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FABRICATION OF GEOPOLYMERS FROM CLAYS USING MINERAL INORGANIC POLYMERIZATION TECHNIQUE

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Abstract - *In many civil engineering constructions, the soft soils are often stabilized with chemical stabilizers like Ordinary Portland Cement and lime. The production processes of traditional stabilizers are energy intensive and emit a large quantity of CO_2. Geopolymers, with its high strength, low cost, low energy consumption and CO_2 emissions during synthesis, offers a promising alternative to OPC. In this work, a geopolymer has been fabricated from Kaolin clay (precursor), silica sand (filler), alkaline activator namely sodium hydroxide and distilled water. Homogeneous samples of known amounts of materials were mixed with different ratios of clay, sand and NaOH solutions ranging from 11 to 14 mol/l. To accelerate the chemical polymerization reactions, the molded reactants were cured at 80°C in oven for 24 h to form stable and hard geopolymeric material. The fabricated polymers were subjected to uniaxial compression and tension testing under dry and saturated conditions. Based on the results of experiment, a geopolymer with compressive strength of 20.6 MPa and tensile strength of 85 kPa was achieved by clay to sand ratio of 1.8 and NaOH of 13 M under dry conditions and 16.6 MPa and 55 kPa under saturated conditions. The suitability of using Kuttanad clay as precursor had also been evaluated on basis of mechanical strength and was found to be respectively of 0.25 and 0.31 times. Water absorption of fabricated polymers was seen to be relatively low and inversely proportional to NaOH concentration. Geopolymerization also reduced the Liquid limit and Plastic limit values of the clayey soils.*

***Key Words*: Geopolymer, Kuttanad Clay, NaOH, Compressive Strength, Tensile Strength.**

1. INTRODUCTION

Soft or highly compressible soils are often encountered on many civil engineering project sites, which lack sufficient strength to support loading either during construction or throughout the service life. To improve strength and stiffness of those less competent soils, chemical stabilization with cementitious materials like OPC and lime has been widely practiced. A major issue with conventional soil stabilizers is that their production processes are energy intensive and emit a large quantity of CO_2. Therefore, civil engineering industry is always searching for new, viable sustainable alternatives to replace Portland cement as soil stabilizers. It has been shown worldwide that Mineral Inorganic Polymerization Technique (MiP) seems to meet the above requirements. This technique refers to the reactions between alkalis and a group of minerals, which are rich in aluminosilicates and are produced at atmospheric pressure and ambient temperatures by using minimal energy input. In this project, a new geopolymeric material, as a replacement to conventional soil stabilizer, is synthesized from the following components: kaolin clay mineral (rich in aluminosilicates), silica sand, NaOH, and distilled water, using MiP technique. In addition, possibility of geopolymers manufactured using Kuttanad Clay is also investigated. Homogeneous samples of known amounts of materials mixed with different ratios of clay, sand and NaOH solution of concentration ranging from 11-14 mol/l, would be cured at 80°C in an oven for 24 h to form stable and hard geopolymeric material. The quality of the fabricated geopolymers is evaluated according to their compressive strength and tensile strength.

1.1 Scope

- Effective key for facile synthesis and characterization of geopolymers from locally available clays characterized by sustainability and low cost construction.
- Experimental feasibility study of clay-based geopolymers as the next generation soil stabilizer.

2. LITERATURE SURVEY

In 1970, Davidovits addressed the synthesis of geopolymer from inorganic material composed of silica and alumina. They are essentially produced from materials rich in aluminosilicates such as kaolinitic clay, metakaolin, fly ash, mixed with hydroxide alkali and alkali silicate solutions at ambient conditions [1]. Many studies have utilized kaolinite or metakaolinite as a secondary source of soluble Si or Al in addition to waste or natural aluminosilicates materials to synthesis geopolymers [10]. Chakchouk [2] studied the physical properties of Tunisian clay samples from different locations. Clays rich in kaolinite have demonstrated the highest resistance and best pozzolanic activity. Nmiri [9] synthesized kaolinite-based geopolymer with both NaOH and KOH solutions, followed by its characterization. Produced geopolymers showed good mechanical and physical properties. The water absorption was relatively low, which provides us with the information about the long term durability of the material. Faqir [8] focused on a fabrication of sustainable and low cost construction geopolymeric material using kaolin clay by applying the technology of MiP. This geopolymer showed a compressive strength of 27.1 MPa (dry) and 18.1 MPa (wet).

Parameters, such as NaOH concentration and curing regime, are important parameters that must be taken into consideration while designing a kaolin-based geopolymer product for a specific application. In the preparation of geopolymer with alkaline activators NaOH and KOH conducted by Nmiri [8] the optimum concentration of KOH was found to be 15 M and that of NaOH to be 13 M. The decrease of compressive strength when the NaOH concentration is above 13 M, could be explained by the formation of cracks on the cured geopolymer samples resulting from efflorescence caused by the reaction of excess sodium hydroxide with atmospheric CO_2. Alshaaer [7] cured the kaolin-based geopolymers at different temperatures: 80°C, 105°C and 130°C. There were no significant changes in compression strength, density or water absorption of specimens due to increasing curing temperature.

However, from the previous studies, it is clear that no effort is taken to judge the quality and suitability of locally available clayey soils for the fabrication of geopolymers. From the study on the composition and fabric of Kuttanad clay by K Suganya [5], it was found that soil has unique combination of Metahalloysite, Kaolinite, Iron and Aluminium oxides. Almost all clayey soils contain aluminosilicates which are the main constituent of geopolymer. Hence more chances are there for fabricating geopolymers with comparable strength and durability. Furthermore, strength and quality of geopolymer as a soil reinforcement material should not only be evaluated based on compressive strength, but also on grab tensile strength.

3. METHODOLOGY

3.1 Materials

Table -1: Basic Properties of Soil

Sl No	Properties	Kaolin Clay	Kuttanad Clay	Silica Sand
1	Water Content %	20	92	17
2	Specific Gravity	2.45	2.32	2.56
3	% of Clay	55	71	-
4	% of Silt	45	21	39
5	% of Sand	-	8	61
6	Liquid Limit %	54	116	15
7	Plastic Limit %	29	47	13
8	Plasticity Index %	25	69	2
9	Maximum Dry Density (g/cc)	1.68	1.2	1.74
10	Optimum Moisture Content (%)	20	45	15
11	Compressive Strength (kPa)	189	75.87	-
12	Angle of Internal friction (Φ)	-	-	38.5°

The geotechnical properties of soil samples were tested as per Indian Standard specification (IS 2720) and as shown in table 1. NaOH solution was used as an alkaline activator for the dissolution of aluminosilicate phases. Analytical grade of sodium hydroxide pellets was used to prepare fresh solution with concentration varying from 11-14 mol/l (440-560 g) before each run. Distilled water was used for the preparation of solutions.

3.2 Fabrication of Geopolymers

Table -2: Design of experiment for the synthesis of geopolymer

Components	ID	Ratio / Concentration
Kaolin Clay/Kuttanad Clay : Sand	A1	1:2
	A2	3:4
	A3	4:3
	A4	3:2
	A5	9:5
	A6	2:1
	A7	5:2
NaOH	B1	11 M
	B2	12 M
	B3	13 M
	B4	14 M

All the materials required for the experiment were crushed and grinded to a particle size passing through 425 µ. The NaOH solution was prepared by adding the water to the NaOH crystals in plastic bottle. Homogeneous samples of known amounts of clay minerals and sand were mixed with different ratios of clay/sand and NaOH concentrations. The synthesis parameters such as mass of clay, sand, NaOH and water are varied in order to identify the optimum conditions for the synthesis of the mineral polymeric material as shown in table 2. The used amount of water is limited by the plastic limit of clay [8], which is determined to be around 29% and 47% for kaolin clay and Kuttanad clay respectively.

For the formation of a strong alkali solution, the aqueous solution of NaOH and distilled water was mechanically mixed for 1 min until the alkali solution cooled down to room temperature. Silica sand and clayey soils were dry mixed first and then prepared alkali solution was added. All reagents were mixed for 15 min to form a slurry paste. The final mixture of each series was divided into 24 specimens (95g each). The mixture was moulded in cylinder of 38 mm diameter and 76 mm height, immediately after weighing to avoid drying and decrease of the workability of the mixture. The specimens were dried for 24 h at 80° C in a well-controlled ventilated oven to allow for the geopolymerization reactions to take place. The samples were sealed with thin plastic layers during the curing stage to prevent moisture loss. After curing, the moulded specimens were removed from the oven and allowed to cool down at room temperature as shown in figure 1. Finally, the specimens were later weighed. The obtained specimens were divided into two groups, each of 12 specimens. The first group was left at room temperature for 7 days (dry specimens), and second one was immersed in a tap water for 7 days (wet specimens).

Fig- 1: Fabricated Geopolymers from Kaolin Clay (left) and from Kuttanad Clay (right)

4. COMPRESSIVE AND TENSILE STRENGTH

Compressive strength and tensile strength of the specimens were determined using ASTM D2166 by breaking cylindrical specimens in a uniaxial compression testing machine shown as in figure 2. Three specimens were used for each run and the average value was calculated. Tensile strength was obtained by fixing the samples on to the plate of compression testing machine with help of clampers.

Fig- 2: Uniaxial Compression Testing Machine

Kaolin-based geopolymers had obtained a value of maximum compressive strength of 20.6 MPa for dry specimens and 16.6 MPa for wet specimens, whereas the Kuttanad clay-based geopolymers attained compressive strength of 5 MPa and 3 MPa for dry and wet specimens respectively. It is observed from figure 3 that increasing the mass of clay until less than twice the mass of sand or decreasing the mass of sand and increasing the concentration of NaOH will increase the compressive strength of the produced geopolymeric material. But in case of tensile strength, geopolymers with mass of clay less than that of sand had attained maximum tensile strength of about 85 kPa (dry) and 55 kPa (wet) for kaolin and about 26.5 kPa (dry) and 13.2 kPa (wet) for Kuttanad geopolymers. The increase in tensile strength of geopolymers as mass of sand is increased may be caused by the significant role of silica content in sand. Therefore, for obtaining a geopolymeric material with maximum strength clay mineral which is the precursor and silica sand used as filler should be taken in equal amounts.

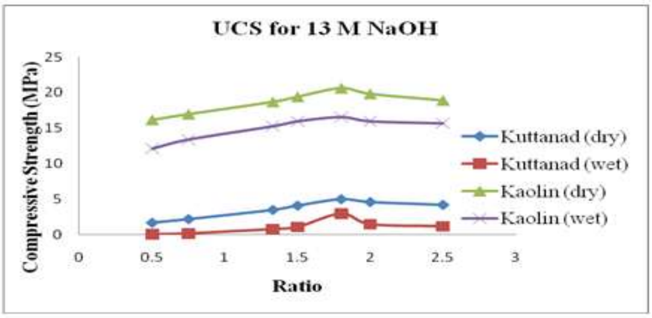

Chart -1: Variation of Compressive strength for different ratio of clay to sand

In the present experimental investigation done to evaluate the strength property of Kuttanad clay for fabricating geopolymers, it was observed that the produced geopolymeric material attained compressive strength of 0.25 times and tensile strength of 0.31 times that of kaolin-based geopolymers. Therefore, Kuttanad clay can be used as replacement either partially or fully to kaolin clay to produce geopolymers.

4.1 Optimum Concentration of NaOH

The results show that the maximum value of compressive and tensile strength is reached at 13 M concentration of NaOH and as shown in figure 4. Beyond this optimum, the strength decreases which could be explained by the formation of cracks on cured geopolymer samples resulting from efflorescence caused by the reaction of excess NaOH with atmospheric CO_2. Figure 5 shows the formation of efflorescence on the surface of cured geopolymer samples which induces crack in the materials.

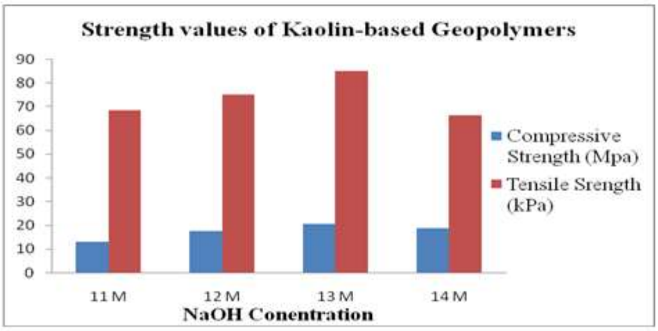

Chart -2: Variation of NaOH concentration for strength values of kaolin-based geopolymer

Fig- 3: Cracks induced and failure pattern of the samples caused by efflorescence

4.2 Comparison of Strength of Geopolymers tested under dry and saturated conditions

Compressive strength and tensile strength for different specimens of fabricated geopolymers were tested under dry and saturated conditions. Values of compressive strength ranging from 7.7-20.6 MPa for kaolin and 0.01-5 MPa for Kuttanad were obtained under dry conditions whereas, under saturated conditions, it was observed to be in the range of 4.2-16.6 MPa for kaolin and 0.002-3 MPa for Kuttanad geopolymers. Hence we can say that a geopolymer placed under saturated conditions would cause decrease in their strength whereas geopolymers cured at dry conditions have attained similar potency of compression to that of a conventional concrete material (28 MPa) and tension to that of a commercially available geopolymer (9-180 kPa).

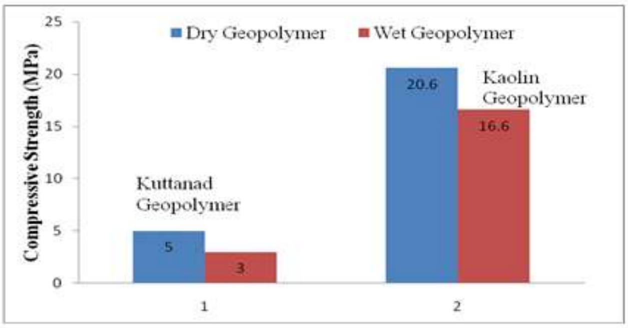

Chart -3: Comparison of Compressive strength for dry and wet samples

5. PHYSICAL PROPERTIES

The geotechnical properties of the fabricated geopolymers such as water absorption, specific gravity and consistency limits were found out as per IS 2720. From the test results, G was found to be in the range of 2-2.4 which is in the limit of a geotextile (1.9-2.9).

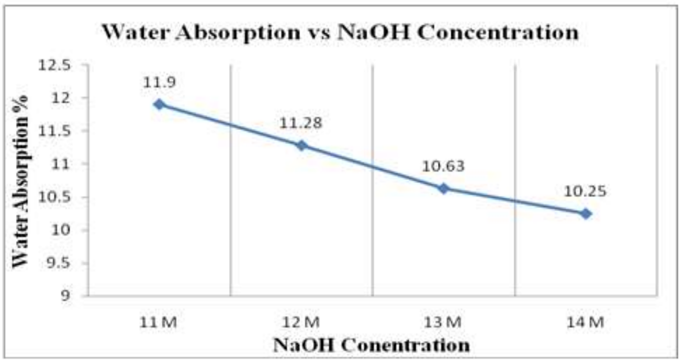

Chart -4: Variation of Water Absorption (%) with NaOH Concentration

Water absorption is an important property that influences the durability of the geopolymer. The produced geopolymer showed stability in water with no disintegration. From the investigation, it has been observed that all absorption values of the produced geopolymers were in the range of 10.2% to 12%, which were lower than the limit of ASTM C90 standard specification of 17 wt. %. As it is seen from figure 7, the water absorption exhibits a decreasing trend by increasing the NaOH content from 11 M to 14 M. This is a result of formation of strong geopolymeric network. This network closes a part of the pores and bonded the clay and filler particles together.

Liquid limit and Plastic limit are significant indices quantifying the response of clay to water. Figure 8 shows the effect of geopolymerization on clayey soils with clay to sand ratio of 1.8 and for 13 M of NaOH. It can be seen that geopolymerization tends to reduce both the LL and PL of clayey soils. This reduction in LL and PL can be attribtuted to the role of the silica sand which was used as the filler in the fabrication of geopolymers. It can also be attributed to the role of chemicals involved in the alkaline activator before the geopolymer gel formation, which tends to assist the sliding between the dry particles.

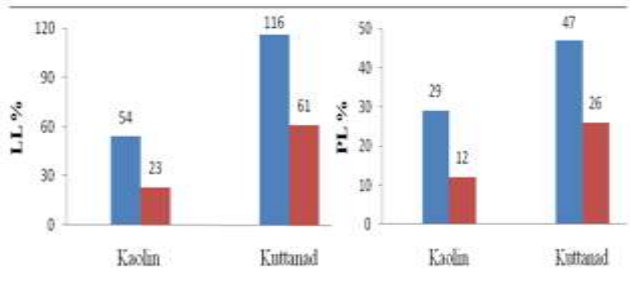

Chart -5: Effect of Geopolymerization on LL and PL of Clayey soils

6. CONCLUSIONS

In order to judge the suitability of Kaolin clay and Kuttanad clay to fabricate geopolymers, the mineral polymers were synthesized from clay minerals, silica sand as filler and alkaline activator as NaOH, using MiP technique. Synthesis parameters such as percentage of kaolin and Kuttanad clay, silica sand and concentration of sodium hydroxide were studied and optimized to achieve materials with high mechanical properties. The following conclusion can be drawn from this work: -

1. The mechanical properties of the produced geopolymer, such as compressive strength and tensile strength are comparable with that of conventional concrete material and commercially available geopolymer respectively.
2. The compressive strength and tensile strength of Kuttanad-based geopolymer was found to be 0.25 and 0.31 times that of the Kaolin-based geopolymer. Hence Kuttanad Clay can be used as precursor to fabricate geopolymer.
3. The mechanical properties of the fabricated geopolymers were found to be increasing with increase in NaOH concentration up to 13 M beyond which causes reduction in strength due to effect of efflorescence.
4. An optimum value of 20.6 MPa and 85 kPa respectively of compressive strength and tensile strength were obtained when kaolin to sand ratio was 1.8 under dry conditions.
5. The compressive strength and tensile strength obtained using saturated conditions was lower than that of dry condition.
6. For all samples, the water absorption is relatively low and was found to be inversely proportional to NaOH content. LL and PL values of clayey soils had also been found to be reduced after the geopolymerization.
7. The advantage of utilizing this geopolymerization technique to naturally occurring clay is the low cost of raw material and the comparable mechanical properties of the produced material to that of conventional ones. This study clearly promotes the use of geopolymer as an effective binder to traditional OPC in soil stabilization.

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