The potential trading value after the ratification of Kyoto Protocol is expected to be between US$20 to US$30, or more per tonne of carbon dioxide (Malhotra 2002a).

The contribution of ordinary Portland cement production worldwide to greenhouse gas emission is approximately 7% of the total greenhouse gas emission to the atmosphere (Malhotra 2002b). The production of 1 tonne ordinary Portland cement consumes 4GJ energy and produces about 1 tonne of carbon dioxide to the atmosphere (Mehta 2001). About half of the carbon dioxide emissions from Portland cement production are due to calcination of limestone, while the other half are due to combustion of fossil fuel. In the year 1995, the global production of ordinary Portland cement was about 1.4 billion tonne, thus emitting about 1.4 billion tonne of carbon dioxide to the atmosphere (Malhotra 2004).

In 1978, Joseph Davidovits developed Inorganic polymeric materials and coined the term "Geopolymer" for it (1990). Geopolymer has the potential to replace ordinary Portland cement concrete and produce fly ash-based Geopolymer concrete with excellent physical and mechanical properties. Geopolymer is used as the binder to completely replace ordinary Portland cement in producing Geopolymer concrete. In order to produce Geopolymer, low-calcium fly ash needs to be activated by an alkaline solution to produce polymeric Si-O-Al bonds. Geopolymer concrete has the potential to reduce greenhouse emissions from the concrete industry by 80% (Daniel et al 2006).

Many researches have been carried out to study the compressive strength of Geopolymer concrete with different mixing proportion and test variables. However, there are very limited research data on Geopolymer mortar available in the literature. This paper presents the results of a study on the strength and setting times of fly ash-based geopolymer mortar.

2. Past Research on Geopolymer Material

Geopolymer concrete is also known as Alkali-activated concrete or Inorganic polymer concrete. Many researches have been carried out to study the mechanical properties of Geopolymer. From these researches, Geopolymer was shown to possess excellent properties such as a high early strength, low shrinkage, high resistance to freezing and thawing, sulphate attack and corrosion (Davidovits et al 1990, Davidovits 1987, Sofi et al 2006).

Davidovits (1999) conducted researches to investigate the chemical properties of Geopolymers based on silico-aluminates. He used a pure calcined kaolin called KANDOXI (**KA**olinite, **N**acrite, **D**ickite **OXI**de) produced from calcinating kaolinitic materials for 6 hours at 750 ^{o}C as the source material for geopolymer. A Geopolymer mortar sample (Si/Al ratio = 2) made from PZ-Geopoly ® cement with KANDOXI possesses compressive strength of 20MPa after 4 hours of curing at 20 ^{o}C. The final 28 days compressive strength is in the range of 70MPa to 100MPa. From his research, it was discovered that longer curing time will result in higher compressive strength.

In a research conducted by Palomo, Grutzeck, and Blanco (1999), the effects of curing temperature, curing time, the solution / fly ash ratio, and alkali activation of fly ash with high concentration of activator, on the mechanical properties of Geopolymer were studied. Palomo et al. concluded that the amorphous polymer produced in the alkaline activation of metakaolin was a zeolitic precursor. From their research, they concluded that the effect of the activator-fly ash ratio was insignificant and the increase of curing temperature accelerated fly ash activation.

Furthermore, a study was carried out by Alonso and Palomo (2001) to investigate the variables that influences the alkaline activation of metakaolin with the presence of calcium hydroxide. They concluded that the increase in activator solution concentration above 10M induced a delay in the formation of the alkaline polymer due to the stability of ion species and ion mobility, thus resulting in a decrease in flexural strength.

A study was carried out by van Jaarsveld, van Deventer and Lukey (2002) which aimed to investigate the effects of composition of source materials and curing process on the final chemical and physical properties of Geopolymers. They concluded that rapid curing and curing at high temperature resulted in cracking and can impose negative effects on the physical properties of Geopolymer.

A research was conducted by Barbosa, MacKenzie and Thaumaturgo (1999) to study the effects on the polymerization process of the molar composition of the oxides present in the mixture and the water content used. The source material was Kandoxi, which has been prepared by heating kaolinite at 700 ^{o}C for 6 hours. In their research, the authors discovered that the optimum formation and curing of sodium poly (sialate-siloxo) polymer occurred when the ratio of $Na_2O:SiO_2$ was 0.25 and the ratio of $H_2O:Na_2O$ was 10. The sodium content represents a sufficient amount to satisfy the charge balance requirements within the structure, without providing an excess which can form sodium carbonate and may disrupt the polymerization process. The authors claimed that the effect of excess water might be to dilute the reaction or to leach the more soluble components and to transport them away from the reaction zone. The water content of the mixture is also critical for satisfactory polymerization.

Van Jaarsveld, van Deventer, Lukey (2003) conducted a study and stated that the source materials determined the properties of geopolymers especially the CaO content and the water-to-fly ash ratio.

A study was carried out by Daniel, Sanjayan and Sagoe-Crentsil (2006) to study the behavior of fly ash based geopolymer paste and concrete at elevated temperature. In the research, the authors found that when fly ash-to activator ratio decreased, the compressive strength also decreased. The authors found that a longer curing time under elevated temperature did not significantly affect the strength performance of geopolymer paste. The authors drew conclusions that fly ash-to-activator ratio was the most critical parameter for elevated temperature performance following the ratio of sodium silicate-to-potassium hydroxide in activator solution.

3. Experimental Work

Fly ash is the aluminosilicate source material used for the synthesis of geopolymeric binder in this research. In this study, low calcium fly ash (ASTM Class F) from the Sejingkat Power Plant in Kuching, Sarawak, Malaysia was utilized as the source material. The breakdown of the chemical composition of the fly ash as determined by X-Ray Fluorescence (XRF) analysis is shown in Table 1.

The silicon and aluminium oxides constitute 84.6% of the fly ash and the Si to Al molar ratio is 2.06. Davidovits (1999) suggested the molar ratio of Si to Al of about 2 for producing cement and concrete. The fly ash is dark in color which is primarily due to the presence of iron oxide (Fe_2O_3).

A combination of sodium hydroxide solution and sodium silicate solution was used as the alkaline activator. Analytical grade sodium hydroxide in pellets form with 98% purity and sodium silicate with Na_2O = 12%, SiO_2 = 30%, and water = 58% by mass was used in this research. Sodium hydroxide solution was used as alkaline activator because it is widely available and is less expensive than potassium hydroxide solution.

The alkaline activator was prepared in the laboratory. In order to avoid the effect of unknown contaminants, distilled water was used to dissolve the sodium hydroxide pellets. The alkaline activator was prepared by mixing the sodium hydroxide solution with sodium silicate solution together just before the mixing of mortar to ensure the reactivity of solution. The aim of adding sodium silicate is to enhance the formation of Geopolymer precursors or the polymerization process (Xu et al 2000). Locally available fine aggregate (river sand) in saturated surface dry condition was used.

The fly ash and the fine aggregate were first dry mixed together in N50 Hobart Mixer at gear 1 for 2 minutes to ensure homogeneity of the mixture. The Hobart mixer was stopped and the mixture was activated by adding activator solution containing sodium hydroxide and sodium silicate according to the required concentration range of 8 to 16M and mixed for a further 10 minutes. Within the 10 minutes of mixing, the prepared amount of added water was poured into the mixture on two separate times, if necessary. All mixing were conducted in an air conditioned room at temperature of approximately 25 ^{o}C. Then, the Geopolymer paste was cast into 50 x 50 x 50 mm cube moulds immediately after mixing in two layers as described in the ASTM C109 standard. Each layer was tamped 25 times with a rod. All the cast specimens were vibrated on a vibrating table for 2 minutes to remove air voids.

All the specimens were transferred to the oven without delay for curing at elevated temperature of 65 ^{o}C, 70 ^{o}C, and 80 ^{o}C for 24 hours. After the oven curing, all the specimens were removed from their moulds and cured undisturbed at room temperature until the day of testing.

The testing method and formulas for volumetric density of Geopolymer mortar was evaluated according to the Archimedes method. The density test was carried out before the compressive test of the specimens.

The Geopolymer mortar specimens were tested for 7, 14, and 28 days compressive strength using the Universal Testing Machine. The specimens were subjected to a compressive force at the rate of 160kN per minute until the specimen failed. The reported strengths were the average results of the three tests.

ASTM Standard C191 for Portland cement paste was modified for measuring the setting times of fly ash-based geopolymer mortar using Vicat needle. The needle used was 1.00 ± 0.05mm in diameter. In this case, fine aggregates were excluded from the mixture proportion.

The fly ash and activator solution were placed in the mixer bowl. Then, the Hobart mixer was operated at gear 1 for 5minutes. Within the 5 minutes, the prepared amount of extra water was poured into the mix on two separate times. The Geopolymer paste was cast into the 40 mm height, 80 mm diameter conical mould in two layers. Each layer was tamped with a rod for 25 times.

The specimen was placed into the oven for curing at required elevated temperature, 65 ^{o}C, 70 ^{o}C, and 80 ^{o}C. For every 15 minutes interval, the specimen was placed on the Vicat apparatus to measure the initial setting time. First, the centre of the specimen was placed under the 10mm end of the Vicat needle and the movable rod was lowered until the 1mm needle end makes contact with the Geopolymer paste. The indicator was set to zero. The movable rod was allowed to free fall and the penetration of 1mm needle was recorded. After that, the specimen was returned to the oven for curing at the same elevated temperature.

These procedures were repeated and the penetration for every 15minutes interval was recorded until a penetration of 25 mm or less was obtained. The time for 25mm penetration was determined by interpolation. This was the initial setting time. The final setting time, defined as the time when the needle did not sink visibly into the Geopolymer paste, was also determined.

4. Results and Discussions

In this section, the experimental results are presented and discussed. Each of the test data points plotted in the figures corresponds to the mean value of the compressive strengths of the three test cubes in a series. The standard deviations are plotted on the test data points as the error bar.

A total of fourteen mixtures were made to study the influence of various parameters on the compressive strength. The details of these mixtures are presented in Table 2. The ratio of sodium silicate to sodium hydroxide solution by mass was 2.5 for all mixture proportion. This ratio was fixed at 2.5 for all mixture because the sodium hydroxide solution is more expensive than the sodium silicate solution. The mass ratio of fine aggregate to fly ash was 2.75 for all mixture.

4.1 Concentration of Sodium Hydroxide Solution

Mixtures 3 to 7 were prepared to study the effects of concentration of sodium hydroxide solution on the compressive strength of Geopolymer mortar. From Figure 1, it was observed that alkaline concentration is proportionate to the

compressive strength of Geopolymer mortar. This is rather unexpected compared to the study conducted by Alonso and Palomo (2001). Their study indicated that when activator concentration increased above 10M, a lower rate of polymer formation was produced resulting in the decrease of flexural strength. This might be due to differences in the type of source materials. Alonso and Palomo (2001) used a high purity metakaolin produced by subjecting the raw kaolin to thermal treatment at 750°C in the air for 24 hours. In contrast, low calcium fly ash (ASTM Class F) was used as the source material in this research. In addition, Alonso and Palomo carried out metakaolin activation in the presence of calcium hydroxide which was not the case in this research.

4.2 Ratio of Alkaline Activator Solution-to-Fly Ash, by Mass

Mixtures 6, 8 and 9 were prepared to study the effects of activator-to-fly ash ratio by mass on the compressive strength of Geopolymer mortar. The concentration of sodium hydroxide solution was fixed at 10M. The curing time was 24 hours at constant curing temperature of 65°C. Figure 3 shows that Mixture 8 with activator-to-fly ash ratio of 0.4 resulted in the highest compressive strength compared to Mixture 6 and Mixture 9.

From Figure 2, it was observed that the optimum activator-to-fly ash ratio is 0.40. As the amount of activator content increases, and consequently increasing the activator-to-fly ash ratio up to 0.40; the compressive strength of geopolymer mortar increases. However, when additional activator content is added, increasing the activator-to-fly ash ratio to 0.45, the compressive strength decreases. This might be due to excess of OH^{-} concentration in the Mixture 9, which will decrease the strength of geopolymer mortar. Excess sodium content can form sodium carbonate by atmospheric carbonation and may disrupt the polymerization process (Barbosa et al 1999).

Palomo, Grutzeck, and Blanco (1999) stated that activator solution-to-fly ash ratio was not a relevant parameter on the compressive strength of geopolymer binder. Current research shows a contradictory result, whereby activator-to-fly ash ratio has influence over the compressive strength of geopolymer mortar. However, this requires further studies to refine the results as the data consist only of three mixtures.

4.3 Curing Temperature

Mixtures 6, 10 and 11 were prepared to study the influence of curing temperature on the compressive strength of Geopolymer mortar. All the other test variables were held constant while the curing temperature varied. The activator-to-fly ash ratio and water-to-geopolymer solids ratio were held constant at 0.35 and 0.324 respectively.

It was observed that the strength increased when the specimens were cured at room temperature of about 25 ^{o}C after curing in the oven at elevated temperature for 24 hours. This means that the fly ash activation was incomplete within the introduced curing regime (curing at elevated temperature 65 ^{o}C, 70 ^{o}C, and 80 ^{o}C for 24 hours). From Figure 4, it was observed that the higher curing temperature does not ensure higher compressive strength at 28 days. However, as the curing temperature increases, the early compressive strength increases as well. Figure 4 shows that Mixture 6 cured at 65°C produced the highest compressive strength of 16.1MPa at 28 days. There is a significant increase in the compressive strength at 28 days for Mixture 6 compared to Mixture 10 and Mixture 11. The results shown in Figure 4 indicated that geopolymer mortar cured at curing temperature above 70°C resulted in a decrease in the compressive strength for 24 hours of curing. This results shows that curing temperature plays an important role in the geopolymerisation process of fly ash-based geopolymer mortar.

4.4 Ratio of Water-to-Geopolymer Solids, by Mass

Mixtures 1, 2, 6 and 7 were prepared to study the effects of water-to-Geopolymer solids ratio on the compressive strength of Geopolymer mortar. The total mass of water in the mixture is the sum of the mass of water in the sodium silicate solution, the mass of water in the sodium hydroxide solution and the mass of the extra water. The mass of Geopolymer solids is the sum of the mass of fly ash, the mass of sodium hydroxide solids and the mass of sodium silicate solids (mass of Na_2O and SiO_2 in sodium silicate solution).

Mixtures 1 and 7 have identical mixture composition, but different water-to-Geopolymer solids ratio of 0.273 and 0.333 respectively. The concentration of sodium hydroxide solution was held constant at 8M. The difference is the amount of extra water added to the mixtures. From Figure 5, it was observed that Mixture 1 with lower water-to-geopolymer solids ratio shows higher compressive strength compared to Mixture 7 with higher water-to-geopolymer solids ratio.

Mixtures 2 and 6 have identical mixture composition, but different water-to-geopolymer solids ratio of 0.264 and 0.324 respectively. The concentration of sodium hydroxide solution was held constant at 10M. The difference is the amount of extra water added to the mixtures. From Figure 5, it was observed that Mixture 2 with lower water-to-geopolymer solids ratio shows higher compressive strength compared to Mixture 6 with higher water-to-geopolymer solids ratio.

From Figure 5, Mixture 2 shows higher compressive strength than Mixture 1. Mixture 6 shows higher compressive strength than Mixture 7. This is mainly due to higher concentration of sodium hydroxide solution in Mixtures 2 and 6. As the water-to-Geopolymer solids ratio increases, the compressive strength of geopolymer mortar decreases. The trends of these results were similar to those observed by previous researches (Rangan et al 2006, Barbosa et al 1999,

Hardjito et al 2004). This trend is analogous to the well known effect of water-to-cement ratio on the compressive strength of Portland cement concrete, except that the chemical reaction involved is different.

4.5 Setting Times

The initial and final setting time of Geopolymer is important in practice because it establishes the time available for transport, placing and compaction of Geopolymer. Mixture 6 minus the fine aggregate was used to investigate the setting time of geopolymer paste. The activator-to-fly ash ratio was held constant at 0.35.

The test results on the setting time of geopolymer paste shows that the fresh fly ash based geopolymer mortar could be handled up to 120 minutes without any sign of setting for curing temperature ranging from 65 ^{o}C to 80 ^{o}C. Similar results were obtained by previous research conducted by Hardjito et. al (2004). Furthermore, it was observed that fresh fly ash-based geopolymer paste did not harden at room temperature for at least one day. From Figures 6 to 8, it is obvious that the geopolymer mortar setting time is much faster when curing temperature increases. This might be due to the increase in curing temperature, which will also increase the rate of chemical reaction.

The initial and final setting time of geopolymer paste at curing temperature of 65 ^{o}C was 230 minutes and 270 minutes respectively. The geopolymer paste needed 40 minutes to reach final setting time after the initial setting time. The initial and final setting time of geopolymer paste at curing temperature of 70 ^{o}C was 177 minutes and 240 minutes respectively. The geopolymer paste needed 63 minutes to reach final setting time after the initial setting time. The initial and final setting time of geopolymer mortar at curing temperature of 80 ^{o}C was 129 minutes and 165 minutes respectively. The geopolymer paste needed 36 minutes to reach final setting time after the initial setting time.

Wang and Cheng (2003) conducted a research to fabricate a coal fly ash based geopolymer for fire resistance purposes. They found that the initial and final setting time of geopolymer paste at curing temperature of 60 ^{o}C were 60 minutes and 65 minutes respectively. However, it should be noted that the source material used was metakaolinite which was prepared by calcining kaolinite at 750 ^{o}C for 6 hours following the methods proposed by Davidovits (1999). The different source of material yielded different test results compared to the current research (Van Jaarsveld et. al. 2003). Another research conducted by Cheng (2003) to produce a waste serpentine-based geopolymer for fire resistance purpose showed that the initial and final setting time at curing temperature of 60 ^{o}C was 79 minutes and 89 minutes respectively. In this research, the initial and final setting time of geopolymer mortar is longer than those reported by Wang and Cheng (2003) and Cheng (2003).

From the test results, it can be concluded that the higher the curing temperature, the higher the rate of geopolymerization process of geopolymer mortar, which eventually accelerates the hardening of Geopolymer mortar.

5. Conclusions

This paper presented the study of geopolymer mortar cured in elevated temperature. From the experimental results reported in this paper, the following conclusions are drawn:

a) Higher concentration of sodium hydroxide solution results in a higher compressive strength of geopolymer mortar.

b) The activator-to-fly ash ratio, by mass of 0.40 produced the highest compressive strength.

c) Curing temperature plays an important role in the geopolymerization process.

d) As the ratio of water-to-geopolymer solids by mass increases, the compressive strength of geopolymer mortar decreases.

e) The setting time of Geopolymer mortar due to different curing temperature has been determined. The initial setting time and final setting time ranged from 129 minutes to 270 minutes. The higher the curing temperature, less setting time is required.

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Table 1. Chemical Composition of Fly Ash as determined by XRF

Oxides	Mass (%)
SiO_2	59.9
Al_2O_3	24.7
Fe_2O_3	6.3
$SiO_2 + Al_2O_3 + Fe_2O_3$	90.8
CaO	2.0
MgO	1.9
TiO_2	1.0
K_2O	2.9
Na_2O	0.3
SO_3	0.1
Loss on Ignition (1000 ^{o}C)	0.3

Table 2. Details of mixtures

Mixture No.	Fine Aggregate	Fly Ash	Sodium Silicate Solution	Sodium Hydroxide Solution		Super-plasticiser	Added Water	Curing Temperature	Curing Time
	(kg/m³)	(kg/m³)	(kg/m³)	Mass kg/m³	Concentration	(kg/m³)	(kg/m³)	(°C)	Hours
1	1173	909	228	90	8M		89	65	24
2	1173	909	228	90	10M		89	65	24
3	1173	909	228	90	16M		151	65	24
4	1173	909	228	90	14M		151	65	24
5	1173	909	228	90	12M		151	65	24
6	1173	909	228	90	10M		151	65	24
7	1173	909	228	90	8M		151	65	24
8	1128	909	261	103	10M		151	65	24
9	1082	909	293	116	10M		151	65	24
10	1173	909	228	90	10M		151	70	24
11	1173	909	228	90	10M		151	80	24
12	1173	909	228	90	10M	6.2	151	65	24
13	1173	909	228	90	16M		151	65	48
14	1173	909	228	90	10M		151	60	24
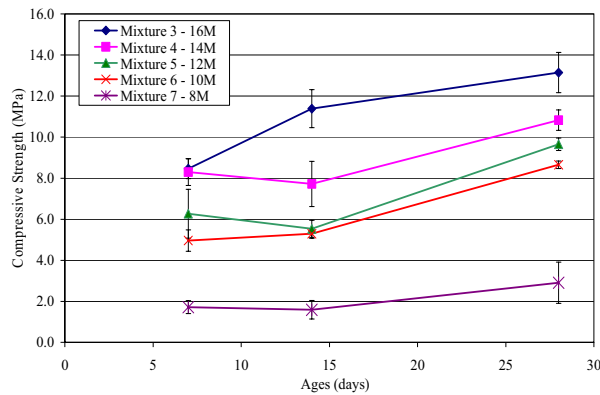
Figure 1. Effect of Concentration of Alkaline Activator on Compressive Strength
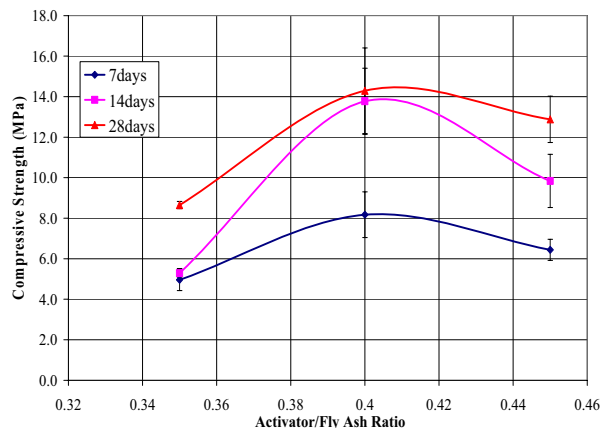

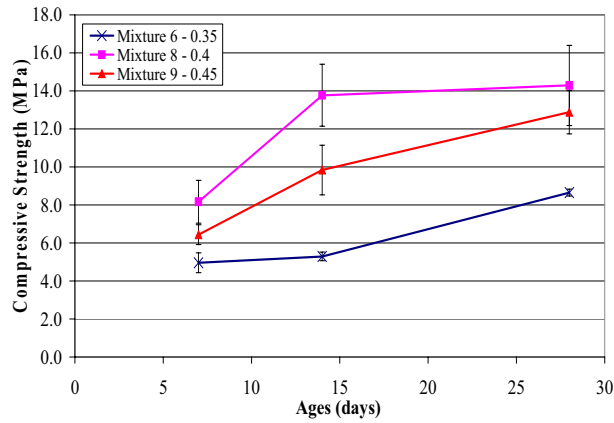Figure 2. Optimum Activator-to-Fly Ash Ratio

Figure 3. Effect of Activator-to-Fly Ash Ratio on Compressive Strength
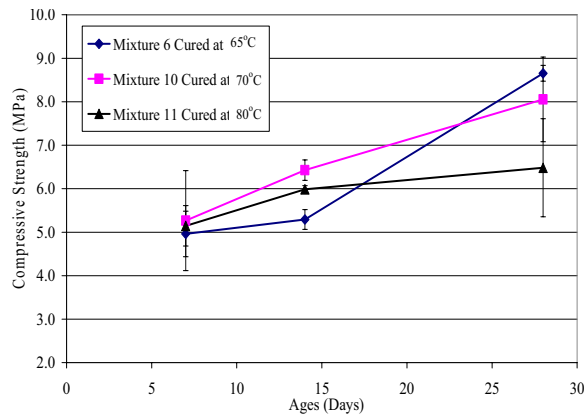

Figure 4. Effect of Curing Temperature on Compressive Strength
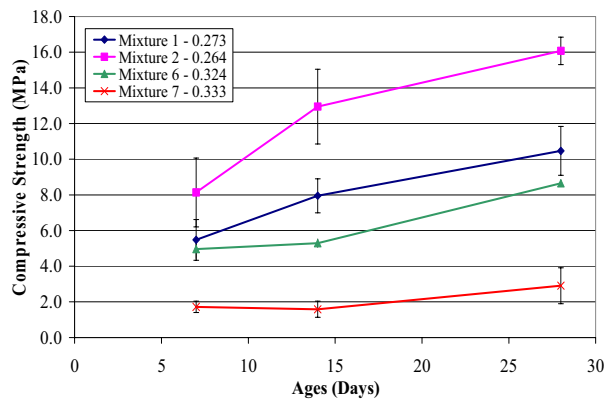

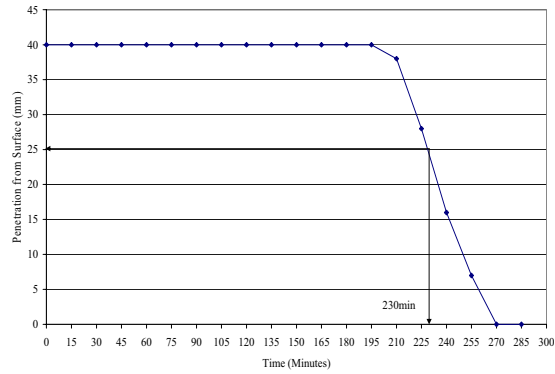Figure 5. Effect of Water-to-Geopolymer Solids Ratio on Compressive Strength

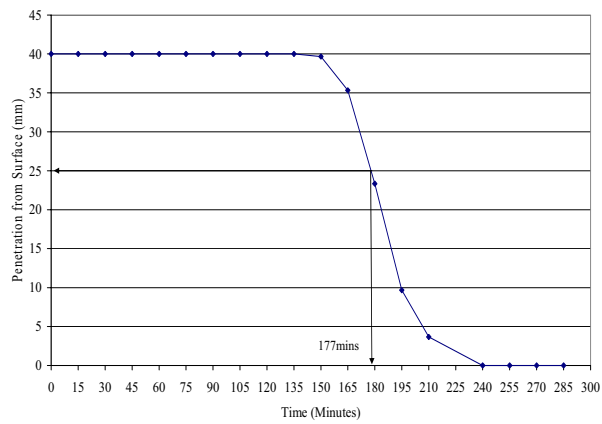Figure 6. Initial and Final Setting Time of Geopolymer Paste at Curing Temperature of 65oC

Figure 7. Initial and Final Setting Time of Geopolymer Paste at Curing Temperature of 70oC
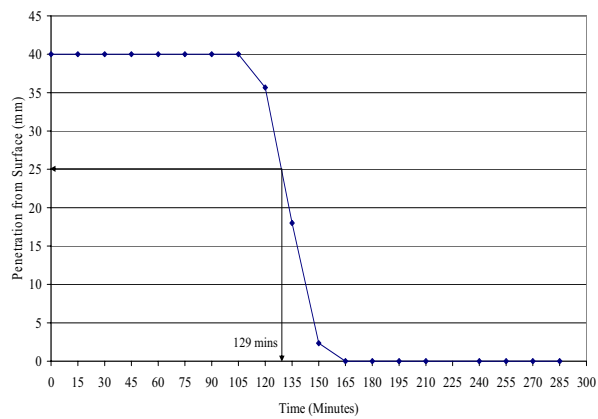

Figure 8. Initial and Final Setting Time of Geopolymer Paste at Curing Temperature of 80oC