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STUDY ON THE CONSISTENCY AND SETTING CHARACTERISTICS OF FLYASH BASED GEOPOLYMER

Manimaran, E [*1] and Mohankumar, G.[2]
[*1]Research Scholar, PRIST University, Thanjavur
[2]Professor, Department of Civil Engineering, ARUNAI Engineering College, Tiruvannamalai- 606 603

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ABSTRACT
Production of Portland cement is consuming significant amount of natural resources and emitting considerable fraction of the totally generated carbon dioxide to the atmosphere. Research made on making concrete without cement has come to a viable level of developing geopolymer concrete. Considerable research has been carried out to study the compressive strength of geopolymer concrete with different mixing proportion and test variables but, there is not much data available on the short term engineering properties like consistency and setting characteristics even though it establishes the time available for placing, compaction and transport of Geopolymer. For a wide range of applications and acceptance of GPC in concrete industry, much more studies on the binding property of geopolymers are needed apart from other properties of geopolymer mortar and geopolymer concrete. The dosages of activating solution, the curing temperature and curing period are yet to be optimized for the requirements. A fundamental study on the setting characteristics of low calcium flyash based geopolymer with molar concentration of Sodium hydroxide 8-14M and liquid ratios of sodium silicate to sodium hydroxide as 1.5-3.0 and curing temperature of 65oC is made and the informative results are presented.

INTRODUCTION
Concrete is the most versatile building material and no doubt there is no substitute for conventional cement concrete. Therefore, the demand of concrete is increasing day by day but, the environmental aspects of concrete are now being discussed with a view to develop an eco-friendly material of construction. As the emission of CO_2 and the exploitation of natural resources in producing cement and concrete, lead to environmental degradation, there is a need to find an alternative to cement binder. Cement being an important ingredient of the conventional concrete and responsible for binding property cannot be easily ignored. However, in 1980s, Joseph Davidovits, a French scientist initiated research on linear organic polymers and later the alumino-silicate solid state chemistry resulting in the development of new concrete by polymerization called geopolymer concrete (GPC).

Geopolymer is an inorganic alumino-silicate compound, synthesized from source materials like calcined clay, kaolinitic clay, lateritic clay, volcanic rocks, mine tailings and industrial wastes or byproducts like flyash, silica fume, ground granulated blast furnace slag, rice husk ash, red mud or metakaolin. Joseph Davidovits (2011) has explained the geopolymers based on, kaolinite/hydrosodalite, metakaolin MK-750, Calcium, Rock, Silica, flyash, Phosphate and organic minerals and also reported the development of GPC from these source materials as the attempts of scientists from various origins. Of all these materials, flyash is available abundantly in India, but to date its utilization is limited inspite of the recommendations of IS: 3812-1981.

Anjan Chatterjee (2010) addressed the issues pertaining to the intrinsic quality parameters of the Indian flyashes, potential of chemical activation, feasibility of adopting the newer milling systems such as vibration and attrition milling, etc., By 2010 survey, 120 million tons of flyash has been found generated annually in India, with 75000 acres of land being occupied by ash ponds leading to the environmental hazards. Hence, it was felt that the management of flyash is the urgent and challenging task. The flyash generation in India touched 170 MT by 2011-12. Therefore, utilization of flyash other than high volume flyash concrete is a felt need. Flyash rich in silica and alumina reacts with alkaline solution and forms aluminosilicate gel that acts as the binding material and by thermal activation produces GPC.

The blend of sodium hydroxide (or potassium hydroxide) solution and sodium silicate (or potassium silicate) solution is termed as alkaline solution. The source material is mixed with an activating solution that provides the

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alkalinity needed to liberate the Si and Al and possibly with the additional source of silica (sodium silicate). The various parameters that influence include, ratio of alkaline liquid-to-flyash (by mass), concentration of sodium hydroxide solution, ratio of sodium silicate solution-to-sodium hydroxide solution, water content of mixture, dosage of super plasticiser, mixing time, rest period prior to curing, handling time, curing temperature, curing time, curing method and age of concrete were studied. Most of the reports specifically aimed for development of GPC for strength. Consistency of geopolymer plays a major role in the workability based on the additional water requirements and also setting properties will be useful for assigning the mixing time and rest period. However, only limited studies were reported on these fundamental properties of Geopolymer.

Joseph Davidovits (1994) reported that, Geopolymer cement hardens rapidly at room temperature and provides compressive strength of 20MPa after only 4 hours at 20^{o}C and the 28 day strength in the range of 70-100MPa. Djwantoro et al (2008) studied the influence of various parameters on the short term properties of fresh low calcium flyash based Geopolymer mortar. Sodium hydroxide/ sodium silicate as alkaline activator (8-16M) with constant ratios of activator/flyash (0.35) and water/geopolymer solids (0.324), oven curing at temperature ranging from 65 to 80^{o}C for 24 hours and air curing at room temperature until the day of testing, it is reported that the initial setting and final setting time ranged from 129 minutes to 270 minutes and observed that higher the curing temperature, lesser the setting time.

Mallikarjuna and Gunneswara (2015) investigated the normal consistency and setting time of geopolymer. The parameters considered in this investigation were geopolymer source material (flyash and GGBS) and alkaline activator consisting of sodium meta silicate and sodium hydroxide of different molarities (8, 12, 16 M) with constant ratio of 2.5. The test results indicated that combination of fly ash and GGBS resulted in decreased final setting time and increased compressive strength.

Yellaiah et al, (2014) investigated the consistency and setting times of low calcium flyash based geopolymer cement under varied heat curing. It is observed that the consistency (28% of alkaline activator solution) of the geopolymer did not show any variation when mixed with different combinations of alkaline activator solution; whereas, the setting times are dependent on the concentration of NaOH, alkaline liquid ratio and variation in temperature as a whole.

EXPERIMENTAL INVESTIGATION

Low calcium flyash (ASTM Class F) from the Mettur Thermal power station is used as the source material. The chemical composition of the flyash is shown in Table 1. A combination of sodium hydroxide 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 based solution was used because of its wide availability and less expensive than potassium based solution.

Table 1. Chemical Composition of Class F flyash

No	Elements	Mass %
1	SiO_2	54.40
2	Al_2O_3	25.64
3	Fe_2O_3	11.32
4	CaO	02.03
5	Na_2O	00.41
6	K_2O	00.73
7	TiO_2	01.53
8	MgO	00.92
9	SO_3	01.70
10	LOI	01.32

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Consistency of Geopolymer

The consistency of the flyash based geopolymer paste is determined by the procedure followed as per IS: 4031(Part-4) -1988, Standard for Portland cement paste using Vicat's apparatus, duly modified by replacing the cement and water with flyash and alkaline solution respectively. Preparing a trial paste with alkaline solution and testing with varying percentage is done until the amount of alkaline solution necessary for making up the standard consistency is obtained. This procedure is repeated for varied molarities (8-14M) and liquid ratios 1.5 to 3.0. The variations in the depth of penetration are presented in Table 2 and the standard consistency for various molar concentrations is presented in figure 1.

Initial Setting time

The fluid binder is taken as 85% of Normal consistency. The flyash and activator solution are placed in the mixer bowl and mixed thoroughly for five minutes. The Geopolymer paste is cast into the standard mould of the Vicat's apparatus in two layers with proper tamping and finishing. The specimen is placed into the oven for curing at an elevated temperature of 65^{o}C. For every 15 minutes interval initially and latter ten minutes, the specimen is placed on the Vicat's apparatus to measure the initial setting time. To begin with, the centre of the specimen is placed under the end of the Vicat's needle (1mm) and the movable rod is lowered until the 1mm needle end makes contact with the Geopolymer paste. The indicator is set to zero and the movable rod is allowed to freely fall and the penetration of the needle is recorded. The specimen is then taken back to the oven for curing continuity at the same elevated temperature.

Table 2. Consistency of Geopolymer for various Molarities and liquid ratios

No	Molarity and Liquid ratio		Depth of plunger penetration(mm) for various % of alkaline solution of varying liquid ratios								
			24%	25%	26%	27%	28%	29%	30%	31%	32%
1	8M	1.5	0	0	3	9	18	25	34	-	-
2	10M		0	0	3	8	16	22	29	34	-
3	12M		0	0	3	7	14	21	27	31	34
4	14M		0	0	2	5	12	19	25	30	36
5	8M	2.0	0	0	2	6	16	28	35	-	-
6	10M		0	0	2	8	14	20	28	35	-
7	12M		0	0	3	9	14	21	27	32	35
8	14M		0	0	2	8	13	20	24	32	36
9	8M	2.5	0	0	3	7	14	29	36	-	-
10	10M		0	0	3	9	15	20	28	35	-
11	12M		0	0	3	10	14	21	27	32	35
12	14M		0	0	2	8	13	20	25	31	35
13	8M	3.0	0	0	3	8	12	28	36	-	-
14	10M		0	0	2	8	15	22	28	36	-
15	12M		0	0	3	9	15	22	27	31	36
16	14M		0	0	2	8	15	21	24	30	35
17	Cement		0	2	5	14	25	37	-	-	-

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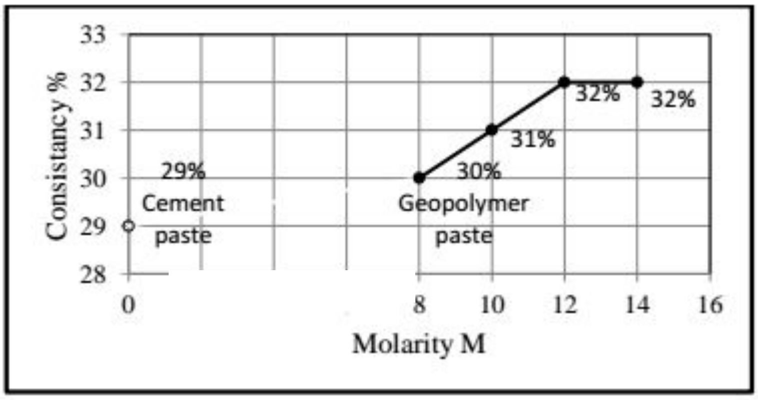
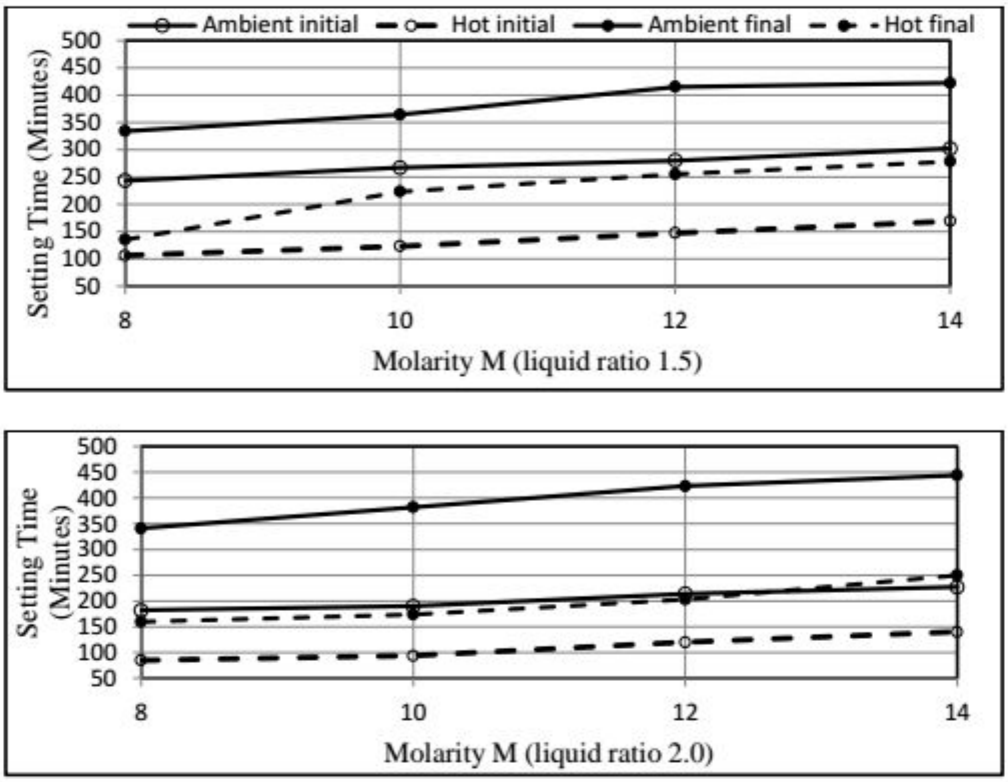


Fig. 1 Consistency of Geopolymer paste for various Molarities

To begin with, the centre of the specimen is placed under the end of the Vicat's needle (1mm) and the movable rod is lowered until the 1mm needle end makes contact with the Geopolymer paste. The indicator is set to zero and the movable rod is allowed to freely fall and the penetration of the needle is recorded. The specimen is then taken back to the oven for curing continuity at the same elevated temperature. These procedures are repeated and the penetration at interval is recorded until a penetration of 33-35mm is experienced. This time is recorded as 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. The variations in the initial and final setting time are presented in figure 2.



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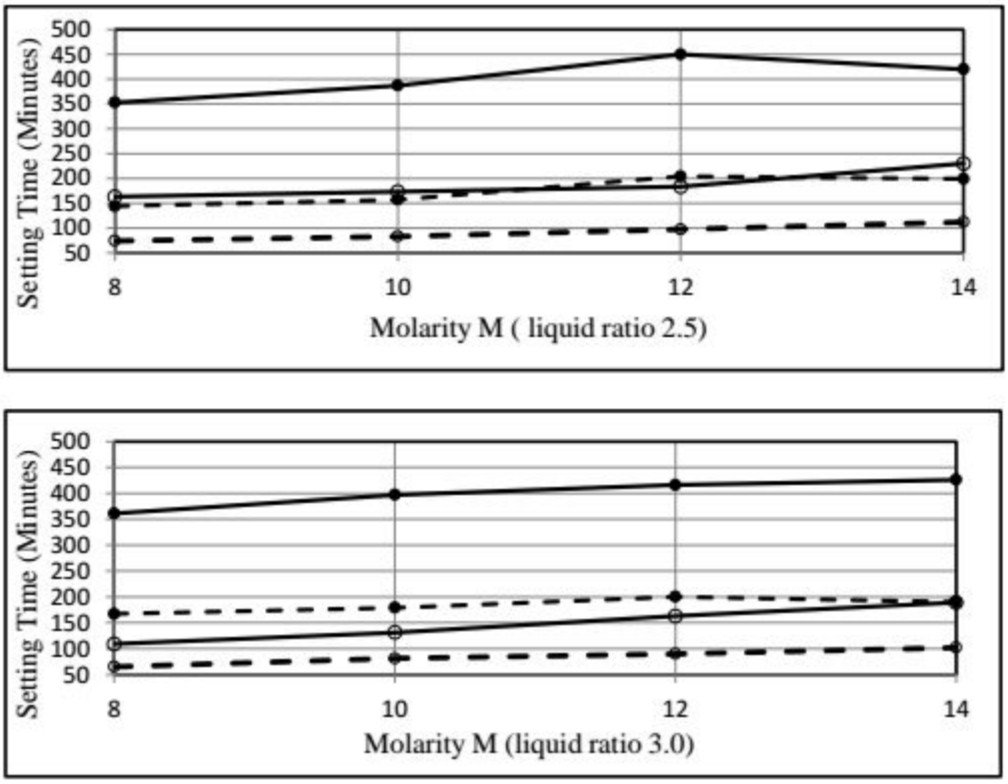

Fig.2 Setting time for varying molarities, liquid ratio and curing temperature

DISCUSSION OF TEST RESULTS

The standard consistency of flyash paste is obtained as 29% and the consistency of flyash based geopolymer paste increased for the increase in molar concentration upto 14 M irrespective of the liquid ratios. The consistency for 8M paste is obtained as 30%, for10M paste as 31% and for both 12M and 14M pastes as 32%. It is observed that the consistency is not at all influenced by the liquid ratios considered. This reveals that higher molar concentration only can demand for extra water requirement irrespective of the liquid ratios.

The matter of setting time is different from the consistency. By ambient curing the initial setting time increases gradually as the molarity increases for all liquid ratios but, decreases for increase of the liquid ratio 1.5 to 3.0. On the other hand, the final setting time increases as the molarity increases and also increases but not significantly for increase in the liquid ratio 1.5 to 3.0.

For hot curing at 65°C, the initial setting time and final setting time in general increase for increase in molarity for all liquid ratios. In particular the rate of increase is comparable and slightly high for initial setting time than final setting time for liquid ratios 1.5 – 2.5. But for liquid ratio of 3.0, the rate of increase of both initial and final setting time is almost same.

The initial setting time of hot cured Geopolymer having liquid ratio 1.5 and molar ratios 8M to 14M is respectively 44% to 56% that of ambient curing. For liquid ratios 2.0, 2.5 and 3.0 the initial setting time of hot cured Geopolymer are respectively, 47%-62%, 45%-49% and 60%-54%. On the other hand the final setting time of hot cured Geopolymer having liquid ratios 1.5, 2.0, 2.5 and 3.0 for molar ratios 8M to 14M are respectively 40%-66%, 47%-56%, 41%-45% and 46%-48% that of ambient curing.

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CONCLUSION

1. The standard consistency of flyash paste is less than that of geopolymer paste.
2. The standard consistency of flyash based geopolymer increases with increase of molar concentration.
3. The consistency is not at all influenced by the liquid ratios considered. Therefore, higher molar concentration can demand for extra water requirement irrespective of the liquid ratios.
4. In case of ambient curing the initial setting time increases gradually as the molarity increases for all liquid ratios but, decreases for increase of the liquid ratio 1.5 to 3.0.
5. The final setting time increases as the molarity increases and also increases but not significantly for increase in the liquid ratios (1.5 to 3.0).
6. For hot curing at 65°C the initial setting time and final setting time in general increases for increase in molarity for all liquid ratios (1.5 – 3.0).
7. In particular the rate of increase is comparable and slightly high for initial setting time than final setting time for liquid ratios 1.5 – 2.5. But for liquid ratio of 3.0, the rate of increase of both initial and final setting time is almost same.
8. The initial setting time of hot cured Geopolymer having liquid ratio 1.5 -3.0 and molar ratios 8M to 14M 44% to 62% that of ambient curing.
9. The final setting time of hot cured Geopolymer having liquid ratios 1.5, 2.0, 2.5 and 3.0 for molar ratios 8M to 14M are 40%-60% that of ambient curing.

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