Effect of Adding Metakaolin Based Geopolymer to Improve Soft Clay Under Different Conditions

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Abstract. Soft clay soil spreads in Iraq and some countries globally; this type is usually with low compressive strength. Therefore, soft clay shows problematic behaviors. Improving soft soil with chemical additives is one of the suitable methods. Thus, the focus of this study was to investigate the usage of Metakaolin-based geopolymer in geotechnical engineering to enhance the properties of soil. Three percentages of MK were used in the present study, which are 8%, 10%, 12%, and 14%. Unconfined Compressive Strength UCS, Scanning Energy Microscopy SEM, and X-Ray Diffraction XRD had to be conducted in untreated and treated soil to illustrate the effect of MK on soil stabilization. The results show that the optimum value of strength was observed with the addition of 10% MK cured for 28 days under 80°C the strength of soil increase by 93.4 %. SEM test for treated soil altered the texture of clay soil by reducing the cracks. XRD pattern for treated soil clarified the intensity of the peaks obtained with calcite and quartz.

Keywords: soft clay, Metakaolin-based Geopolymer, chemical additives, improvement.

1. Introduction

Soft clay is one of the problematic soils that face many geotechnical engineers in the field. This soil is commonly associated with a change in water content that leads to a reduction in shear strength causes swelling, shrinkage, settlement, consolidation, which can cause severe damage to the civil engineering building. Several techniques are used to improve soft clay (i.e., stone columns are ideally suited for structures with extensive loads) [1]. Also, traditional binder (i.e., cement, lime, slag, and others) was used widely worldwide. Mixing of soil and cement in the presence of water improves soil properties like an increase in internal friction, decrease in shrinkage-swelling behavior, and reduction in the settlement [2]. The hydration products are responsible for strength gain in the soil-cement system by holding soil particles within themselves [3]. Nowadays, several materials are available to stabilize soft clay soil (Fly ash, rice husk ash, reeds) are used alternatively, of expensive lime and cement [4-5]. Geopolymer technology was firstly published by Joseph Davidovits more than 30 years ago [6-7].

Cristallo et al., 2012 used fly ash-based Geopolymer to stabilize sandy soil for self-compacting rammed earth structure conducted by UCS. The samples cured at 80 °C by curing time-examined are 1day, 3 days, and 7 days by adding some concrete admixtures such as calcium hydroxide, sodium chloride, and concrete superplasticizer. The results illustrated an optimum liquid over total solids ratio; no enhancement was observed when concrete admixtures mentioned above were used [8]. Zhang et al. [9] studied the potential of Metakaolin based geopolymer, and the results showed the unconfined compressive strength of Metakaolin based geopolymer improved soils are extremely higher than the soil and higher than 5% Portland cement stabilized soil when Metakaolin based Geopolymer additions are higher than 11%, and the strength increase from 7day to 28 days curing is non-significant [9]. Also, the effect of temperature on the strength of soft clay soil improved with fly ash-based geopolymer had
studied by [10, 11]; the results show the gain of strength for treated soil is influenced by temperature and concentration of hydroxide sodium. Based on the previous studies, the soil improvement using Geopolymer additives was limited and can be used as a new technique in the next years.

2. Materials

2.1 Soil
Soft clay soil used in this study was brought from Al-Bawya Village/ Diyala Governorate/ Iraq. Basic tests were performed to investigate natural soil's physical and chemical properties, as given in Table 1.

| Property                        | Index value | Specification   |
|---------------------------------|-------------|-----------------|
| Specific gravity                | 2.72        | ASTM D854       |
| Liquid limit                    | 39          | ASTM D4318-00   |
| Plastic limit                   | 25          | ASTM D4318-00   |
| Plasticity index                | 14          |                 |
| Passing No.200                  | 96%         |                 |
| Percent of sand                 | 4%          | ASTM D422       |
| Percent of clay                 | 56%         | ASTM D422       |
| Percent of silt                 | 44%         | ASTM D422       |
| Maximum dry density             | 16.8 kN/m³  | ASTM D          |
| Optimum moisture content        | 18.4 %      | ASTM D698       |
| Unconfined compressive strength | 0.433 MPa   | ASTM D2166      |
| USCS Classification             | CL          | ASTM D2487      |

2.2 Metakaolin
Is an Aluminosilicate material with a dull luster in white color is active thermally, which is present in Metakaolin, as calcining kaolinite clay within the range of temperature 650-800°C. The chemical reaction of kaolinite forming Metakaolin as follows:

\[
2\text{Al}_2\text{Si}_2\text{O}_5 (\text{OH})_4 \rightarrow \text{Al}_2\text{Si}_2\text{O}_7 + 2\text{H}_2\text{O}
\]

Kaolinite Metakaolin

2.3 Alkali activators
MK is considered a secondary binder. Therefore, two types of activators were used: the liquid alkaline activator by mixing sodium silicate and sodium hydroxide [12].

2.4 Sodium hydroxide
Also known as caustic soda, is an organic compound with the chemical formula NaOH. Sodium hydroxide used is commercially manufactured in flakes form. That flakes should be dissolved in distilled water at a specific weight to achieve the desired molar concentration. The molar concentration of sodium hydroxide used for activation was 10 M.

2.5 Sodium silicate
Also known as water glass or liquid glass with chemical formula Na2SiO3. Sodium silicate was originally in solution form.

2.6 Water
The distilled water was used in the experimental work.
3. Methodology

3.1 Samples preparation
Soil specimens for the unconfined compressive strength test were made up using a cylindrical mold with an inner diameter of 44 mm and a height of 100 mm. Firstly alkali solution was prepared by mixing silicate sodium with hydroxide sodium with a ratio of ½ due to the viscosity of silicate sodium. Dry mixture was prepared by mixing a 330 g of dry with several percentages of Metakaolin (MK) (8%, 10%, 12%, and 14%) by weight of the dry soil. This mixing is prepared homogenously. After that, the alkali solution added 38% to the dry soil-geopolymer mix and mix all components well to be homogenous.

3.2 Curing conditions
Samples were placed in the oven for two hours, at temperatures 20°C and 80°C, and then stored in the curing chamber with a room temperature of about 23 °C to complete-time 1 d, 3d, 7d, 14d, and 28 days. Figure 1 shows the curing chamber.

4. Experimental work

4.1 Unconfined compression strength test (UCS)
After the samples were cured, they were assessed with unconfined compressive strength according to ASTM D-2166 [13]. Generally, this test is used to determine the strength of treated samples. The load is applied as axial on the samples until failure with a loading rate 2% mm/min and records the maximum load and axial deformation after failure to plot the stress-strain curve.

4.2 Scanning electron microscopy test (SEM)
This test was used to study the microstructure properties and mechanical behavior of treated samples with MK. SEM was used to observed clay structure and analyze the pore space of different treated soil. SEM was conducted by using Tescan VEGA 3 SB operated at 20 kV.

4.3 X-Ray Diffraction (XRD)
This test is used to determine the mineralogical composition of the natural soils and treated soils to characterize the mineralogical changes and confirm the treated soil's resulting gels.

5. Results and discussion

5.1 The Effect of MK percentage on UCS
The samples were tested after cured of 1d, 3d, 7d, 14d, and 28 days to evaluate their strength. Figure 2a shows the effect of Metakaolin-based Geopolymer on UCS of treated soil with 8%, 10%, 12%, and 14% MK by weight of dry soil at temperature 20°C after curing time. Figure 2b shows the Effect of MK percent on UCS at temperature 80°C. Results of specimens cured for 28 days for an initial duration 2 hrs under 80 °C indicate the lowest percent 8% MK was observed to be 89% higher compared with
untreated soil while the intermediate percent 10% MK and 12% MK were shown much higher strength increase by about 93.4% and 91.7% respectively. Higher percent 14% shows an increase in strength of approximately 91.8% compared with untreated soil. From the UCS result, the intermediate addition of 10% MK was found to be sufficient for increasing the strength of soft soil. Some percentages indicate ineffective due to the exhaustion of different components in the reaction environment; this is also recommended by [14].

5.2 The Effect of Temperature on UCS
In general, the influence of temperatures affects the simulation and acceleration of pozzolanic reactions of the geopolymer, which enhances its bonding and gain strength even in a low curing time. The effect of high temperature is significant as shown in Figure 3. The results reveal the high values of UCS produce with temperature 80°C with most percent.

![Graph showing effect of temperature on UCS](image)

**Figure 2.** The Effect of Metakaolin-based Geopolymer on UCS of treated soil at 20°C and 80°C.

5.3 Stress-strain curve
Figure 4 represented the stress-strain attitude of natural and treated soil with specific concentrations of MK under temperature 20°C by initial duration for 2hr and completed curing time for 28 days. Firstly, the stress gradually develops with the increase in axial strain. After achieving peak stress, it decreases with the increase in axial strain for all the treated and untreated soil samples. The maximum values of UCS recorded with the addition of 10% MK, and 12% MK confirmed these percent produce the best and high results for UCS comparison with untreated soil. Besides, Fig. 4b shows the stress-strain behavior for samples untreated and treated at 80°C. The results reveal the peak of UCS is higher compared with samples treated at 20°C.
Figure 3. The variation between temperatures 20°C and 80°C of treated soil along curing time.

Figure 4. Stress-strain curves for natural and treated soil curing after 28 days at 20°C and 80°C.
5.4 Scanning electron microscopy (SEM)
SEM tests are conducted to observe the microstructure characteristics and hydration products in the bulk binder phase. Figure 5 shows the SEM images of samples at curing time 7d with 12% MK contents at various amplifications. Figure 5a shows the SEM image with 500x amplification for untreated soil that illustrated the microstructure of the mixture without Metakaolin-based Geopolymer additive is loose and contains a lot of void pore. While Fig. 5b shows the soil treated with 12% with curing time at 7 days with 500x amplification, indicating the additive substance is clear. After all, it is considered a binder because it contains sodium silicate, which leads to a decrease in cracks. As well as, Figures 5c and 5d show the untreated soil with 2x amplification and the treated soil with 12% MK also with 2x amplification to illustrate the binder of soil particles.

![SEM images of soil samples](image)

(a) Untreated soil with 500x amplification  
(b) Treated soil with 500x amplification  
(c) Untreated soil with 2x amplification  
(d) Treated soil with 2x amplification.

**Figure 5.** Formation gel by SEM images of untreated soil and natural soil with the addition of 12% MK cured for 7 days at different amplifications.

5.5 X-Ray diffraction (XRD)
The mineralogical percent of the natural and treated soil are shown in Table 2. Figure 6 clarified the XRD pattern for natural soil and treated soil with the addition of 12% MK at 20° C cured by 7 days. The
results of natural and treated soils indicate that the peaks intensity obtained with Calcite and Quartz. The reduction of difference in the XRD pattern of the treated soil represented that no new clay minerals were made with the added MK to the soil. As well, Geopolymer gels can improve mechanical characteristics.

| Natural soil | Treated soil |
|--------------|--------------|
| Mineral material | Percent (%) | Mineral material | Percent (%) |
| Calcite | 29.8 | Calcite | 18.4 |
| Quartz | 29.4 | Quartz | 12.7 |
| Kaolinite | 18.4 | Kaolinite | 9.5 |
| Muscovite | 7.5 | Muscovite | 5.2 |
| Vermiculite | 6.4 | Vermiculite | 4.7 |
| Montmorillonite | 6.1 | Montmorillonite | 5.8 |
| Illite | 2.3 | Illite | 7.7 |
| Anataes | 4.1 | Sodium silicate | 22.2 |
| - | - | Sodium tecto-alumosilicate hydrate | 19.1 |

**Table 2.** Clay mineral percent for natural and treated soil samples.

**Figure 6.** XRD pattern of a) natural soil, b) treated soil.
6. Conclusion

- Curing temperature and duration is essential in the activation of MK-based geopolymer to produce higher compressive strength.
- The addition of 10% and 12% MK led to a moderate increase in the UCS of most soil samples for both temperatures.
- A high percent of MK to treated soil may be ineffective in some conditions due to the exhaustion of different components in the reaction environment.
- Higher heats have a noticeable effect on soil behavior.
- SEM test shows that the microstructure of the untreated soil contains many cracks and pores due to weak soil bonds, while the treated soil shows a decrease in cracks and void pore, which contributes to the enhancement of its strength.
- XRD pattern for treated soil shows the intensity of the peaks achieved with calcite and quartz.

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