Compressive Strength of High Plastic Clay stabilized with Fly Ash Based Geopolymer and Its synthesis Parameters

Neeraj Varma[1], Tulasi Kumar[2] and Vamsi Nagaraju[3]

[1] Research Scholar, Department of Civil Engineering, NIT-Rourkela, Rourkela, Odisha, India.
[2]Research Scholar, Department of Civil Engineering, IIT-Tirupati, Andhra Pradesh, India
[3] Research Scholar, Department of Civil Engineering, NIT-Surathkal, Karnataka, India
dnvarma02@gmail.com

Abstract. Geopolymerization is an effective technique for utilizing industrial solid waste material as stabilizing material. This paper studies the effect of class-F fly ash based geopolymer on compressible strength characteristics of high plastic clay using unconfined compression strength (UCS) test. Sodium silicate and sodium hydroxide were used as alkali activators in proportions of 60:40 respectively. The fly ash content was varied by 0%, 10%, 20% and 30% by dry weight of soil and alkali activator was varied by 5%, 10% and 15% by dry weight of soil-fly ash mix. UCS tests were carried out on the specimens contaminated under controlled curing environment. Unconfined compression strength increased with increase in fly ash and liquid activator content. The maximum UCS value of 790 kPa observed at 30% of fly ash content under elevated temperature of 50º C. The influence of Si/Al and Na/Al ratios on compressive strength of geopolymeric materials was also identified. Further, numerical analysis was carried out to check the significance of factors effecting the compressive strength of the material.

Keywords: Geopolymerization, Fly ash, Liquid activator, Curing period, UCS.

1 Introduction

High plastic clay is one of the complex soil materials spread across various parts of the world. The usage of such soil as foundation material is limited because of its low strength properties. Indeed, the performance of high plastic clay is erratic in the presence of moisture [1]. Various stabilization techniques such as cement stabilization, lime stabilization with well-understood mechanism are widely practiced to improve the strength of soils with inadequate engineering properties [2] [3]. The usage of cementitious products always left with carbon footprints, which is one of the major environmental concerns. Approximately one ton of CO_2 emitted from one ton of cement production [4-5]. Therefore, various researches are working on new soil stabilizer, which is viable.

Geopolymer is an environmental friendly, amorphous 3-dimentional alumina-silicate binder material consists of a chain structure. Aluminum (Al), silicon (Si) ions and a member of the family of inorganic polymers are the key elements in the formation of chain structure [6]. Any material is a possible source for the production of geopolymer if it contains rich amount of silica (SiO_2) and alumina (Al_2O_3) in amorphous state [7]. Hence, geopolymers can be synthesized from the low cost industrial wastes such as fly ash, ground granulated blast furnace slag, rice husk ash, etc., [8]. Under alkali environment, the inactive Si and Al leached out of the source material and tetrahedral silica $(SiO_4)^{-4}$ and alumina $(AlO_4)^{-5}$ component are formed with ionic deficiency. Hence, with the sharing of oxygen atom between multiple Si and Al tetrahedral units, polymeric chained structures are formed and poly-condensation takes place [9]. Geopolymers have tremendous compressive strength along with excellent resistance towards acids and organic solvents [10].

Fly ash is the by-product obtained from the combustion process of coal that rises with flue gases. It has fine spherical glassy structure, rich in silica and alumina components in amorphous state. As per 2018-19, the fly ash production in India was raised to 217.03 million tons [11]. Although, fly ash was utilized in various industries, still 48.64 million tons per year was left as waste material in landfill and ash ponds, which create serious environmental problems. Due to its abundant availability with useful chemical composition, fly ash can be effectively synthesized into geopolymeric material [12].

Most of the industrial waste material behaves inert under normal environmental conditions. Hence, alkali environment is essential to propagate the geopolymerization. Various alkali material such as sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium silicate (Na_2SiO_3) etc., are used for prompting alkali environment. The effect of NaOH in geopolymerization is comparatively higher than KOH. Due to smaller atomic radius, mobilization of sodium atom is easily through the pores of polymeric material during the initial stages [13]. The combination of sodium silicate and sodium hydroxide is more effective in producing a geopolymeric material with superior compressive strength [13]. Most of the researchers observed that behavior of geopolymer material not only depends upon the type of alkali liquid but also on the concentration, amount of alumina and silica content etc., [14-18].

In the present study, Na_2SiO_3 and NaOH were used for preparing alkaline environment. The ratio of Na_2SiO_3 and NaOH fixed as 60:40. Soil specimens were prepared by mixing different percentages of liquid activator with varying percentages of soil-fly ash mix. The effect of curing period and curing temperature on compressive strength characteristics of high plastic clay were determined. Thereafter, statistical analysis was conducted to determine the influence of Si/Al ratio and Na/Al ratios on compression strength of geopolymeric material.

2 Materials and methods

2.1 Collected Soil

For the present study, soil sample collected at a depth of 1.5 m from Guntur, India. About 99% of the soil particles (by weight) passed through 75 micron IS sieve. The soil consists of mostly clay particles, which are soft in nature and not suitable as a foundation material. Hence, the soil best suits for geopolymerization. Soil has liquid limit of 84.5 % and plasticity index of 58 %. As per IS Soil Classification System, the soil sample was classified as CH (Inorganic Clay with High Plasticity) with high degree of swelling. Various index and engineering properties of collected soil sample were illustrated in the Table-1.

2.2 Fly ash

Fly ash collected from VTPS, Vijayawada is used as a precursor in geopolymerization. The chemical composition of fly ash was illustrated in Table-2. As CaO component is 0.22 less than 10%, the given fly ash was classified as class-F as per ASTM C618. Prior to the geopolymerization, soil mixed with different percentages of fly ash (FA), such as 10%, 20% and 30% by dry weight of soil. IS light compaction test was performed as per IS 2720 Part 7, 1980 to determine the compaction characteristics of soil fly ash mix [19]. The optimum moisture content (OMC) and maximum dry density (MDD) of clayey soil with varying proportions of fly ash were presented in Table-3. These compaction characteristics were used in preparing soil specimens and subjected to unconfined compression strength (UCS) test as per IS 2720 Part 10, 1991 [20].

2.3 Preparation of liquid activator (L)

In the present study, the liquid activator (L) is prepared from commercially available sodium silicate solution (Na_2SiO_3) and sodium hydroxide (NaOH). Na_2SiO_3 contains molecular weight of 284.20 g/mole and specific gravity of 1.5. While the sodium hydroxide (NaOH) originally in the form of pellets with a molecular weight of 40 g/mole and specific gravity of 2.13 at 20º C with 95-99% purity. Most of the research work revealed that the 8 to 10 M concentration of NaOH was effective in dissolution of Si and Al [21-22]. Hence, 8 M NaOH solution was fixed for synthesis of geopolymer. Initially 8 M NaOH solution was prepared by mixing pellets with required amount of distilled water. Then after, readily available Na_2SiO_3 mixed with NaOH solution at a ratio of 60:40 to prepare liquid alkaline activator. The liquid activator was prepared one day prior to the preparation of soil specimens.

2.4 Specimen preparation

For studying the effect of geopolymerization on compressive strength characteristics of soil-fly ash mix, re-molded cylindrical specimens of 38 mm diameter and 76 mm height were prepared. Initially, oven dried soil sample was mixed with varying percentages of fly ash (such as 10%, 20% and 30% by dry weight of soil). Following the homogeneous soil-fly ash mix, liquid activator added in different proportions (such as 5%, 10% and 15% by dry weight of soil-fly ash mix) to varying percentages of soil-fly ash mix. For ensuring a uniform mix, the components thoroughly mixed for 5 minutes. Thereafter, specimens were prepared using standard cylindrical molds. After taking out, the samples were wrapped in polythene covers to avoid the fluctuations in moisture content. About 190 specimens were prepared for the present study. For maintaining accuracy, three specimens were tested for each mix and the average of three was recorded. The Si/Al and Na/Al ratios for different mixes are present in Table-4.

As chemical reactions are involved in geopolymerization, curing time will play a crucial role in improving the strength of soil [23]. The soil specimens were stored in desiccators at varying curing periods of 3, 7, 21 and 28 days at a room temperature of 27^o C. Further, the effect of curing temperature was also determined by placing the cylindrical specimens in electric oven at 50^o C for 1 day, 3 days and 7 days.

Table-1. Index and engineering of collected soil sample

SR. No	Properties	Value
1	Grain Size analysis	
	a) Gravel Content	0%
	b) Sand Content	1%
	c) Fines Content	99%
2	Plasticity characteristics	
	a) Liquid limit	84.5%
	b) Plastic limit	26.5%
	c) Plasticity Index	58%
3	IS Soil Classification	CH
4	Specific gravity	2.46
5	Differential free swell index	75%
6	Compaction characteristic	
	a) Maximum Dry Density	14.6 kN/m^3
	b) Optimum Moisture Content	23%
7	Unconfined Compression Strength	121.6 kN/m^2

Table-2. Chemical composition of fly ash

Chemical composition	Content (%)
SiO_2	59.04
Al_2O_3	34.08
Fe_2O_3	2.0
CaO	0.22
SO_3	0.05

MgO	0.43
Na_2O	0.5
K_2O	0.76
LOI	0.63

Table-3. Compaction Characteristics of soil-fly ash mix

FA (%)	MDD (kN/m^3)	OMC (%)
0	14.6	23%
10	14.47	24%
20	14.35	26%
30	14.3	27.50%

Table-4. UCS values for different mix proportions at 27° C and 50° C

FA (%)	L (%)	Na/Al	Si/Al	UCS (kPa) at 27° C					UCS value (kPa) at 50° C		
				0 days	3 days	7 days	14 days	28 days	1 day	3 days	7 days
10	5	0.92	1.84	154	155	159	176	188	185	201	311
	10	1.84	2.21	156	160	239	245	282	271	291	346
	15	2.76	2.58	157	181	276	290	369	461	511	644
20	5	0.46	1.66	155	166	190	212	230	189	216	341
	10	0.92	1.84	157	184	251	272	394	282	315	457
	15	1.38	2.02	162	210	300	328	469	679	708	760
30	5	0.31	1.60	158	168	194	227	248	202	230	373
	10	0.61	1.72	161	224	288	352	450	440	521	701
	15	0.92	1.84	165	230	308	363	650	691	731	791

2.5 Statistical Analysis

Multiple liner-regression analysis was carried out based on the results obtained from UCS test by employing regression analysis in excel data. FA and L percentages were considered as independent variables and corresponding UCS values as output response. The significance of variables are analyzed with 95% confidence by considering p-value as 0.05.

3 Result and Discussions

3.1 Curing period

In the process of ascertaining the compressive strength of geopolymerized soil fly ash mix, UCS tests were conducted on soil specimens. After completing the respective curing periods at 27° C, the specimens taken out and left isolated for 2 hours and tested under UCS testing machine. The test results were presented in Table-4. For varying contents of fly ash (FA), the UCS values of high plastic clay increased with increase in liquid activator (L) content. It is observed that immediately after the sample preparation (at zero days curing period), not much variation observed in UCS values. All the mixed proportions carried out similar set of values and the same was presented graphically in Figure-1. Therefore, feasible time elapse is required for leaching of Si and Al components and for effective poly-condensation. With increase in curing period, the USC values increased and the maximum value observed at 28 days curing period. Similar trend in strength was also reported by Cristelo et al., 2013 [23]. The increase in UCS values is predominantly due to poly-condensation of Si and Al monomers during curing period [24]. At 5% of liquid activator (L), the enhancement in UCS values were insignificant. The available liquid activator was not sufficient for dissolution of required amount of Si and Al from FA. This may leads to the development of poor geopolymer network through soil pores which can be ruined under compressive load. For 10% and 15% liquid activator, the increased values of UCS indicated that alkaline activator is sufficient for geopolymerization. Yaghoubi et al. 2019, [25] reported that the particular amount of liquid activator was necessary for dissolution of Si and Al from the precursors. When sufficient amount of activator was available, higher dissolution of Si and Al takes place and helps in the developing a strong geopolymer network. Hence, with increase in fly ash content, the amount of liquid activator required will also increases. Maximum UCS value observed at a mix proportion of 30% FA and 15% L.

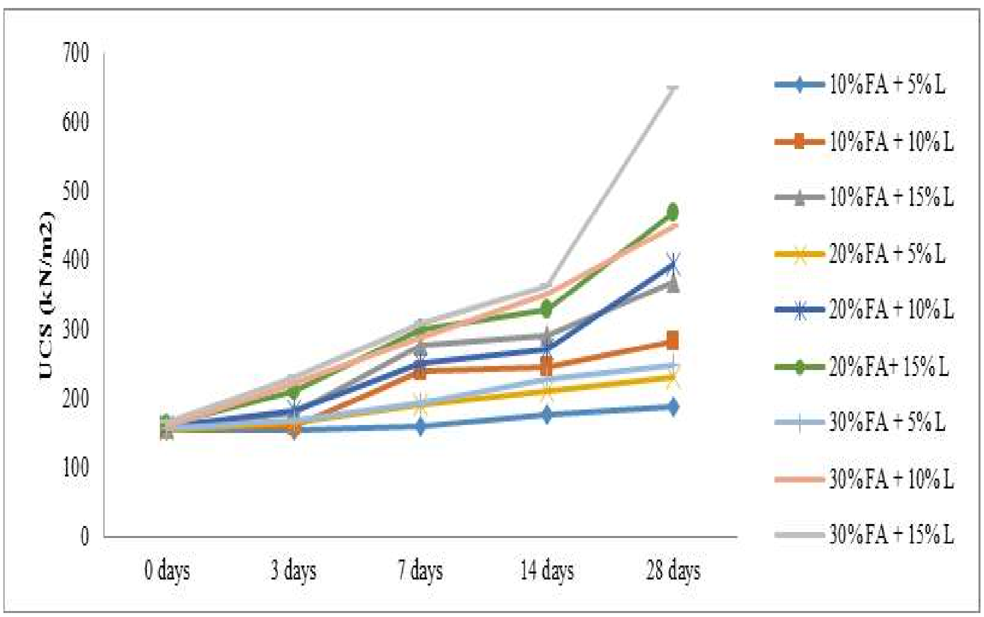

Fig.1. UCS values for varying curing periods at 27° C

3.2 Curing temperature

The effect of curing temperature on geopolymerized soil-fly ash mix was ascertained by curing the cylindrical soil specimens at 50º C for 1, 3 and 7 days. The UCS values of cylindrical specimens at 50º C for varying curing periods are presented in Table-4. With increases in the curing temperature, the enhancement in geopolymerization reaction takes place and enhanced the strength of geopolymer stabilized soil [15]. The trend in increase of UCS values for varying curing periods were shown in Figure-2. The influence of curing temperature on different mix proportions was clearly observed. At 7 days curing period, slight cracks were developed on the surface of cylindrical specimens for a mix proportion of 30% FA and 5% L. At elevated temperature with less activator content, evaporation of water from surface of soil specimen takes place. Due to the difference in water content on the surface and inside of specimen, temperature stresses may developed on the surface of the specimen. Hence, under the combination of temperature stresses and poor geopolymeric network at lesser alkali content, cracks on the surface of material was developed.

Bar charts shown in the Figure 3 and 4 outlined the impact of curing temperature on geopolymerized soil fly ash mix. For the mix proportion of 30% FA+15% L, at 7 days curing period, the UCS value at 50º C increased by 2.56 times when compared with specimens at 27º C curing temperature. Further, UCS value of soil specimen (with 30% FA+15% L) cured for 7 days at 50º C increased by 1.21 times when compared with soil specimen cured for 28 days at 27º C. As a result, it is experiential that curing temperature has an effective mark on compressible strength characteristics of geopolymer stabilized soil.

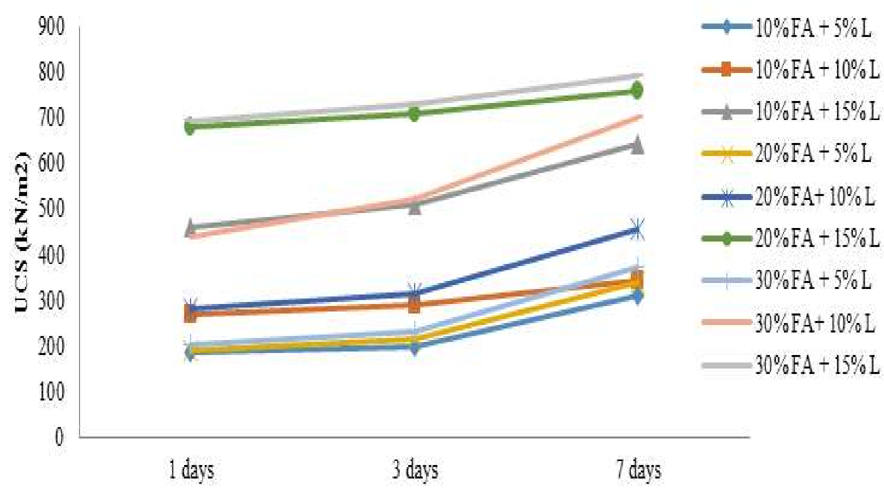

Fig.2. UCS values for varying curing periods at 50º C

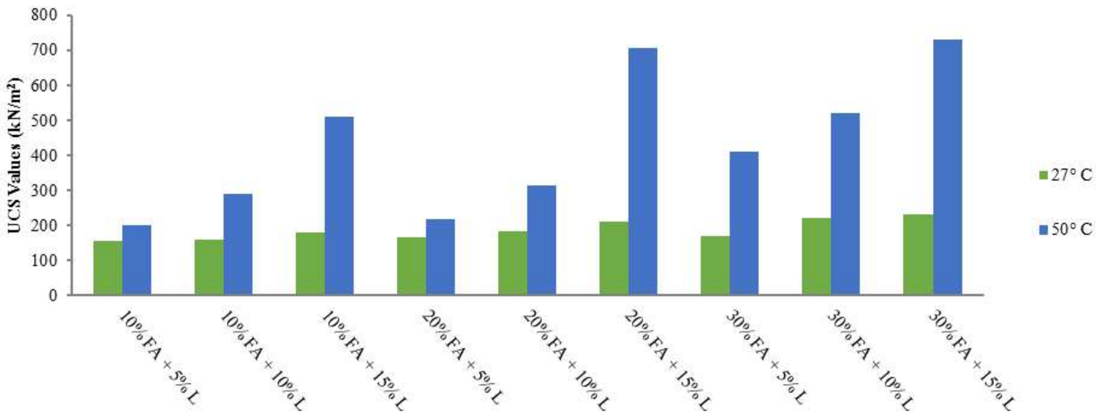

Fig.3. Variation of UCS values for 3 days curing period at 27° C and 50° C

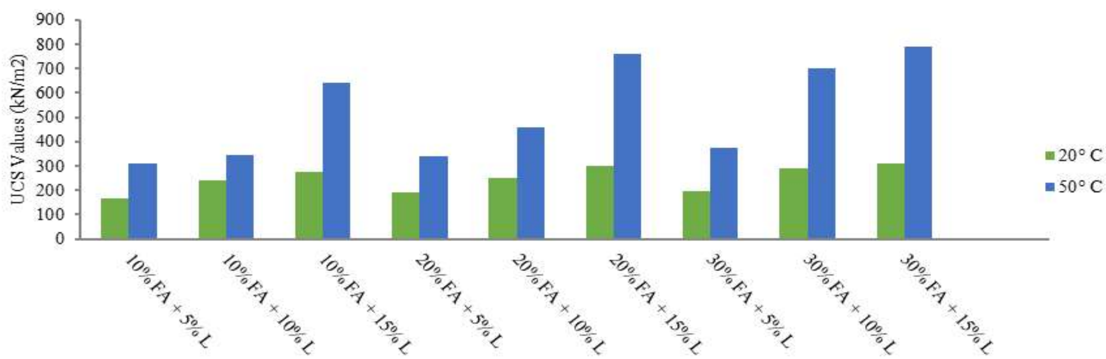

Fig.4. Variation of UCS values for 7 days curing period at 27° C and 50° C

3.3 Effect of Si/Al and Na/Al ratios

From the test results illustrated in Table-4, for each FA percentage with increase in L%, the molar ratios of Si/Al and Na/Al increased. The increase in Si/Al ratio is due to the availability of more silica from Na_2SiO_3 and Na/Al ratio increase due to the availability of more sodium from NaOH and Na_2SiO_3. However, with increase in FA content, the Si/Al and Na/Al molar ratio decreased due to the increase of alumina content. When UCS values were compared with Si/Al and Na/Al ratio, it was observed that UCS value increased with increase in both Si/Al and Na/Al ratio. The maximum UCS value was observed at Si/Al ratio of 1.84. Binod Singhi et al., [26], also reported that maximum UCS values were observed for Si/Al ratio greater than one. Similarly, for Na/Al ratio, the maximum UCS values were observed at 0.92, which was in the range of 0.75 to 1.25 as reported by Xu and Deventer [27]. Although, Si/Al and Na/Al ratios shows some effect on UCS values, the level of significance is very less when compared with FA % and L %.

3.4 Statistical Analysis

By using the UCS test data, multi-linear regression was carried out by considering the FA and L values as independent variables for varying curing periods of 27° C and 50° C separately. ANOVA test was used to identify the extent of linear interaction between variables. The statistical significance of variables were determined by using p-value obtained from the ANOVA table. The variables are statistically significant to the output value for 95% confidence level, when the p-value is less than 0.05. The results of statistical analysis were illustrated in Table-5 and 6.

From multi-linear regression analysis data, the predictive model is significant with least standard errors for 0, 3 and 7 days curing period at 27° C. However, with increase in curing periods (such as 14 and 28 days) and curing temperatures (1, 3 and 7 days at 50° C), the predictive model was insignificant due to the non-linear relationship between the independent variables and output response. Hence, linear regression analysis can be used for predicting models up to 7 days curing periods under ambient curing temperature. For curing at longer durations and other temperatures non-linear analysis may effectively use in the predicting models as proposed by Ruhel [28].

Table 5. Coefficient values for regression analysis

Coefficients	At 27° C					At 50° C		
	0 Day	3 Day	7 Day	14 Day	28 Day	1 Day	3 Day	7 Day
X_o	147.110	99.089	86.312	111.987	-17.116	-179.376	-180.480	-53.392
X_1	0.287	2.073	2.176	0.520	6.085	6.951	7.997	9.410
X_2	0.501	4.629	11.373	17.522	24.400	41.821	43.427	38.995

Table 6. ANOVA test result

Response Model	At 27° C					At 50° C		
	0 Day	3 Day	7 Day	14 Day	28 Day	1 Day	3 Day	7 Day
Standard error	1.390	12.400	13.187	48.052	33.553	73.857	70.472	70.142
R^2	0.8430	0.8167	0.9402	0.6924	0.9238	0.8653	0.8868	0.8733
F-value	22.4804	18.8334	63.9487	10.00	49.525	26.704	32.342	28.579
Significance of F	0.0016	0.0025	9×10^{-5}	0.0122	0.00018	0.00103	0.00061	0.00085
P-value								
X_o	0.448×10^{-10}	0.00055	0.001593	0.1005	0.6860	0.0898	0.0771	0.5498
X_1	0.00234	0.00639	0.006796	0.7997	0.0043	0.0606	0.0320	0.0166
X_2	0.00449	0.00380	4.23×10^{-5}	0.0042	0.0001	0.0004	0.0002	0.0004

4 Conclusions

From the discussions carried on laboratory test data, the following conclusions are coined out.

1. Appropriate time elapse should be required for developing proper bonding through poly-condensation. Maximum UCS of 650 kN/m^2 obtained for a mix proportion of 30% FA and 15% at a curing period of 28 days for 27° C.
2. The rate of chemical reaction enhanced with increase in curing temperature. At 7 days curing period, the UCS value at 50° C curing temperature increased by 2.56 times and 1.21 times when compared with 7 days and 28 days curing at 27° C at a mix proportion of 30% FA+15% L.
3. The significance of fly ash and liquid activator was effective when compared with Si/Al and Na/Al ratio. However, for higher compressive strength, the optimum range of Na/Al ratios should be 0.75 to 1.25 and Si/Al ratio should be greater than one.
4. Simple linear regression analysis are sufficient for predicting models for initial curing periods. Whereas, for long-term predictions, non-linear analysis was required.

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