Chapter 1
Studies on Geopolymer-Based Earthen Compacts

R. K. Preethi and B. V. Venkatarama Reddy

1.1 Introduction

Geopolymer mechanism involves the silicates and aluminates in the presence of alkali to undergo the process of geopolymerisation. Geopolymer products are originated by poly-condensation of aluminosilicates with alkali-activating metals yielding polymeric Si–O–Al bonds (Davidovits 1999; Duxson et al. 2007; Provis 2014). Earlier geopolymer was named as "Gruntosilikat" and "Gruntocement-geocement" (Gluchovskij 1959). Sodium hydroxide (NaOH) or potassium hydroxide (KOH) along with sodium silicate solution is used as an alkali activator solution in preparing geopolymer products (Davidovits 1988, 1994). Hardening process of the geopolymers in the presence of alkali metals takes place at the temperatures between 25 and 90 °C. Curing the geopolymer specimens beyond 90 °C results in the dehydration which will lead to the formation of cracks in the specimens (Hardjito et al. 2003; Khale 2007; Rovnanik 2010; Heah and Kamarudin 2011; Slaty et al. 2013).

Cement is the most commonly and widely used binder material in the construction industry. To reduce the consumption of cement in the building industry, alkali-activated products (Geopolymers) are emerging as alternative binder materials. Replacing Portland cement with geopolymer binder as an alternative in the conventional concrete has been attempted (Rangan 2008a, b, 2009; Hardjito 2004; Kunal Kupawade Patil and Allouche 2013). Geopolymer binders are energy efficient as they result in reduced carbon emission (Mclellan et al. 2011).

In the manufacturing process of clay bricks, clay is subjected to high temperature (1000–1400 °C) where the clay mineral changes from its natural form to a stable form

R. K. Preethi (✉) · B. V. Venkatarama Reddy
Department of Civil Engineering, Indian Institute of Science, Bangalore, India
e-mail: preethirk@iisc.ac.in

B. V. Venkatarama Reddy
e-mail: venkat@iisc.ac.in

B. V. V. Reddy et al. (eds.), *Earthen Dwellings and Structures*,
Springer Transactions in Civil and Environmental Engineering,
https://doi.org/10.1007/978-981-13-5883-8_1

called mullite (Grim and Bradley 1940). Burnt clay bricks possess high embodied energy (Reddy and Jagadish 2003; Praseeda et al. 2015). Production of masonry units using Portland cement, autoclaving or firing at higher temperature results in higher amount of energy consumption and carbon emissions. Alkali activation of natural clays and natural soils is an alternative method in the manufacturing process of masonry units (Munoz et al. 2015; Maskell et al. 2014). The current study is focused on exploring geopolymer binder using natural soil and clay minerals for the manufacture of masonry units.

1.2 Scope of the Study and Experimental Programme

The scope of the present study included the utilisation of geopolymer binders in the manufacturing process of masonry units. The earlier studies have indicated the benefit of using the geopolymer binders in manufacturing the masonry units. An attempt was made to examine the wet compressive strength of the alkali-activated earthen compacts in the presence of ground granulated blast-furnace slag (GGBS) and fly ash materials, with various molar concentrations of NaOH solution.

Different mix proportions were considered for casting the specimens. One set of specimens were cast by varying the clay content in the mix. Additional source of silica and alumina materials such as GGBS and fly ash was also used in casting the specimens. Second set of specimens were cast using GGBS and fly ash with fixed

Table 1.1 Details of the experimental programme

Materials	Clay (%)	GGBS or fly ash (%)	NaOH		
			8 M	10 M	12 M
Kaolinite/ Montmorillonite mineral	10	0	√	√	√
	15	0	√	√	√
	20	0	√	√	√
		4	√	√	√
		8	√	√	√
		12	√	√	√
		15	√	√	√
Red soil	20	0	√	√	√
	30	0	√	√	√
		5	√	√	√
		10	√	√	√
		15	√	√	√
		30	√	√	√
	41	0	√	√	√

clay content in the mix. The experimental programme considered in the study is given in Table 1.1.

1.3 Materials Used in the Study

The materials used in the investigations include locally available soil, river sand, natural clay minerals (kaolinite and montmorillonite), ground granulated blast-furnace slag (GGBS) and fly ash. Laboratory grade sodium hydroxide (NaOH) with 99% purity was used in the study.

The lime reactivity of GGBS and fly ash was tested as per IS: 1727–2004 code guidelines; the results were 9.74 and 2.99 MPa, respectively. Figure 1.1 gives the grain size distribution curves of kaolinite, soil, sand, soil with 20% clay fraction and 20% kaolinite in the mix used. It was difficult to obtain grain size distribution curve for montmorillonite clay mineral using hydrometer analysis. The natural soil has 41% clay fraction (<2 μm) containing predominantly kaolinite clay mineral. Kaolinite clay mineral possesses clay size fraction of 54.69%. The clay fraction (<2 μm) of the soil mix with 20% clay content and that of mix with 20% kaolinite are 18.73 and 10.96%, respectively.

The chemical composition and physical properties of some of the materials used in the study are given in Table 1.2. The elemental composition was determined by energy-dispersive X-ray (EDX) spectroscopy. Silica (Si) and alumina (Al) are the major components present in the materials.

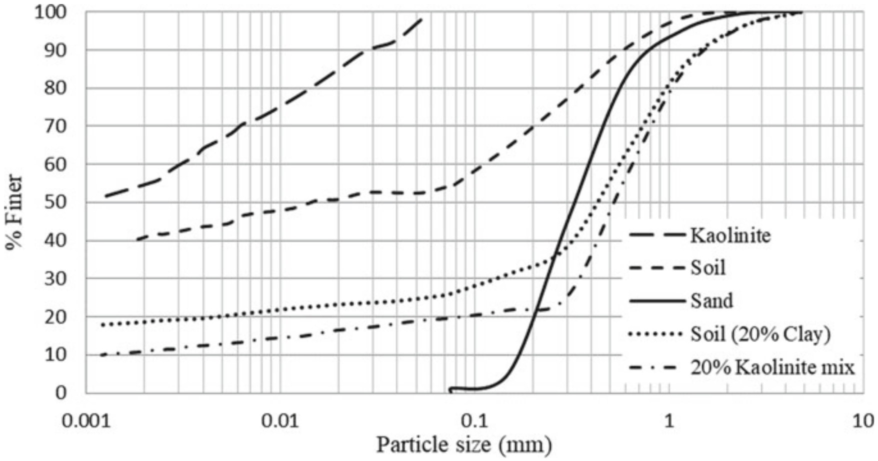

Fig. 1.1 Particle size distribution curve for river sand, natural soil, kaolinite, soil (20% clay fraction) and 20% kaolinite mix

Table 1.2 Chemical composition and physical properties of the materials used in the study

Element	Composition (% by weight)				
	Red soil	Kaolinite	Montmorillonite	GGBS	Fly ash
Al	15.68	22.13	10.79	10.87	20.85
Si	24.56	29.82	20.31	18.31	26.77
Ca	0.32	0.61	0.26	21.61	1.27
Fe	9.28	1.41	2.83	0.48	5.08
Ti	0.8	0.81	2.02	0.41	1.93
K	1.17	0.78	–	0.4	2.08
Mg	0.19	–	1.36	4.41	–
Na	–	–	2.11	–	–
S	0.3	–	–	0.65	–
Physical properties					
Specific gravity	2.68	2.63	2.39	2.91	2.28
Liquid limit	31	37.1	264.0	–	–
Plastic limit	19.48	18.87	158.0	–	–
Shrinkage limit	15.99	15.98	–	–	–

1.4 Casting and Testing Procedure

The effectiveness of geopolymer binders was evaluated through the determination of compressive strength using the cylindrical specimens of size 38 mm diameter and 76 mm height. Sodium hydroxide pellets were dissolved in the distilled water to prepare three different molar concentrations of 8, 10 and 12 M solution. The alkali solution was used after 24 h of its preparation.

1.4.1 Mixing and Casting

The materials were mixed in the dry state to achieve a homogenous mixture; later, the alkali activator solution was added to the dry mix. The moulding moisture content (MMC) (containing alkali and silica) was in the range of 10–15% of the dry mix. MMC depends upon the quantity of clay minerals in the mix. Higher percentage of clay demanded higher MMC to achieve a consistency needed for compaction. Mortar mixer was used in mixing the ingredients for 7 min to obtain the uniform mixture. The dry density of the specimens was controlled and kept at 1.8 g/cc. The cylindrical specimens were cast by compacting the partially saturated mix in a screw press.

1.4.2 Curing and Testing

Specimens after 24 h of casting were cured in an oven at 80 °C for 72 h. Cured specimens were dried in air for 24 h before testing. The specimens were tested for the wet compressive strength by soaking them in water for 48 h prior to the testing.

1.5 Results and Discussions

1.5.1 Alkali-Activated Earthen Compacts

The wet compressive strength of the alkali-activated natural soil (containing kaolinite clay) compacts was determined. The strength results are shown in Fig. 1.2. The figure shows the relationships between strength and clay content of the natural soil with varying molar concentrations of the alkali solution. The relationships show that the wet strength of the specimens increases with the increase in clay content in the mix, irrespective of the molar concentration. The strength and clay content are linearly related. There is about 50% increase in strength as the clay content was increased from 20 to 41%. Higher the clay content in the mix, more amount of reactive silica and alumina available, which resulted in the higher strength. Also, the increase in molarity of the activator solution increased the wet strength of the specimens. High alkali content (>12 M) and higher clay content in the mix result in maximum compressive strength for the soil compacts. The maximum strength obtained was 1.72 MPa with 12 M NaOH solution and with 41% clay fraction in the mix.

An attempt was made to examine the strength of alkali-activated compacts using pure clay minerals. The compacts were prepared using pure clay minerals (kaolinite and montmorillonite) and sand. The percentage of pure clay minerals in the mix was varied between 10 and 20%. Figure 1.3 shows the variation in wet compressive

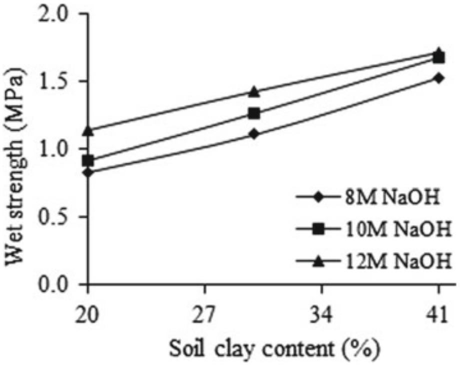

Fig. 1.2 Wet compressive strength of alkali-activated natural soil compacts

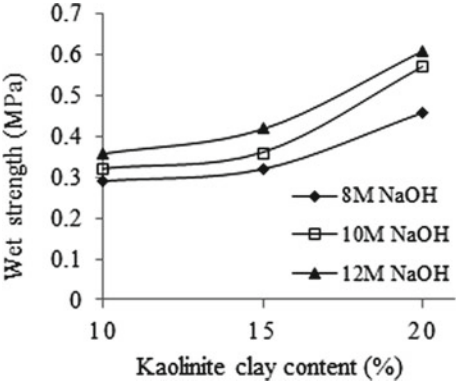

Fig. 1.3 Variation in wet compressive strength of clay–sand compacts

Fig. 1.4 Disintegrated montmorillonite compacts when soaked in water for 48 h

strength with kaolinite mineral as well as molarity of the alkali activator solution. Strength increases with increase in clay content. There is about 70% increase in wet strength as the clay percentage was increased from 10 to 20%. The strength increases marginally (10–12%) as the molarity was increased from 8 to 12 M. The maximum wet strength of 0.61 MPa was obtained with 20% kaolinite and 12 M molarity. This strength is nearly half of that obtained using natural soil using similar clay content and 12 M solution.

The compacts using montmorillonite clay mineral disintegrated upon soaking in water for 48 h prior to the testing. Figure 1.4 shows the condition of compacts using montmorillonite upon soaking in the water. The compacts using natural soil and the pure clay minerals form lumps upon mixing with higher molarity alkali solution (>12 M) as shown in Fig. 1.5. It becomes very difficult to prepare cylindrical compacts using such a lumpy mass.

Fig. 1.5 Increase in alkalinity and clay content resulted in the lumpy mix

1.5.2 Effect of Using GGBS and Fly Ash as Additional Source of Silica and Alumina

The wet strength achieved using soil and pure clay minerals was low (<1.75 MPa). Hence, addition of GGBS and fly ash was explored to improve the strength of the compacts.

1.5.2.1 Effect of Using GGBS

Natural soil with 30% clay fraction and the mix using natural clay and sand containing 20% kaolinite clay were used in preparing the compacted specimens. For natural soil compacts, GGBS was varied between 5 and 30%, and for kaolinite clay compacts, it was varied between 4 and 15%. The strength results of natural soil compacts and compacts with 20% kaolinite clay using GGBS are shown in Figs. 1.6 and 1.7, respectively. The figures show the strength variation in the compacts with GGBS and varying molarity of the activator solution. The following points emerge from the strength results shown in the figures;

- The strength varies linearly with GGBS content. As the GGBS content increased, the wet strength increased irrespective of the molarity of the alkali activator.
- The maximum wet compressive strength achieved was 7.68 MPa with the soil compacts, where soil with 30% clay content, 30% GGBS and 12 M NaOH solution were used. Increase in GGBS content from 5 to 30% results in about two-times increase in wet strength.
- Molarity of alkali solution has strong influence on the strength at lower dosages of GGBS. Its effect reduces at higher GGBS contents. For example, at 5% GGBS the strength of the soil compacts with 30% clay increased by about 50% as molarity

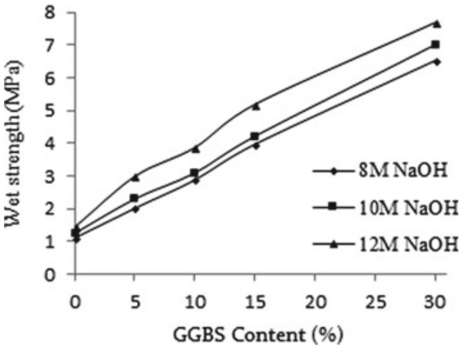

Fig. 1.6 Variation in wet strength with GGBS content for soil compacts with 30% clay

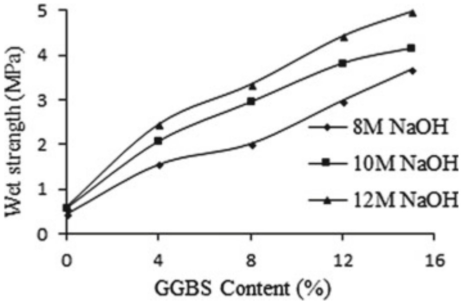

Fig. 1.7 Variation in wet strength of specimens with 20% kaolinite and with GGBS content

was increased from 8 to 12 M, whereas at 30% GGBS content, there is only 25% increase in strength.

- The maximum wet compressive strength of the compacts with 20% kaolinite was 4.96 MPa with 15% GGBS and 12 M NaOH. Strength increased by about 100% with the increase in GGBS content from 4 to 15%. Increase in molarity of the activator solution from 8 to 12 M increased the strength of the 20% kaolinite specimens. The strength increases by about 60% and 35% at 4% and 15% GGBS contents, respectively.

1.5.2.2 Effect of Using Fly Ash

The strength of alkali-activated soil compacts with 30% clay fraction as well as with 20% kaolinite clay and using fly ash was examined. The fly ash content was varied between 5 and 30% with soil compacts and between 4 and 15% with kaolinite compacts. The variation in compressive strength of soil compacts and pure clay compacts using fly ash is shown in Figs. 1.8 and 1.9, respectively. The figures show the relationships between strength and fly ash content with varying molar concentrations. From these results, the following observations can be made:

- Increase in fly ash content increased the wet strength of the compacts. There is a linear relationship between the strength and the fly ash content irrespective of the molar concentrations of the alkali activator.
- Strength increases by about 1.6 times as the fly ash content was increased from 5 to 30% in natural soil compacts. The maximum strength obtained was about 6 MPa with 15% fly ash and 12 M molarity with 30% clay fraction. Compressive strength of the compacts using natural soil increased as the molarity increased from 8 to 12 M. Strength increased by about 60% and 46% at 5% and 30% fly ash content with increase in molarity from 8 to 12 M.
- Increase in fly ash content from 4 to 15% resulted in 100% increase in wet strength of the compacts with 20% kaolinite, irrespective of the molarity of the solution. The maximum strength obtained was about 3 MPa with 15% fly ash. It was observed that the strength increased by about 70% with the increase in molarity from 8 to 12 M at 4% fly ash content and that about 60% at 15% fly ash content.

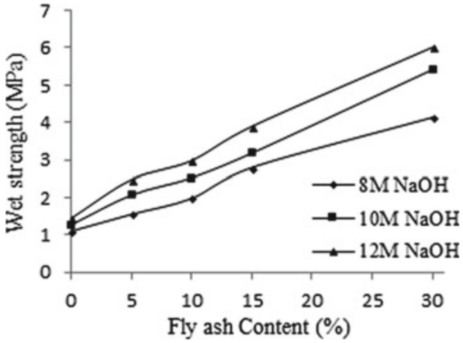

Fig. 1.8 Wet strength variation of soil compacts with 30% clay and with fly ash content

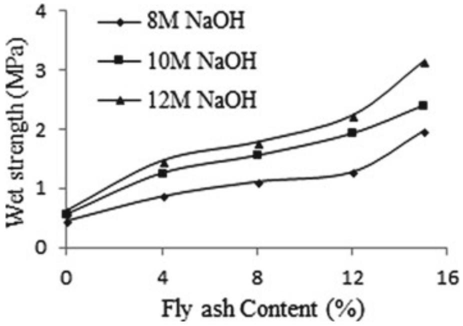

Fig. 1.9 Wet strength variation of kaolinite compacts with 20% clay and fly ash content

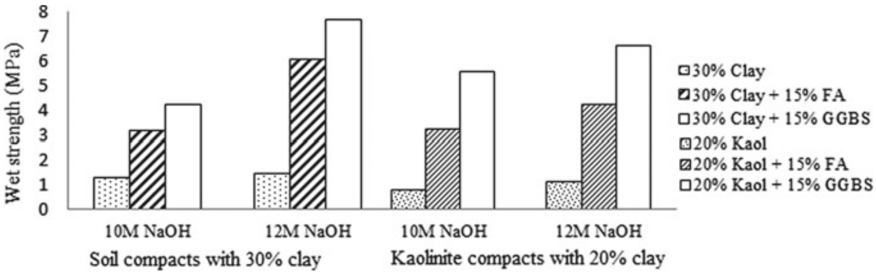

Fig. 1.10 Variation in wet strength of geopolymer compacts with natural soil (30% clay) and 20% kaolinite with 10 and 12 M activator solution using GGBS and fly ash (FA)

1.5.2.3 Comparison of Results Using GGBS and Fly Ash

Addition of GGBS and fly ash as an additional source of silica and alumina plays a key role in improving the wet compressive strength of the specimens using natural soil and kaolinite mineral. The strengths increased with the addition of GGBS and fly ash. The comparison of compressive strength results of soil compacts with 30% clay fraction and compacts with 20% kaolinite using GGBS and fly ash with 10 and 12 M alkali activator are shown in Fig. 1.10. Addition of GGBS resulted in higher strength when compared with the compacts using fly ash. The increase in strength using GGBS is associated with the high lime reactivity of GGBS than that of fly ash and with the high calcium content present in the GGBS, the presence of calcium ensures the lime pozzolana reaction along with geopolymerisation. The GGBS shows higher lime reactivity than fly ash. This is attributed to the fact that the higher lime reactivity is due to the presence of higher quantity of reactive alumina and silica. Hence, the material has more tendencies to react with the alkali activator used. The specific surface area of GGBS is >500 m^2/kg and that of low-calcium fly ash ranges between 200 and 300 m^2/kg (Mehta and Monterio 2014). Finer material possesses higher pozzolanic activity and results in formation of more cementitious products.

1.5.3 Efflorescence on the Specimens

Alkali-activated cylindrical specimens showed the efflorescence on the surface prior to curing. Deposition of salts on the cylindrical specimens with kaolinite clay and natural soil before curing is shown in Fig. 1.11a, b respectively. The sodium-rich solution evaporates from exposed surface of the cylinders, leaving the salts as white deposits on the surface. Leaching of salts was also observed when the specimens were soaked in water for 48 h prior to the testing. Excessive leaching of salts is a serious concern. There is a need for understanding the effect of leaching on the strength and durability of alkali-activated compacts.

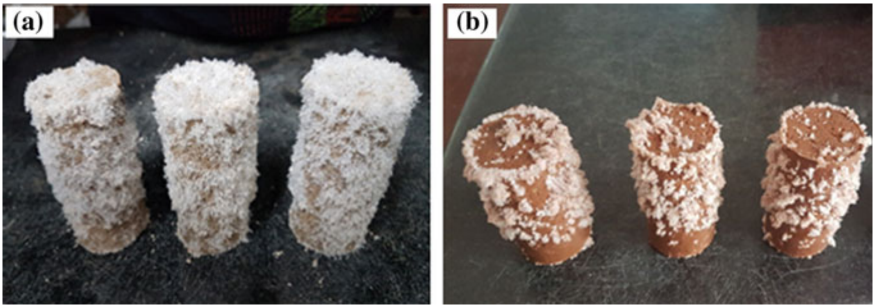

Fig. 1.11 Deposition of salts, **a** kaolinite compacts and **b** soil compacts

1.6 Conclusions

Tests on various mix proportions indicate a linear relationship between strength and clay content. Strength increased with increase in clay fraction in the mix and with increase in molarity. Alkali-activated natural soil compacts resulted in wet strength <2 MPa. Pure clay specimens showed very low strength and are practically insufficient. The specimens using kaolinite clay resulted in very low strength of 0.61 MPa with 20% clay fraction and 12 M solution in wet condition. Cylindrical specimens with montmorillonite disintegrated when soaked in water prior to the testing.

The wet strength of alkali-activated earthen compacts improved with the addition of fly ash and GGBS as an additional source of silica and alumina. The maximum wet strength of cylindrical specimens achieved was 7.68 MPa with the soil compacts, where the red soil with 30% clay content, 30% GGBS and 12 M sodium hydroxide solution. The specimens with kaolinite showed up to 5 MPa wet compressive strength with 15% GGBS and 20% clay fraction. Test results reveal that strength increases with increase in GGBS or fly ash content. High strengths were obtained with the use of GGBS rather than fly ash due to high lime reactivity of GGBS.

MMC to achieve a homogenous mixture was controlled by the clay fraction in the mix. Increase in clay content and increase in alkalinity (>12 M) of the solution resulted in the formation of lumpy mix, which is difficult to compact.

Alkali-activated specimens showed salt deposition on the surface prior to curing. Leaching of salts was observed when the specimens were soaked in water for 48 h prior to the testing. Addressing the leaching in an earth-based geopolymer products is a serious concern.

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