Geotech Geol Eng (2017) 35:1907–1920
DOI 10.1007/s10706-017-0216-x

ORIGINAL PAPER

Mechanical Behavior and Sulfate Resistance of Alkali Activated Stabilized Clayey Soil

Binod Singhi · Aminul Islam Laskar · Mokaddes Ali Ahmed

Received: 28 June 2016 / Accepted: 20 March 2017 / Published online: 27 March 2017

Abstract Clayey subgrade soil requires treatment in order to make the subgrade stable for pavement structures. Treatment of clayey soil i.e. stabilization of clayey soil by cement, lime, and fly ash are established techniques used in geotechnical and highway engineering. Stabilization by alkali activation of fly ash is reported recently but literatures are limited. Present study investigates the stress strain behavior, peak stress and ultimate strain of clayey soil stabilized by slag and slag-fly ash blending by alkali activation. The peak stress as high as 25.0 N/mm^2 may be obtained at 50% slags content when 12 molar sodium hydroxide solutions were used. Peak stress, ultimate strain and slope of stress–strain curve of stabilized clay are controlled by Na/Al and Si/Al ratios. Stress–strain response and peak stress of slag and fly ash blended specimen are not governed by Na/Al and Si/Al ratios; rather the behavior is dependent predominantly on slag content.

Keywords Soil stabilization · Alkali activation · Geopolymer · Slag · Fly ash · Sulfate resistance

B. Singhi (✉) · A. I. Laskar · M. A. Ahmed
Civil Engineering Department, National Institute of Technology Silchar, Silchar, India
e-mail: bksinghi@gmail.com

A. I. Laskar
e-mail: aminul.nits@gmail.com

M. A. Ahmed
e-mail: ali.mokaddes@gmail.com

1 Introduction

Knowledge of performance of the subgrade soil is necessary prior to the construction of the pavement. Better the strength and stiffness, better is the long term performance. Subgrade constructed with clayey soils may not have enough strength and stiffness to support pavement loading. Clayey subgrade soil requires proper treatment in order to make the subgrade stable for overlying layers for pavement construction. Pavement stabilization using cementitious binders is a cost effective method used for improving the mechanical properties of subgrade, sub base, and base layers (Zhang and Tao 2008). Subgrade is the lower most layers in the pavement structure underlying the base course and sub base course of pavement. Soil possesses excellent performance at the optimum moisture content; however, the strength and stiffness of soil reduces drastically as the moisture content increases beyond the optimum. The effect of increase in moisture content on soil behavior has been a major concern among the geotechnical as well as pavement engineers. In addition, some soils have great tendency to shrink/swell with moisture content variation and often creates serviceability problems during or after construction of the foundations or pavement layers. The replacement of such soil with better quality of borrow soil filling is not always a good option especially in pavements due to high associated cost of excavation and hauling of the materials. In order to cope with this problem, various techniques have been applied by engineers depending upon the types of the soil. It is customary to treat the soils with some chemical stabilizers (Palmer et al. 1995). These stabilizers not only provide the working platform for construction through enhancing the strength of treated subgrade layer but also give the relatively stable sub-base for pavement (Van Ganse 1973). Stabilization by lime, cement and fly ash are established stabilization methods used widely around the world (Little 1995). Cement is an excellent stabilizing agent used extensively for base, sub-base and sub-grade construction in pavement engineering since 1950 (Little 1995). It is an established technology to improve the engineering properties of wide variety of soils including granular materials, silt and clay (Portland Cement Association 1992). Mixing of soil and cement in presence of water causes marked improvement in soil characteristics like increase in internal friction, decrease in shrinkage—swelling behavior and decrease in settlement due to continuous evolution of hydration products (Baker 2000). These hydration products are responsible for gaining strength in soil cement. The replacement of the cement by waste materials, such as fly ash, rice husk ash, and biomass ash, has been extensively applied in practice. The application of fly ash in soil stabilization is reported by researchers such as (Kawasaki et al. 1981; Kehew 1995) among others. There are lots of literatures available at present on soil stabilization by lime, fly ash, rice husk and slag (Sherwood 1993; Veith 2000; Wild et al. 1996; Bergardo et al. 1996; Chew et al. 2004; Probaha et al. 2000). At the end of the twentieth century, the alkali activation of alumino–silicate material like slag, fly ash and metakaolin emerged as alternative cementing materials among scientific community. At present there are abundance of literature available for alkali activated fly ash (El-Sayed 2011; Khater 2013; van Jaarsveld and van Deventer 1999; Lee and van Deventer 2002). These literatures are limited mainly to concrete, mortar and paste. Only recently, limited literatures for soil stabilization by alkali activated fly ash, metakaolin and slag have been reported (Verdolotti et al. 2008; Cristelo et al. 2011, 2012; Zhang et al. 2013; Yaolin et al. 2015; Du et al. 2016). Among the works reported on alkali activation of slag (Yaolin et al. 2015; Du et al. 2016), study reported by Yaolin et al. (2015) mainly emphasizes on effect of several alkali activator on the strength and microstructure properties of soft clay in grouting process. Other study available on alkali activation of slag by Du et al. (2016) investigated the efficacy of alkali activated slag stabilized clay for manufacturing light weight cementitious material.

In the present work, an attempt has been made to stabilize clayey soil by alkali activation of slag considering its applicability for improving engineering behavior of subgrade soil. Special emphasize has been given to study the stress strain behavior of the treated specimens in connection to its importance for evaluating both the initial, time dependent and long term movements of pavement embankment under static and dynamic loads (Weng and Wang 2011).

It has been recognized that for alkali activation of individual source material along with many advantages poses some disadvantages. For instance, slag activated systems have very rapid setting time and low workability and can be improved by addition of fly ash to slag system (Nicholson and Fletcher 2005). Likewise, strength gain process is very slow for fly ash based system at ambient temperature and significant strength can only be achieved by elevated temperature curing (Verdolotti et al. 2008; Cristelo et al. 2011, 2012). However, from practical consideration, curing at ambient temperature is much more convenient and realistic contrary to elevated temperature curing. Another way to overcome this is can be addition of slag to fly ash systems to expedite the strength gain process. Therefore, in blending individual drawbacks are counterbalanced, so the blends benefit from a synergy, resulting in geopolymer product with improved properties both in fresh and hardened state (Provis and van Deventer 2013). In view of the above discussion, testing programs of blend of slag with fly ash was also undertaken.

In this paper, results from a laboratory investigation of stress–strain response of soil stabilized by alkali activated slag and blend of slag–fly ash at different proportions were presented. Effect of varying experimental parameters such as binder content i.e. % slag, % slag + % fly ash in a blended mix and % fly ash, molar concentration of alkali solution (M), alkali to binder ration (R) and curing period on the peak stress, failure strain and elastic modulus of treated specimens are investigated Effect of M and R is evaluated in terms of Na/Al and Si/Al ratio of the mix which will be discussed in more detail in relevant sections of the study. A comparative study on the stress–strain response of the stabilized specimen of cement treated and alkali activated specimen immersed in a sodium sulphate solution was also investigated. The study on initial and final setting time of geopolymer paste of slag and blend of slag-fly ash at different molar concentration is presented. Finally, shrinkage characteristics of different mortar specimens prepared with alkali activated slag, alkali activated slag- fly ash and cement was also investigated.

2 Materials and Specimen Preparation

Clay soil was collected from the construction site near National Institute of Technology, Silchar. As per USCS classification, the soil is classified as clay with low plasticity (CL). The key characteristics of the untreated soil are presented in Table 1.

Soil was processed by oven drying for 24 h and sieved through 4.75 mm sieve in accordance with the standardized approach to prepare remolded specimen for laboratory testing (IS: 2720- Part 1).

Ground granulated blast furnace slag used in the laboratory investigation was commercially available ultra-fine ggbs, as the source material for geopolymer binder. The properties of ggbs are presented in Table 2. ASTM class C fly ash obtained from thermal power plant at Farakka (India) was used in the present study. The properties of fly ash are also shown in Table 2.

Cement used in the experimental study was Ordinary Portland Cement conforming IS 8112:1989 is used for preparing samples of cement stabilized specimen. The physical properties are as follows:

- Specific gravity = 3.12
- Standard consistency = 28%
- Initial setting time = 52 min
- Final setting time = 6 h
- 28 days compressive strength = 50.4 N/mm^2

Alkaline liquids used in geopolymerization are either combination of sodium hydroxide (NaOH) and sodium silicate, or potassium hydroxide (KOH) and potassium silicate. Sodium hydroxide solution was chosen in the present study as alkali activator. Sodium based solutions were chosen because they are cheaper. It is also reported that NaOH possesses greater capacity to liberate silicate and aluminate monomers (Zhang 2003). Research done by Bakharev (2005a, b), van Jaarsveld and van Deventer (1999) and van Jaarsveld et al. (1998) found that the geopolymer materials prepared with sodium hydroxide are more crystalline than those prepared with sodium silicate activators. It is worthwhile to mention that more crystalline the activator, the more stable will be geopolymer in an aggressive environment. Again, it is found that sodium cations have better zeolitization capabilities in geopolymer forming systems (Duxson et al. 2007).

Commercial graded sodium hydroxide in pallets (purity 98%; specific gravity 2.13) was used to prepare the solution with tap water. The mass of NaOH pallets in a solution varied according to molar strength (M). Preparation of the alkali solution was planned one day prior to use. The sodium silicate used in the study had a specific gravity 1.5 and its purity was 97%. The molecular weight of the sodium silicate ($Na_2SiO_3.5H_2O$) was 212. The weight ratio of SiO_2/Na_2O is 0.97 and percentage of Na_2O and SiO_2 are 29.25 and 28.30% in sodium silicate respectively.

3 Experimental Program and Test Details

Different percentages of binder content for slag (i.e. in terms of dry weight of soil) taken are 12, 20, 30, 40 and 50%. In case of blending of slag with fly ash, a fixed binder content of 20% is considered. However, relative proportions of slag and fly ash in the blended mix is varied to produce a range of blended geopolymer specimens as shown in Table 6. Similarly for fly ash i.e. both pulverized and un-pulverized, a fixed binder content of 20% is adopted. Molar concentration, M of

Table 1 Physical properties of soil

Soil type	Liquid limit	Plastic limit	Plasticity index	MDD ($gm\ cm^{-3}$)	OMC
S	37.68	23.61%	14.07%	1.69	19.05%

MDD Maximum dry density, *OMC* Optimum moisture content

Table 2 Chemical composition and fineness of source materials

Particulars	Content (mass, %)			
	Slag	Typical range	Fly ash	Typical range
CaO	34	34–43	0.67	1–40
Al_2O_3	20	7–18	22.63	5–35
Fe_2O_3	2	<2	5.3	4–40
SO_3	0.8	1–1.9	0.41	0.23–3
MgO	8	7–15	0.16	1.5–15
SiO_2	35	27–38	66.39	15–60
Specific surface area (m^2kg^{-1})	800	800–1000	330	350–500

alkali solution considered in the study are 4, 8, 12 and 14.5 M whereas three different alkali to binder ratio (i.e. 0.45, 0.65 and 0.85) were chosen.

Following procedure was followed while preparing the sample for stabilized alkali activated sample.

- Oven dried soil was weighed to the nearest gram;
- The requisite quantity source material i.e. slag/blend of slag and fly ash/fly ash (pulverized or un-pulverized) was weighed to the nearest gram;
- The soil and source material were mixed thoroughly for five minutes;
- Alkali was added to soil–source material mixture and mixed thoroughly until a uniform mix was prepared. Mixing can be done easily using mixing appliance used in kitchen.

Specimens were prepared at a consistency equal to the plastic limit of soil. It was observed that desired workability for uniform mixing was achieved at plastic limit of soil. Studies such as (Yaolin et al. 2015; Du et al. 2016) also reported a consistency equal to either liquid limit or greater than liquid limit to facilitate thorough and homogeneous mixing of soil binder mixture with alkali solution. The soil mixed with binder and alkali solution were rolled, put in PVC molds having a diameter of 38 mm and a height of 76 mm and compacted manually to expel air voids from the mix. Manual compaction of soil samples was mainly aimed to eliminate air voids and enable proper compaction of specimens (Yaolin et al. 2015). Though, manual compaction is a source of uncertainty in the measurement of degree of compaction and density of treated specimens but subsequent variation was very less and its effect on the strength properties of treated specimens can be neglected due to following reasons.

- Geopolymer gel binds the soil particle which upon hardening produces geopolymer matrix where the soil is primarily distributed as a filler material.
- Strength of the resultant product termed as geopolymer composite (geopolymer + filler) depend upon mainly on the strength of Si–O–Al/Si–O–Si/Al–O–Al bonds (Duxson et al. 2007) where density of filler materials (i.e. soil) contribution toward the strength is marginal compared to that of geopolymer matrix.

The samples were then taken to the curing yard and immersed in water for continuous curing for 7, 14 and 28 days at ambient temperature (35 ± 2 °C). After competition of desired curing period, cured samples were air dried at room temperature for 1 h and was subjected to unconfined compressive strength testing as per Indian Standard Code of Practice IS -2720 (Part 10): 1991—Determination of unconfined compressive strength.

4 Test Results and Discussion

The results and discussions are given in the following section.

4.1 Effect of Slag Content

The stress–strain behavior at various percent contents of alkali activated slag stabilized soil specimens are presented in Fig. 1 at M and R values that are 12 and 0.65 respectively. Increase in percent content of slag leads to increase in the slope of the stress strain curve.

Further, the peak stress of the stabilized soil sample increases with increase in slag content, followed by decrease in axial strain. The higher the slag content,

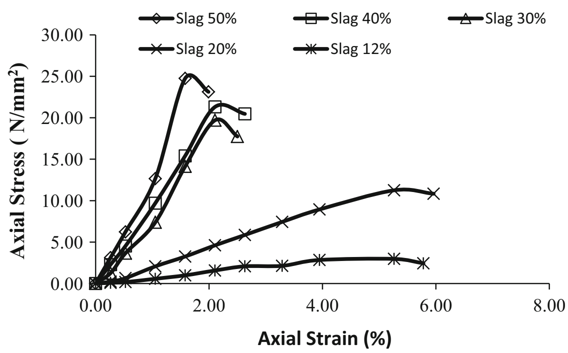

Fig. 1 Effect of slag content on AAS (M = 12, R = 0.65)

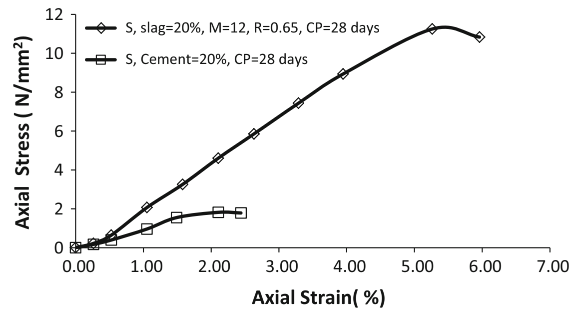

Fig. 2 Stress–strain response of cement and slag based specimens

the higher will be the peak stress. It is clearly seen that with the increase of slag content the stress- strain curve shift towards left hand side. At the same time the strain at failure reduced with the increase in slag content which means increase in elastic modulus and shear modulus of the alkali activated stabilized soil specimens. As the slag content increases, more geopolymer matrix is available to bind relatively less amount of soil resulting in increased peak stress and subsequent rise in stiffness of treated specimens. As seen from Fig. 1, a continuous increase in peak stress and hence elastic modulus with binder content is observed. Beyond a binder content of 20%, a sharp rise in peak stress is observed. Minimum binder content considered in the study is 12%. For binder contents lower than 12%, peak stress values were considerably low as observed by the author on trial specimens probably due to less geopolymer gel dispersed in the soil geopolymer system. It may be observed that ductility of specimens prepared with slag contents 12 and 20% was more compared to that of 30, 40 and 50% and in particular for 20% slag content as it exhibited higher peak stress. In Fig. 1, slag content of 20% is assumed to be optimum binder content as a reasonable trade-off between sufficiently high strength with more ductility and binder content can be achieved corresponding to this slag content. The comparison of soil stabilized specimen by alkali activated slag and cement is presented in Fig. 2. A binder content of 20% is chosen for comparison of slag with cement due to reasons discussed above. It is noticed that at same percentage of binder content i.e. 20%, the alkali activated slag soil (AASS) specimen has a much higher peak stress and elastic modulus compared to the cement treated specimens. The AASS specimen exhibit axial/brittle failure as in case of cement treated specimens. The most noticeable observation was that the AASS specimen shows relatively higher ductility compared to the cement treated specimens as higher values of axial strain of the AASS specimen are easily noticeable (Fig. 2).

It is important to specify here that soil generally fails in shear under compression. However, in the present study as observed from Fig. 3, AASS exhibited cone and split failure mode, typical of that shown by normal mortar or rock during crushing failure (ASTM C39). This suggests feasibility of slag as a geopolymer precursor for yielding a highly deformation resistant geopolymer matrix structure. Furthermore, in Fig. 1, shape of the stress–strain graphs were sufficiently straight indicating the transformation of treated specimens from soil to rock like behavior. Based on the result of the present investigation, variation of UCS with elastic modulus of the AASS is presented in Fig. 4. The best fitting line with $R^2 = 0.83$ suggests that UCS is strongly correlated

Fig. 3 Slag based stabilized soil sample after failure (30% slag content)

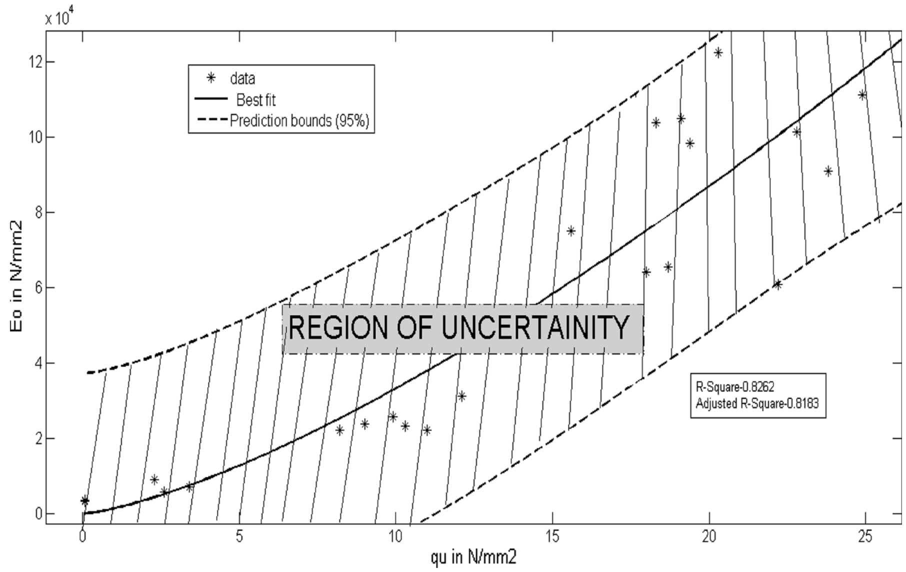

Fig. 4 UCS—Elastic Modulus of alkali activated slag

with elastic modulus. Following relationship between unconfined compressive strength and elastic modulus of AASS is obtained for the best fitting line indicating that elastic modulus varies as a power of UCS.

$f(x) = ax^b$;

where, a = 1378 and b = 1.383 where, x is the UCS in MPa, f(x) is the response i.e. elastic modulus.

As shown in Fig. 4, prediction bounds are plotted corresponding to 95% confidence interval and signifies that possibility of any future prediction of elastic modulus from UCS of falling outside the bound is not more than 5%. It may be observed that all the elastic modulus values lies within the prediction bounds. In general, it may be concluded that scatter of elastic modulus values are normally distributed w.r.t the mean value (i.e. best fitting line) with 95% of its data values are within 1.96 times (As the z-score for 95% confidence interval is 1.96) of standard deviation of the mean.

4.2 Effect of Curing

AASS samples of 20% slag content for M = 12, R = 0.45 were tested for three curing periods: 7, 14 and 28 days. Effect of curing on stress–strain behavior is presented in Fig. 5.

The slope of stress–strain curves of 7 and 14 days are almost identical, although 14 days cured samples exhibited higher peak stress and subsequent axial strain. Highest slope and peak stress was observed for specimen cured for 28 day. It

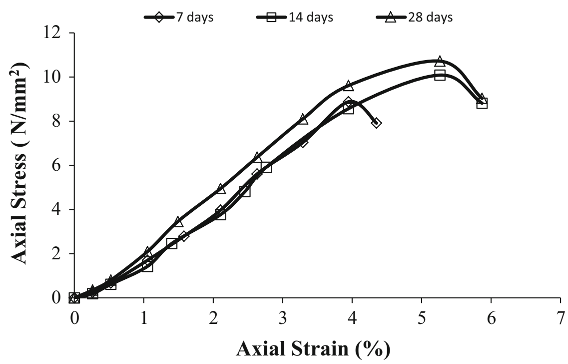

Fig. 5 Effect of curing on stress–strain behavior of alkali activated slag (Slag 20%, M = 12, R = 0.45)

may be observed that, specimen cured for 28 day showed same axial strain as that of 14 day. Marginal increase in peak stress from 7 to 14 and 14 to 28 day may be attributed to the rapid geopolymerization of AASS specimens where most of the geopolymerization takes place within first seven days of curing. Slag being highly reactive and when used as a geopolymer precursor contributes to the high early strength gain. Zhang et al. (2013), also observed no appreciable gain in strength after first seven days of curing of alkali activated metakaolin for stabilizing clay soil.

4.3 Effect of Na/Al Ratio for Slag Based Geopolymer-Soil

Generally, geopolymer mix is represented in terms of molar concentration of alkali (M) and binder to alkali

Table 3 Calculation of Na/Al and Si/Al ratio for mixes

Molar concentration (M)	Alkali to slag ratio (R)	Na/Al ratio	Si/Al ratio
4	0.45	0.31	1.35
8	0.45	0.55	1.35
12	0.65	1.06	1.35
12	0.85	1.39	1.35
14.5	0.45	0.84	1.35
14.5	0.65	1.21	1.35
14.5	0.85	1.58	1.35

ratio (R) as UCS of geopolymer paste, mortar and concrete, are dependent on M and R (Palomo et al. 1999). Recent literatures on geopolymer binder suggest that Na/Al and Si/Al ratios are crucial parameters which significantly affect the kinetics of geopolymer paste and mortar (Khale and Chaudhary 2007; Xu and van Deventer 2003; Rees 2007). The sources of these cations are alkali (Na^{+}) and source material (Si^{+} and Al^{+}). In the present investigation, the stress–strain response was studied using Na/Al and Si/Al ratio for 20% slag content. Detail calculation of these ratios for mixes under investigation is presented in Table 3.

The stress–strain behavior of clayey soil stabilized by alkali activated slag (20% slag content) for different Na/Al ratios ranging from 0.31 to 1.58 after 28 days curing are presented in Fig. 6. For clarity and better understanding of the Fig. 6, peak stress values corresponding to different Na/Al ratios are presented in Table 4. Continuous increase in peak stress and slope of stress–strain curves are observed with the increase in Na/Al ratio from 0.31 to 1.06. When Na/Al ratios are 0.31 and 0.55, ultimate stresses of the

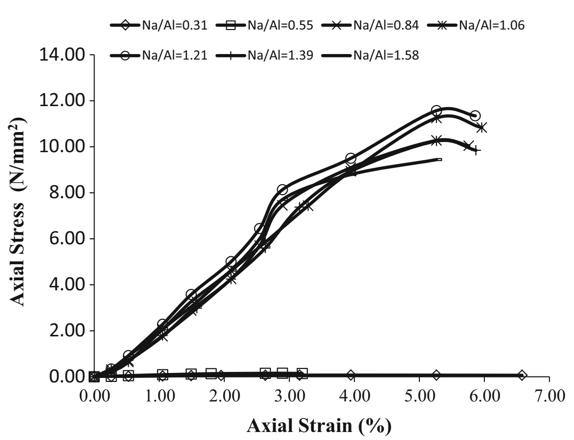

Fig. 6 Effect of Na/Al ratio on stress–strain behavior of alkali activated slag

Table 4 Peak stress values of AASS system corresponding to different Si/Al ratios

Na/Al ratio	Peak stresses in MPa	Na/Al ratio	Peak stresses in MPa
0.31	0.075	1.21	11.7
0.55	0.156	1.39	10.35
0.84	10.4	1.58	9.46
1.06	11.2		

stabilized samples are not significant. Whereas at Na/Al = 0.84, peak stress observed is 10.3 N/mm^2 (Fig. 6). At Na/Al ratio 1.06 and1.21 peak stresses observed are 11.25 N/mm^2 and 11.57 N/mm^2 at same axial strain. Increase of Na/Al ratio beyond1.21 and upto1.39 there was little decrease in the ultimate stress (10.25 N/mm^2). Further increase of Na/Al (1.58) there was appreciable, decrease in the peak stress (Fig. 6). The trend of the peak stress and stress–strain response with change in Na/Al ratio indicates that there exists an optimum range of Na/Al ratio in an AASS matrix. Xu and van Deventer (2003) and Rees (2007) also reported an optimum range Na/Al ratio where maximum strength values were achieved and the optimum range was found to be 0.75–1.25 in the case of geopolymer paste.

4.4 Effect of Si/Al Ratio

The Si/Al ratio of a particular alumino silicate source material is constant, as proportions of SiO_2 and Al_2O_3 of the source material is fixed. Any variation in the Si/Al ratio in an alkali activated mix can be done either by changing the Si^{+} content or Al^{+} content. In the present study, a variation in the Si/Al ratio was done by adding sodium silicate in the mix as a secondary source of Si^{+}. When sodium silicate was added into the AASS mix, the content of sodium hydroxide was reduced in

Table 5 Different Si/Al ratios with sodium silicate addition, slag = 20%

NaOH (%)	Si/Al (Na/Al = 1.15)
100	1.35
80	1.49
60	1.63
40	1.81

such way that the overall weight of the sodium remains constant as sodium silicate also contains Na^{+}. To study the effect of Si/Al ratio on UCS of AASS, the slag percent was kept constant at 20%. The details of the mix preparation are presented in Table 5. Experimental results are presented in Fig. 7.

It may be observed from Fig. 7 that as the Si/Al ratio increases, the axial stress continuously increases. However, the increase in slope of stress–strain curve and peak stress are not significant. Peak stress as high as 15.1 N/mm^2 is observed at Si/Al ratio = 1.81. In the present study the range of Si/Al ratio was (1.35–1.81). Minimum peak stress was noticed when Si/Al = 1.35. However, for all values of Si/Al, sufficiently high strength was achieved for AASS system. This is due to the fact that source materials with Si/Al ratio greater than 1 will always yield higher UCS values provided Na/Al ratio and binder content are within optimum range (Xu and van Deventer 2003; Rees 2007). In the present study, variation in Si/Al ratio were done to study its effect on the strength behavior of AASS systems. However, from results in Fig. 7 it may be concluded that for source materials such as slag with Si/Al ratio greater than 1, variation in Si/Al will not have significant effect in enhancing the strength of treated specimens.

4.5 Effect of Na/Al Ratio for Fly Ash Based Geopolymer–Soil

The effect of the Na/Al ratio on peak stress and stress–strain response of fly ash based geopolymer-clayey soil was also studied and the results are presented in Fig. 8. Na/Al ratio corresponding to R equal to 0.45, 0.65 and 0.85 at constant M = 12 were 1.26, 1.82 and 2.38 respectively. Similarly, Na/Al ratios corresponding to R values 0.45, 0.65 and 0.85 at M = 14.5 were respectively 1.42, 2.06 and 2.69. The minimum Na/Al ratio in the present study alkali activated fly ash soil (AAFS) system is 1.26 for 12 M. The peak stress of the stabilized samples is maximum when the Na/Al is 1.26. The peak stress of the stabilized specimen of Na/Al = 1.46 for the AAFA system shows comparable results with Na/Al = 1.26.

At all other combination of Na/Al ratio (1.82–2.69), the observed peak stress and slope of the stress- strain curve shows decreasing trend. In fact the specimen with the highest Na/Al ratio shows the lowest peak stress and so as the slope of the stress–strain curve. The peak stress obtained for AAFA system as shown in Fig. 8 are nowhere in comparison with the peak stresses of the AASS system as shown in Fig. 6.

It is because of the fact that the dissolution rate of fly ash is quite less compare to slag during alkali activation. This finding is consistent with the findings Xu and van Deventer (2003) in case of fly ash based geopolymer paste. In another study by Cristelo et al. (2012), AAFA system were subjected to heat

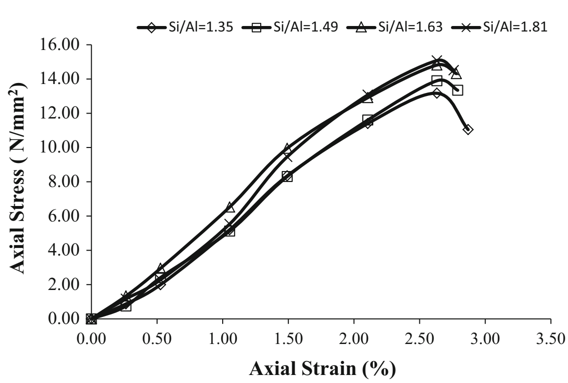

Fig. 7 Effect of Si/Al ratio on stress–strain behavior of alkali activated slag

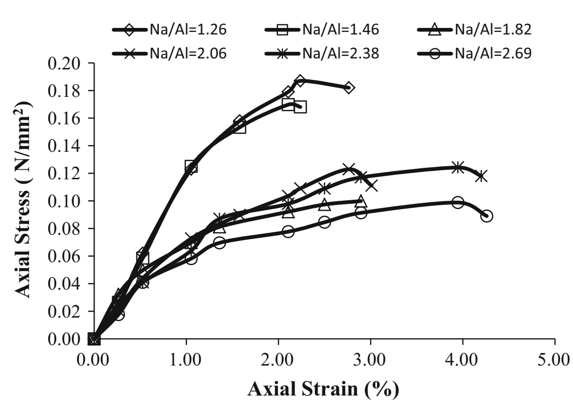

Fig. 8 Effect of Na/Al ratio on stress–strain behavior of alkali activated fly ash

treatment for gaining comparatively high strength of fly ash based soil geopolymer. The heat treatment is necessary for fly ash based geopolymer as the binding energy associated with fly ash is quite higher compared to slag (Hua Jian et al. 2008) and requires external input in terms of energy source to break the internal bonds thereby to liberate Si^{+} and Al^{+} ions. In other words fly ash is less reactive at ambient environment compared to slag during alkali activation.

4.6 Blending of ggbs and Fly Ash

In the present investigation the stress–strain response of the slag and fly ash blends are shown (Fig. 9). Because of the variation in percentage of SiO_2 and Al_2O_3 of slag and fly ash, the change in the Na/Al and Si/Al changes with the blending ratio of two source material (slag and fly ash). Specimens were prepared using 12 molar of NaOH solution for stabilization of clayey soil by blend of slag and fly ash as source material. Variation of Na/Al and Si/Al ratios for samples with blended source material are presented in Table 6.

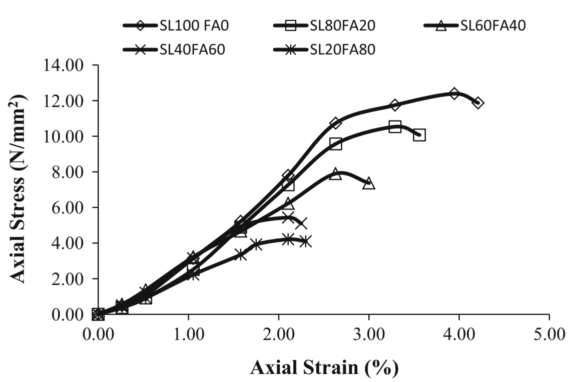

Fig. 9 Effect of Na/Al ratio on stress–strain behavior of alkali activated slag and fly ash blended source material

In Table 6, FA0SL100 means that fly ash and slag contents were 0% and 100% respectively of the total source material (i.e. blend of fly ash and slag). Similarly, FA20SL80 means that fly ash was20% and slag was 80% by weight of source material and so on.

Figure 9 shows the stress strain response of stabilized samples for various blending proportions. As the fly ash content increases relative to slag, both peak stress and ultimate strain decreases. Na/Al ratios for samples in Fig. 9 are in optimum range: 0.75–1.25 as suggested by Xu and van Deventer (2003) for maximum UCS. When Na/Al ratio is within the optimum range, Si/Al ratio is supposed to govern the strength of stabilized samples. But this is not happening in case of blended alkali activated system. Peak stress increase as the Si/Al ratio decreases which is not consistent with the findings of Xu and van Deventer (2003). It is recognized that the dissolution extent of source materials varies with the variation of source materials used. As stated earlier fly ash is less reactive compared to slag and thereby dissolution of Si^{+} and Al^{+} is comparatively less in fly ash because of its high binding energy (Hua Jian et al. 2008).Therefore, participation of fly ash in geopolymer formation is not quite less compare to slag. The strength gain of samples with the decrease in fly ash is predominantly due to availability of more and more slag in the system. Thus in case of slag-fly ash blending, percent content of slag is more dominating rather than the Si/Al ratio of the blend because of two reasons. This is possibly due to two reasons. First, addition of slag in the fly ash based geopolymer mix leads to increase in the overall content source of CaO within the mix due to presence of high lime content in slag, which results in faster and more complete geopolymerisation (Lee and van Deventer 2002).Second, the binding energy of slag is less than that of fly ash and hence, reactivity of slag is faster than fly ash in alkaline environment.

Table 6 Mix proportions of blended source material at R = 0.65

Mix designation	Source materials	Mix parameters (M, R)	Na/Al ratio	Sample designation	Si/Al ratio
SL20FA80	Slag 4%, FA 16%	12 M, 0.65	1.20	Sample 1	2.27
SL40FA60	Slag 8%, FA 12%	12 M, 0.65	1.21	Sample 2	2.03
SL60FA40	Slag 12%, FA 8%	12 M, 0.65	1.22	Sample 3	1.79
SL80FA20	Slag 16%, FA 4%	12 M, 0.65	1.24	Sample 4	1.55
SL100FA0	Slag 20%, FA 0%	12 M, 0.65	1.25	Sample 5	1.35

Consequently, dissolution rate of Si^{+} and Al^{+} ions in slag is higher than fly ash (Hua Jian et al. 2008).

4.7 Setting Time of Geopolymer

Setting time of alkali activated slag and blending slag and fly ash paste was determined using Vicat's needle apparatus as per Indian Standard Code Practice IS: 4031 (Part 5)-1988. The results were presented in Fig. 10.

Initial and final setting times decreased with the increase in molar concentration. Reductions in setting times are due to evolution of more products of geopolymerization at higher M. At higher molar concentrations (such as 7 and 9 M in Fig. 10), initial setting time was 10 min and final setting time was 30 min only. It may be observed that, at higher molar concentration (i.e. greater than 7 M), effect of blending of fly ash with slag in retarding setting time was insignificant. However, at lower molar concentrations (i.e. less than 4 M) a substantial retardation in setting time was observed for blended mixes.

4.8 Effect of Fineness of Fly Ash on UCS of Stabilized Soil

Fly ash based geopolymer are well known for their superior resistance to the impact of aggressive environment, fire resistance, low density and low thermal conductivity (Duxson et al. 2007; Juenger et al. 2011; Panias et al. 2007; Andini et al. 2008; Bascarevic et al. 2013). In a geopolymer synthesis, the nature of the fly ash needs to be amorphous. Slag and fly ash are material of two different origins; thus, there amorphous characteristics are quite different. The fineness of slag and fly ash is presents in Table 2 which indicates that mechanical activation of fly ash is required before blending as planned. Accordingly, fly ash in the present study is pulverized using a laboratory type pulverizer and the fineness value of pulverized fly ash obtained was 430 m^2/kg. Soil-fly ash based geopolymer specimens were prepared as before with pulverized and un-pulverized fly ash for 20% content of M = 14.5 and R = 0.65 and cured for 28 days. The stress–strain response of the pulverized and un-pulverized fly ash is presented in Fig. 11.

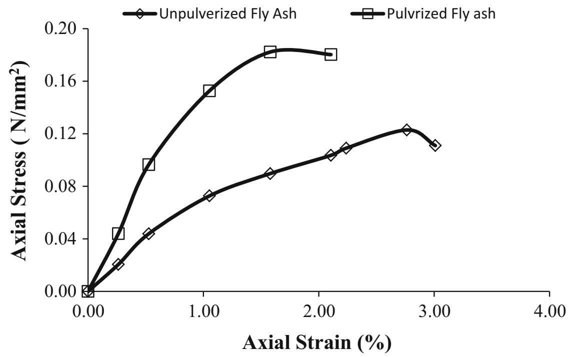

Fig. 11 Effect of grinding of on stress–strain behavior of alkali activated fly ash

It may be observed from Fig. 11 that pulverization improves stress–strain behavior with an increase in axial stress and decrease in the corresponding axial strain, thereby increase in elastic modulus and shear modulus. It is reported that for a geopolymer synthesis, the presence of amorphous characteristics of

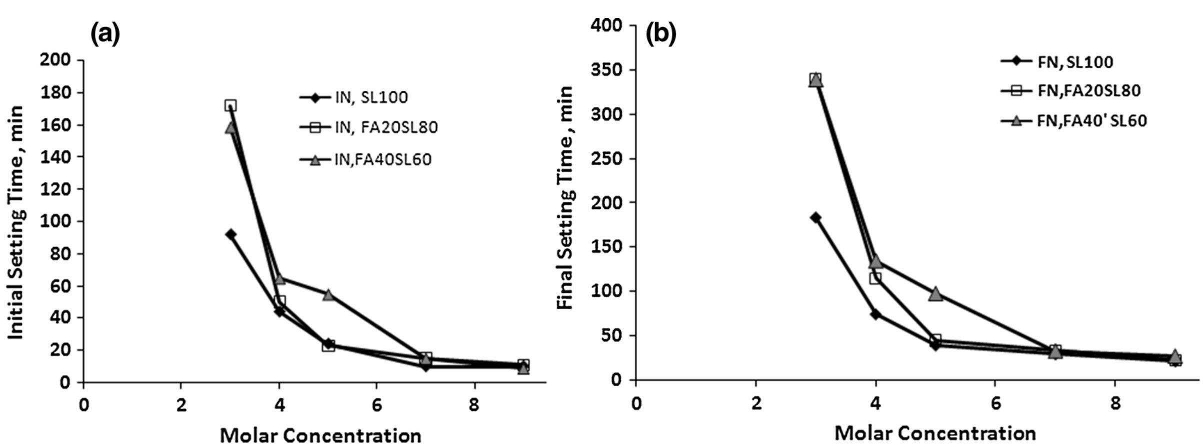

Fig. 10 Initial and final setting times of geopolymer pastes **a** initial setting time **b** final setting time

alumino silicate source is essential. It is also reported that amorphous component of fly ash has better binding potential which can improve mechanical behavior of the geopolymer (Marjanovic et al. 2014; Mehrotra et al. 2008). The mechanical activation of fly ash induces changes in the source material like reduction in particle size, change in particle morphology, increase in specific surface area, structural defect, decrease in degree of crystallinity and structural rearrangement et. (Fernandez-Bertran 1999; Guo et al. 2010; Zhang and Saito 2012; Zivanovic et al. 2002). The most significant consequence of transformation that occur during the mechanical activation is its enhanced reactivity (Marjanovic et al. 2014). The improvement in fly ash after grinding enhance the compressive strength of geopolymer paste was also reported by Marjanovic et al. (2014) and Mehrotra et al. (2008).

4.9 Sulfate Resistance of Alkali Activated Stabilized Specimen

Deterioration of cementitious product in a sulphate environment is an established fact (Neville 2003). Research in the past also highlights that soil cement is vulnerable under sulphate exposure in a similar manner as that of cement concrete though the rate of deterioration is much faster (Cordon 1962) in soil cement. Sulphate induced heave in cement stabilized soil was reported by Mitchell (1986) while investigating a case study of cement treated soil subgrade failure. Besides being adequate pavement structure, the failure of the stabilized road occurred, which he categorized as failure due to sulphate exposed environment. He also concluded that the soil contained significant amounts of soluble sodium sulfate (up to 1.5% by weight).

In the present study, the sulphate resistance behavior of the slag and slag-fly ash blending was carried out. Stabilized soil specimens by alkali activation of slag and mixture of slag– fly ash were prepared at M = 12 and R = 0.65 with 20% source material. Proportions of slag and fly ash in blending are designated as SL80FA20, SL60FA40, SL40FA60 and SL20FA80 as described in (section else when in this paper). Specimens were cured in a curing tank for seven days and then immersed in 5% sodium sulfate solution for another for 3 weeks as shown in Fig. 12.

Fig. 12 Stabilized specimens of during sodium sulphate exposure (alkali activated slag and slag–fly ash blend specimens)

Control specimens were continuously kept in water for 28 days.

After 28 The visual observation of the specimens shows that the alkali activated specimens exposed to sodium sulphate solution did not show any sign of deterioration after 28 days. Contrary to this, cement stabilized specimens completely disintegrated as shown in Fig. 13.

The stress strain behavior of alkali activated specimen after sulfate solution exposure are shown in Fig. 14. Specimens cured under water resulted in the peak stress of 12.3 N/mm^2, and the same after immersion in sodium sulphate solution immersion resulted in relatively lesser peak stress of 11.2 N/mm^2. After seven days of curing when samples were exposed to sulphate environment for 21 days, there was a shift of the stress strain curve towards right side, which indicates a decrease of elastic modulus of the

Fig. 13 Stabilized specimens after sodium sulphate exposure (cement stabilized)

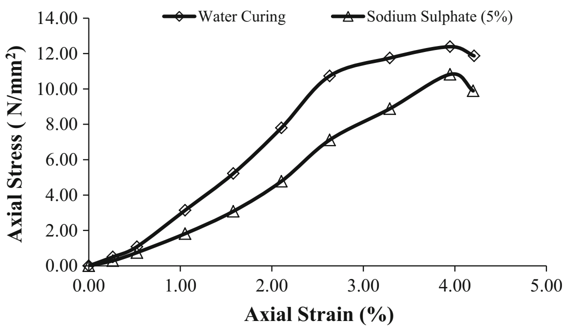

Fig. 14 Effect of sulfate attack on stress- strain behavior of alkali activated slag stabilized soil

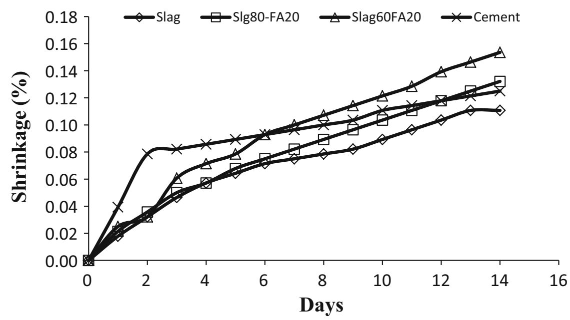

Fig. 15 Shrinkage of geopolymer binder

stabilized specimen, although not much of difference in axial strain were noticeable. A similar such reduction of elastic modulus was found in the blended mix of slag–fly ash activated specimens. The overall 12–15% reduction of peak stress was noticed in all the samples tested after 21 continuous days of inundation in the sodium sulphate solution (5% by weight). A reduction in the strength of slag based geopolymer mortar under sulphate exposure was reported by Khater (2013).

A significant increase in the pH of the sulphate solution was observed during experimental program. Test records of the present study suggest that the pH of the solution increased from 7.0 to 9.2 during the period of sulphate exposure due to cation migration from specimen to solution. Similar increase in pH was also reported by Bahkarev (Bakharev 2005a, b) in case of fly ash based geopolymer paste.

4.10 Shrinkage of Geopolymer

Shrinkage of geopolymer is an important aspect of the geopolymer mix. The shrinkage characteristics of geopolymer mortar and cement mortar were investigated by using mortar bar apparatus used in the laboratory for cement mortar as per Indian Standard.

It was observed from Fig. 15 that shrinkage of slag based geopolymer mortar at M = 12 and R = 0.65 shows a value 0. 08 and 0.11%, while the cement mixed mortar specimen exhibit shrinkage of 0.09 and 0.13% respectively after 7 and 14 days. The blended mix of slag and fly ash show values increase in shrinkage, 0.08and 0.13% for Slag 80–FA 20 blended mix. Again, when fly ash content in the mix increases, i.e. the blended mix of SL60–FA40 shows values of shrinkage 0.09 and 0.15%. It can be concluded from the above figure that though the initial shrinkage of cement based mortar shows the higher rate of shrinkage, but at later age it shows similar trend as that of slag based geopolymer samples. The blended mix although exhibit slightly higher percentage of shrinkage and it increase a bit with the increase of fly ash content In the mix but the shrinkage values are not so differ in terms of values. Thus, it can be concluded that the shrinkage behavior is similar for geopolymer based mortar.

5 Conclusions

Present study investigated the effectiveness of AASS system on improving the engineering behavior of subgrade clay. Effect of blending of slag with fly ash and fly ash alone is also investigated. Unconfined compressive strength tests were done to evaluate the effect of experimental parameters on UCS, peak stress, failure strain and elastic modulus of treated specimens. Furthermore, laboratory tests pertaining to setting time, shrinkage and durability behavior were also conducted. Following general conclusions may be derived from the present study.

- Peak stress, failure strain and elastic modulus of the specimens stabilized by alkali activated slag depends upon the percent content of the slag, molar concentration of alkali activator and alkali to slag ratio.
- Effect of curing on the strength and stiffness of treated specimens beyond first 7 day of curing is marginal with specimens developing most of the strength and stiffness within first 7 days of curing.
- The percentage increase in slag content always lead to increase in peak stress and elastic modulus.

Effect of molar concentration (M) and alkali to slag ratio (R) on strength and stiffness behavior is not straightforward. In fact, Na/Al ratio and Si/Al ratio of the mix (calculated from M and R) plays the crucial role for development of UCS and elastic modulus of the stabilized specimen. The peak stress, failure strain and slope of stress–strain curve of soil- geopolymer is ultimately controlled by Na/Al ratio and Si/Al ratio of the mix at a particular binder content.

- The variation in Na/Al ratio and Si/Al ratio can be done using sodium silicate solution in manner to bring the desired Na/Al ratio and Si/Al ratio.
- Optimum ranges of Na/Al and Si/Al ratio suggested by researchers for peak stress are not applicable in case of blending of two different source materials having different dissolution potentials.
- Blending of fly ash with slag results in decrease in peak stress, failure strain and elastic modulus of treated specimens with increasing fly ash content in the blended mix which is quite evident. However, increase of setting time with increase in fly ash content in the blended mix was only observed at lower molar concentrations i.e. less than 4 M. At higher molar concentrations, increase in fly ash content in the blended mix has insignificant effect on retarding both initial and final setting time.
- The durability under sulphate exposure and shrinkage characteristics of alkali activated slag and blend of slag and fly ash was better than that of cement treated samples. In particular, %shrinkage at 7 and 14 day was lowest for alkali activated slag compared to that of the blended mix.

References

Andini S, Cioffi R, Colangelo F, Grieco T, Montagnam D, Santoro L (2008) Coal fly ash as raw material for the manufacturing of geopolymer based product. Waste Manag 28:416–423

ASTM C39/C39M-05 (2005) Standard test method for compressive strength of cylindrical concrete specimens. American Society for Testing and Maretials, US

Baker S (2000) Deformation behavior of lime-cement column stabilized clay, Ph. D. thesis, Chalmers University of Technology, Sweden

Bakharev T (2005a) Durability of geopolymer material in sodium and magnesium sulfate solutions. Cem Concr Res 35:1233–1246

Bakharev T (2005b) Resistance of geopolymer materials to acid attack. Cem Concr Res 35:658–670

Bascarevic Z, Komljenovic M, Miladinovic Z, Nikllic V, Marjanovic N, Zujovic Z (2013) Effect of concentrated NH_4-NO_2 solution on mechanical properties and structure of the fly ash based geopolymers. Constr Build Mater 41:570–579

Bergardo DT, Anderson LR, Miura N, Balasubramaniam AS (1996) Soft ground improvement in low land and other environment. ASCE Press, New York, pp 234–304

Chew SH, Kamruzzaman AHM, Lee FH (2004) Physicochemical and Engineering behavior of cement treated clays. J Geotech Geoenviron 130:696–706

Cordon WA (1962) Resistance of soil cement exposed to sulfates. 40th Annual Meeting of the Highway Research Board, Highway Research Board Bulletin, 309, Washington, DC

Cristelo N, Glendinning S, Farnandes L, Pinto AT (2011) Effect of calcium content on soil stabilization with alkaline activation. Constr Build Mater 29:167–174

Cristelo N, Glendinning S, Pinto AT (2012) Deep soft soil improvement by alkaline activation. Ground Improv 164:1–9

Du Y, Yu B, Liu K, Jiang N, Liu MD (2016) Physical, hydraulic, and mechanical properties of clayey soil stabilized by lightweight alkali-activated slag geopolymer. J Mater Civ Eng. doi:10.1061/(ASCE)MT.1943-5533.0001743

Duxson P, Fernandez-Jimenez A, Provis JL, Palomo A, van Deventer JSJ (2007) Geopolymer technology, the current state of the art. J Mater Sci 42:2917–2933

EL-Sayed HA, ABO EL-Enein SA, Khater HM, Hasanein SA (2011) Resistance of alkali activated water cooled slag geopolymer to sulphate attack. Ceram Silik 55:153–160

Fernandez-Bertran JF (1999) Mechanochemistry: an overview. Pure Appl Chem 71:581–586

Guo X, Xiang D, Duan G, Mou P (2010) A review of mechanochemisty application in waste management. Waste Manag 30:4–10

Hua Jian L, Sun Heng Hu, Xu Chu TIE, Xiao Xue Jun (2008) A new method to evaluate the hydraulic activity of Al–Si materials. Sci China Ser E 51:113–120

IS: 2720, Part-1 (1995) Preparation of dry soil samples. Bureau of Indian Standard, New Delhi

IS: 4031, Part 5 (1988) Methods of physical tests for hydraulic cement. Bureau of Indian Standard, New Delhi

IS: 8112 (1989) Specification for 43 grade ordinary portland cement. Bureau of Indian Standard, New Delhi

Juenger MCG, Winnefld F, Provis JL, Ideker JK (2011) Advances in alternative cementitious binders. Cem Concr Res 41:1232–1243

Kawasaki T, Niina A, Saitoh S, Suzuki Y, Honjo Y (1981) Deep mixing method using cement hardening agent. In: Proceedings of the 10th international conference on soil mechanics and foundation engineering, Southeast Asian Geotechnical Society, Bangkok, 721–724

Kehew EA (1995) Geology for engineers and environmental scientists, 2nd edn. Prentice Hall, Englewood Cliff, pp 295–302

Khale D, Chaudhary R (2007) Mechanism of geopolymerisation and factors influencing its development: a review. J Mater Sci 42:729–746

Khater HM (2013) Studying the effect of thermal and acid exposure on alkali-activated slag geopolymer. Adv Cem Res 25:1–9

Lee WKW, van Deventer JSJ (2002) Structural reorganisation of class F fly ash in alkaline silicate solutions. Colloid Surf A 211:49–66

Little DN (1995) Handbook for stabilization of pavement subgrades and base courses with lime. Kendall Hunt Publishing, Dubuque

Marjanovic N, Komlijenovic M, Bascarvic Z, Nikolic V (2014) Improving reactivity of fly ash and properties of ensuing geopolymer through mechanical activation. Constr Build Mater 57:151–162

Mitchell JK (1986) Practical problems from surprising soil behavior. J Geotech Eng-ASCE 112:259–289

Neville AM (2011) Properties of concrete. Pearson Education Limited, Edinburgh Gate, Essex

Nicholson CL, Fletcher RA (2005) Geopolymers and methods for their production. Patent No. WO 2005/019130 A1

Palmer JT, Edgar TV, Boresi AP (1995) Strength and density modification of unpaved road soils due to chemical additives, MPC Report No. 95–39, Mountain–Plains Consortium, Fargo, ND

Palomo A, Grutzeck MW, Blanco MT (1999) Alkali-activated fly ashes: a cement for the future. Cem Concr Res 29:1323–1329

Panias D, Giannopoulou IP, Perraki T (2007) Effect of synthesis parameter on the mechanical properties of fly ash based geopolymer. Colloid Surface A 301:246–254

Portland Cement Association (1992) Soil cement laboratory handbook. Portland Cement Association, Skokie, Illinois

Probaha A, Shibuya S, Kishida T (2000) State of the art in deep mixing technology Part III, geomaterial characterization. Ground Improv 3:91–100

Provis JL, van Deventer JSJ (2013) Alkali activated materials: state-of-the-art report, RILEM TC 224-AAM. Springer, Berlin

Rees CA (2007) Mechanism and kinetics of gel formation in geopolymers. Ph. D. thesis, The University of Melbourne, Australia

Sherwood PT (1993) Soil stabilization with cement and lime: state of the art review. HMSO Publication, London

Van Ganse RF (1973) Immediate stabilisation of wet soils. In: Proceedings of the 8th international conference on soil mechanics and foundation engineering, Part 2, 233–237

van Jaarsveld JGS, van Deventer JSJ (1999) Effect of the alkali metal activator on the properties of fly ash based geopolymer. Ind Eng Chem Res 38:3932–3941

van Jaarsveld JGS, van Deventer JSJ, Lorenzen L (1998) Factors affecting the immobilisation of metals in geopolymerized fly ash. Metall Mater Trans B 29:283–291

Veith GH (2000) Engineering properties of sulfate bearing clay soil stabilized with lime activated GGBS, Ph. D. thesis, University of Glamorgan

Verdolotti L, Iannance S, Lavorgna M, Lumanaa R (2008) Geopolymerization reaction to consolidate incoherent pozzolanic soil. J Mater Sci 43:865–873

Weng X, Wang W (2011) Influence of differential settlement on pavement structure of widened roads based on large-scale model test. J Rock Mech Geotech Eng 3:90–96

Wild S, Kinuthia JM, Robinson RB, Humphires I (1996) Effect of GGBS on strength and swelling properties of lime stabilized kaolinite in presence of sulfates. Clay Miner 31:423–433

Xu H, van Deventer JSJ (2003) Effect of source materials on geopolymerization. Ind Eng Chem Res 42:1698–1706

Mehrotra SP, Kumar S, Bandopadhyay A, Kumar R (2008) An improved process for the production of geopolymeric material from fly ash. Australian Patent No. AU 2007200076 A1

Yaolin Y, Cheng L, Songyu L (2015) Alkali-activated ground-granulated blast furnace slag for stabilization of marine soft clay. J Mater Civ Eng 27:04014146

Zhang YS (2003) Research on structure formation mechanism and properties of high-performance geopolymer concrete. Ph. D. thesis, Southeast University, Nanjing, China

Zhang Q, Saito F (2012) A review on mechanically synthesis of functional materials. Adv Powder Technol 23:523–531

Zhang Z, Tao M (2008) Durability of Cement Stabilized Low Plasticity Soils. J Geotech Geoenviron 134:203–213

Zhang M, Guo H, El-Korchi T, Zhang G, Tao M (2013) Experimental feasibility study of geopolymer as the next-generation soil stabilizer. Constr Build Mater 47:1468–1478

Zivanovic B, Sreckovic T, Komlfemovic M (2002) The influence of mechanical activation on the process of reaction sintering of Portland cement clinker. Sci Sinter 34:95–100