

EFFECT OF CURING TEMPERATURE ON THE DEVELOPMENT OF HARD STRUCTURE OF ALKALI-ACTIVATED SOIL

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ABSTRACT: This study is directed to the feasibility of usage of palm oil fuel ash (POFA) as a precursor of alkaline activation reactions which made by potassium hydroxide in order to stabilize the soil and improve its expediency for the different case of construction. The effect of duration and temperature of curing, for the alkaline activation process, on the Unconfined Compression Strength (UCS) of the soil-POFA mixture is important in enhancing the treated soil properties. Use of 30, 50, and 100°C of heating affected the UCS of the soil mixture. The highest strengths were obtained at 100°C for the alkali activation process. Microstructural analysis using The Brunauer–Emmett–Teller (BET) and Infrared spectrometric analysis (FTIR) shows the benefits of alkaline activation in terms of decreasing the duration of the alkaline process. The pore distribution analysis showed a trend to rise pore size volume with increasing temperature, which affects the mechanical characteristics. This was due to the fastest crystallization processes which account for strength gain after a short period of heating. It has been also presented the opportunity of observing the alkaline activation process by FTIR Spectra. This paper highlights a more environmentally friendly procedure of stabilizing soils compared with the traditional binders such as cement and lime.

Keywords: Soil stabilization, Ground improvement, Alkaline activation, Geopolymerisation, Unconfined compression strength

1. INTRODUCTION

The alkaline activation (in other words, 'geopolymerisation') is described as a polycondensation, this reaction integrates minerals chemically, consisting of aluminum (Al) and silica (SiO₂) alternately tetrahedrally interlinked by sharing all the O₂ (oxygen atoms). This process begins by breaking the covalent bonds Al–O–Si, Si–O–Si, and Al–O–Al from the vitreous phase of the source material due to the high concentration of the hydroxyl in the alkaline medium, which results in transforming the alumina (Al₂O₃) and silica (SiO₂) ions in colloids and releasing them into the solution. At this state, aluminosilicates (Al₂SiO₅) become extremely reactive materials to form a well-structured aluminosilicate polymerized framework [3, 4, 13, 18].

Lately, the mechanism of geopolymerisation (alkaline activation) using a wide variety of aluminosilicate (Al₂SiO₅) source material was further investigated by a growing and large body of literature. And even more, they studied alkaline activators role, which is made by mixing a highly-alkaline solution (as sodium hydroxide (NaOH)) with a silica-rich source (such as sodium silicate), and the environmental benefits of the alkaline-activation method [8, 13, 15, 16, 17].

The majority of these studies shows that the

proposition; alkali-activated binders have superior performance compared to cementitious binders performance. It consumes lower energy portion; 80% lower than carbon dioxide (CO₂) emissions, has higher strength and obeys the principles of sustainable development. Hence, due to all the agreements between governments all around the world about taxing carbon dioxide (CO₂) emissions, the promotion of sustainable and environmentally-friendly materials like alkali-activated binders are encouraged, which makes it thought of as the future material [13, 14].

According to this knowledge and advantages, researchers in the synthesis of alkali-activated binder field and identification of reaction mechanisms in civil engineering frameworks have intensified their work to reach their goal. However, it is still early to use this technology in ground improvement applications [5, 7, 9, 10, 12, 13].

Some geotechnical scientists have investigated the efficacy of geopolymeric (alkali-activated) high calcium and low calcium fly ash as alumina and silica nebulous source for soil stabilization [5, 6, 7]. Also, a current report by Zhang [19] examined the practicality of utilizing metakaolin as an antacid initiated soil stabilizer at shallow profundity. Such investigations were directed by mixing the alkali-activated binders with weak soils in existence of

NaOH (sodium hydroxide) and sodium silicate (silica-rich source) as a based alkaline activator, and their outcomes prescribe the use of the alkali-activated binder as an effective technique of soil stabilization. Likewise, the creators presumed that the geopolymerisation (alkaline activation) technique is a basic procedure and it doesn't include any entanglements or high-cost methods, or even any particular systems.

2. EXPERIMENTAL METHODS

Depending on the Unified Soil Classification System [1], the chemical composition and physical properties of two type of soils utilized in this research are listed in Tables 1 and 2, respectively.

Table 1 Physical characteristics of soils

Properties	Standard	Soil 1	Soil 2
Liquid limit (LL) (%)	BS 1377-2	48.7	74
Plastic limit (PL) (%)	BS 1377-2	34.5	43
Plasticity index (PI) (%)	BS 1377-2	14.2	31
Specific gravity (GS)	BS 1377-2	2.52	2.34
MDD (Mg/m ³)	BS 1377-4	1.58	1.32
OMC (%)	BS 1377-4	24	30
Sand (%)	USCS	32	-
Silt (%)	USCS	24	78
Clay (%)	USCS	44	22
UCS (kPa) - after compaction test	BS 1377-7	380-390	180-190

The POFA, utilized as a forerunner for the alkaline activation reactions, was gathered from a plant in Johor, in the southern area of Malaysia. As can be seen from the chemical analyses (Table 3), this outstanding farming deposit has a high measure of silica and alumina. In any case, keeping in mind the end goal to accomplish a suitable chemical composition, and in addition a favorable shape and size, the antecedent was primarily exposed to a pretreatment, which involved calcination and grinding, so as to expand the reactivity level of the Al and Si and change the auxiliary coordination between the Al and the oxygen ions.

Table 2 Chemical analysis of soils

Chemical properties	Soil 1	Soil 2
Silica (SiO ₂)	41.26	60.6
Alumina (Al ₂ O ₃)	36.96	28.31
Iron oxide (Fe ₂ O ₃)	10.07	5.4
Calcium oxide (CaO)	-	-
Potash (K ₂ O)	11.71	3.52
Magnesia (MgO)	-	0.6
pH	5.3	3.8

Table 3 Chemical composition of POFA (before and after pre-treatment)

Constituent	Natural POFA	Treated POFA
Physical properties		
Specific gravity (GS)	2.42	2.51
Chemical properties		
Silica (SiO ₂)	46.04%	55.78%
Alumina (Al ₂ O ₃)	19.39%	17.29%
Iron oxide (Fe ₂ O ₃)	6.10%	4.17%
Calcium oxide (CaO)	13.87%	11.93%
Potash (K ₂ O)	8.61%	7.79%
Magnesia (MgO)	-	-
Loss on ignition (LOI)	9.68%	1.5%

Afterward drying the POFA in a stove at 105°C, for 24 hours, the resulting particles were crushed in an 80-cm diameter ball-mill for 24 h and then passed over a 300-µm filter. Different ball sizes were used, in the range of 12–30 mm. The crushing speed of 60 rpm resembled nearly 65% of the critical speed, which, in turn, was determined by dividing a constant by the square root of the mill diameter. In the subsequent stage, and keeping in mind the end goal to evacuate any unburned carbon, the ground POFA was warmed in an electric stove at 440°C for around 1 has demonstrated in Figure 3.4. After the pretreatment methodology, X-ray fluorescence (XRF) spectrometry was utilized for essential element investigation. A similar pretreatment has been

adopted by different researchers [11, 13], who revealed the technique to be viable in expanding the particular surface and reactivity of the ash.

Potassium hydroxide (KOH), having alkaline cation, was chosen as an activator on account of its outstanding productivity. In spite of the fact that the cost of NaOH is not as much as KOH, the researchers utilized KOH as a soluble base activator in light of the fact that K^+ has a bigger size than the other alkaline metal cation, and in this manner permits more thick and private polycondensation responses, which generously rise the general long-term strength of the alkali-activated soil. As per those analysts' discoveries, networks containing K^+ displayed higher UCS and particular surface region and a lower level of crystallinity in long-term of curing systems. This reagent was provided in pellet form by the organization R&M chemical and was beforehand weakened in refined water to accomplish a predesigned concentration. Note that the response with water of KOH is intensely exothermic, as with every solid base. In this way, the alkaline solution was left to cool to encompassing temperature before utilize.

In this investigation, the UCS tests were directed as per Part 7: Clause 7 of the BS 1377-1990 [2] to assess the effectiveness of source binder and alkaline activators and furthermore to discover the impact of kaolinite content of the original soil that affects the strength behavior of treated soil examples. The UCS tests characterized the corresponding to the peak stress condition. Stress value was estimated at indicated ages in three examples for every mixture and all information focuses veered off under 5% from the normal. For the unconfined compressive strength tests, with a predesigned activator concentration (10M and 12.5M potassium hydroxide) to dry solid amount, a total of two mixtures were prepared by using KOH, with POFA (15 and 20% by dry mass of soil).

To set up the specimens, in the initial step, the alkaline activator was liquefied in the solution at a predetermined absorption. In this progression, refined water was utilized to break down activator pellets to keep away from the impact of obscure contaminants in the mixing water. Since this response was exceedingly exothermic and to avoid expanding the plasticity of the parent soil, the soluble solutes were cooled and arranged for 24 hours before being utilized as a part of the mixture. In the next step, the required measure of characteristic soils was air dried for 24 hours. In the subsequent stage, the necessary dose of air-dried soil was mixed by including the cooled alkaline solution with the predefined dosages of POFA. Soon after mixing, samples were placed in a cylindrical mold (50 mm dia. by 100 mm high), applying steady direct compaction in three equivalent layers, and expelled. Since potassium hydroxide is a strong base, compacted specimens within the

existence of such activator were not sufficiently solid to be remolded. Subsequently, the chamber tests were expelled with the farthest caution. Directly after extrusion, all specimens were put in the oven (Figure 5.1) under different temperature and different time then closely covered in polythene covers to prevent loss of water and cured under different curing regimes (7 and 28 days) prior to testing in a laboratory ambient temperature. After shearing, all specimens were retained for mineralogical analysis.

In order to better understand the effect of temperature on the treated soil mixture, selected specimens were analyzed (after submitted to the respective UCS test) using The Brunauer–Emmett–Teller (BET) method and Infrared spectrometric analysis (FTIR).

3. RESULTS AND DISCUSSION

3.1 Mechanical properties

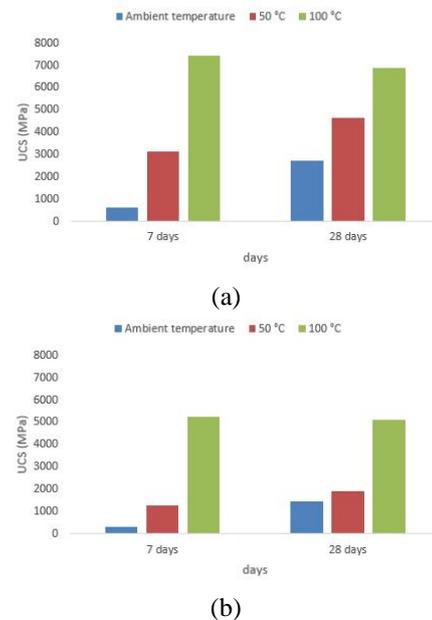


Fig. 1 Development of compressive strength of alkali-activated soils cured at ambient temperature, 50, and 100 °C over time (7 and 28 days). The temperature during curing was carried out for the initial 3 h at 50 and 100 °C. (a) Soil 1 (b) Soil 2

Fig. 1 shows the effect of curing temperature on the compressive strengths of 2 alkali-activated soils at the age of 7 and 28 days with 3 hours of heating duration. The reference alkali-activated soils cured at a laboratory ambient temperature achieved the UCS values of 2.75 and 1.48 MPa for Soil 1 and Soil 2, respectively, at the long-term (28 days). The short-term strengths of geopolymeric (alkali-activated) soils cured at an ambient temperature achieved 0.61 and 0.33 of UCS due to the delayed setting of

specimens. As estimated, the high temperature quickens the development of a tough structure especially in the early age of alkaline activation reaction. The UCS values of both alkali-activated soils cured at 50 or 100°C, respectively, achieved the higher values 7.42 MPa for soil 1 and 5.22 MPa for soil 2 after 7 days and three times surpassed the values noticed for specimens cured at a laboratory ambient temperature. Nevertheless, the quick setting avoids the mixture from the creation of more dense and hard structure; therefore the UCS values in 28 days for soil 1 and soil 2 with 100°C is by 0.54 and 0.12 MPa lower compared to the alkali-activated soils in 7 days, respectively. On the contrary, though the alkali-activated mixtures of soil 1 and soil 2 that were cured at a laboratory ambient temperature exhibit late strengths' improvement, they achieved the values of 2.75 and 1.48 MPa at the age of 28 days. The justification of this performance is similar to the effect of temperature on the strength improvement of metakaolin-based geopolymers [20,21]. At an early age, the strengths increase with the temperature since at elevated temperature the alkaline activation degree is higher, and therefore the quantity of reaction produces rises. On the other hand, a longer time, when the strengths is almost similar with those observed after early age for specimens that were treated at 100°C temperature because the loss of water in the system through curing and the resultant shortage of moisture to completely dissolve the glassy component of POFA (first stage of cementitious gel formation) under these conditions [22].

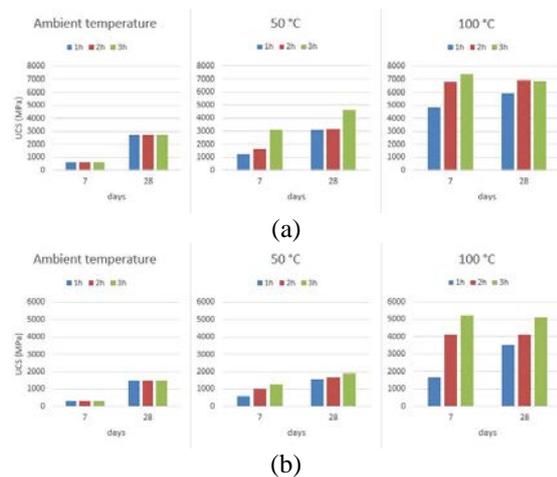


Fig. 2 Influence of curing time on compressive strength of treated soils cured at elevated temperatures (ambient temperature, 50 and 100°C) (a) Soil 1 (b) Soil 2

The study was also concentrated on the effect of curing time at high temperatures. The UCS of treated soils as a function of curing time at identified high temperature is shown in Figure 2.

Longer curing of alkali-activated soil mixtures quickens the improvement of strength in the early stage of hardening. In the meantime 7 days compressive strength of alkali-activated soil 1 and soil 2 cured for 1 hour at 50°C was only 1.21 and 0.57 MPa, the strength augmented nearly three times to 3.14 and 1.25 MPa, respectively, when such treating was extended to 3 hours. The highest results of strengths were achieved in 28 days for specimens treated at an ambient temperature and 50°C. The effect of curing time on the strength improvement of alkali-activated soils cured at 100°C is very similar. The samples that were treated for two or more hours at 100°C achieved their highest strengths at an early age (7 days) with UCS values about 7.42 and 5.22 MPa for soil 1 and soil 2 respectively. On the other hand, when the specimen was exposed to 100°C just for 1 hour, a tendency of the strength improvement is very similar to that detected with 3 hours curing at 50°C. In this case, the short-term UCS values are around 4.85 and 1.63 MPa for soil 1 and soil 2, respectively, but moreover, the long term age (28 days) strength is not reduced.

3.2 Pore structure analysis

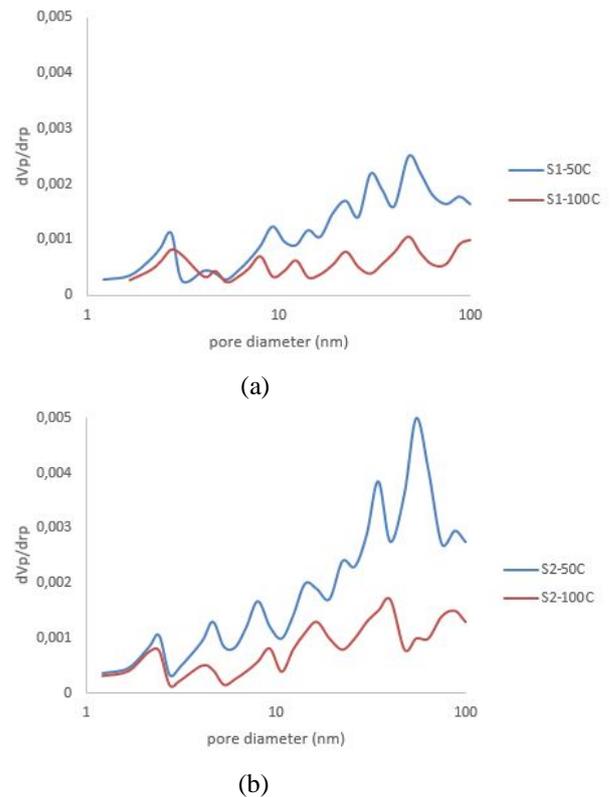


Fig. 3 Comparison of pore distribution of cured at 50 and 100°C at 28 days (curing at 50 and 100°C was carried out for initial 3 h) (a) Soil 1 (b) Soil 2

The changes in the porosity of specimens were also evaluated by using a BET test at 28 days as presented in Figure 3. The higher porosity of samples can quicken the cracking of specimens due to exposure to UCS. Figure 3 (a) reveals the differences in BET pattern between the treated soil 1 with elevated temperature 50 and 100°C after 28 days. As can be seen, the higher porosity was recognized in the treated soil 1 cured in 50°C due to the high volume peaks about 0.0021 and 0.0025 with pores diameter around 29.89 and 47.21 nanometer while the peaks of pores volume in the treated soil 1 cured at 100°C were 0.0007 and 0.0009 with the diameters of 54.42 and 86.83 nanometer, respectively. The effects of curing temperature after 28 days on the treated soil 2 samples cured at 50 and 100 °C are illustrated in Figure 3 (b). The same trend was observed for treated soil 1 samples when cured at elevated temperature. It can be observed that treated soil 2 cured at 50°C had higher porosity of 0.0038 and 0.0049 with a diameter about 34.36 and 54.42 nanometers than the recorded results for samples cured at 100°C, the porosity of 0.0017 and 0.0015, with diameters about 39.78 and 86.83. As can be seen, the volume and the diameter of the pores decreased with elevation in curing temperature.

3.3 Infrared spectrometric analysis

FTIR spectra of treated soils were examined mostly for the asymmetric stretching vibration of Si–O–T linkages that were expected to be signified by the point of maximum absorbance in the main band (section 900–1300 cm^{-1}). Absorption features in the examined area of the spectra are asymmetric and broadened and involve the similarity of more bands. Deconvolution of the main band of hardened treated soils cured at the elevated temperature shown the similarity of numerous important bands (Figure 4). The bands related to the asymmetric stretching mode at 914 and 1006 cm^{-1} of natural soil 1 with POFA were found in all spectra. Also, the band at 1037 cm^{-1} are related with the asymmetric stretching mode of natural soil 2 with POFA and they were observed in all spectra. Noticing this feature even after 28 days implies that an amount of unreacted soil 1 with POFA and soil 2 with POFA still remains in hardened material [23]. The most intensive bands at 1029 and 1113 cm^{-1} characterizes oxygen connections of asymmetric vibration between tetrahedra in geopolymeric structure of treated soil 1 and soil 2, respectively. Weak bands at 1408 and 1410 cm^{-1} is linked with stretching vibrations of band Si–O groups in partially hydrated treated soil 1 and soil 2 [24]. Bands at 752 and 950 cm^{-1} that comes into view in the spectrum of original soil 1 and soil 2 with POFA, respectively, and diminishes through alkaline activation is related with symmetric Al–O–Si stretching vibrations.

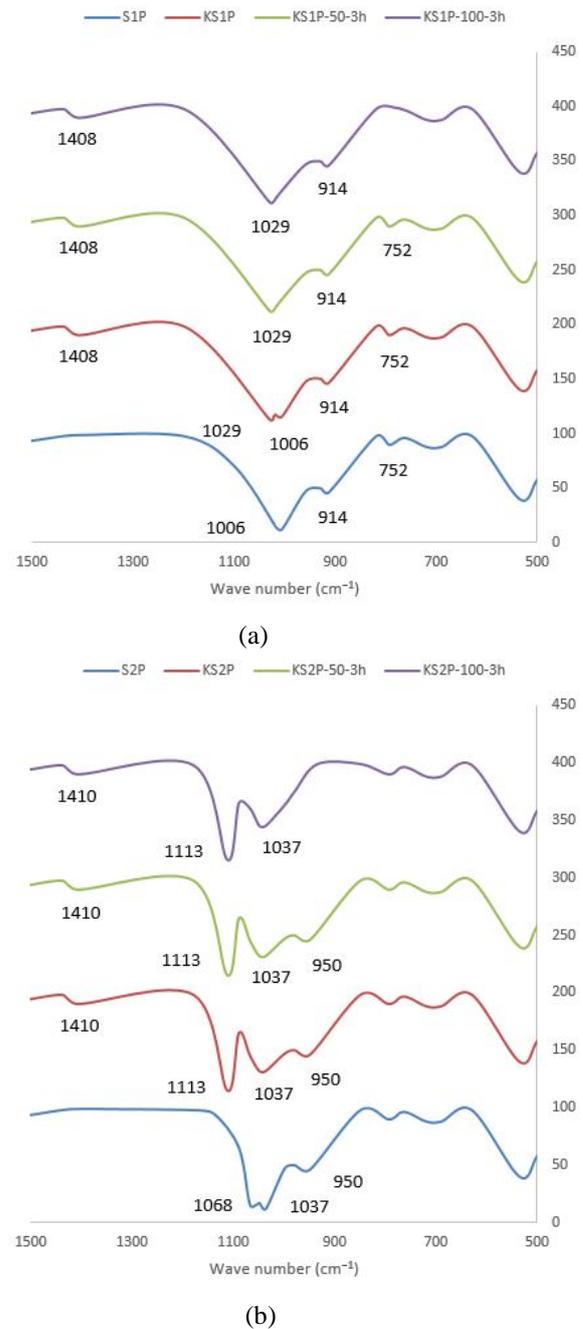


Fig. 4 Deconvolution of the main band related with Si–O–T stretching mode in treated soils cured at elevated temperatures (ambient temperature, 50 and 100°C) (a) Soil 1 (b) Soil 2

Though MAS NMR methods deliver extra comprehensive sight of alkaline activation procedure [20], FTIR spectroscopy is, nevertheless, a potential and unpretentious technique for the checking of alkaline activation procedure and creation of the tough structure.

Examination of FTIR spectra in the area of Si–O–T stretching vibrations shown remains of unreacted soils in hardened treated samples regardless of curing time. It supports a proposition that original materials (soil with POFA) are not completely dissolved through alkaline activation procedure but the reaction occurs in the external layer of the solid elements [20]. The reaction degree is then measured mostly by diffusion of hydroxide and silicate ions during primary gel which is influenced mostly by curing temperature at the short-term of the reaction.

4. CONCLUSIONS

In this experiment, the influence of temperature during curing and curing time at high temperatures on the mechanical characteristics of two treated soils was examined by determining compressive strengths' improvement over time. The achieved values were clarified on the base of pore structure investigation executed by utilizing mercury intrusion porosimetry apparatus and the changes of microstructure on the molecular level were observed by FTIR spectroscopy. The investigational and analysis led to subsequent conclusions.

1. The temperature during curing has an important influence on setting and hardening of treated soils. At high and an ambient temperature, the matter set practically within 3 hours at the latest. Evaluated mechanical tests showed that 7 and 28 days mechanical characteristics of treated soils are significantly reliant on curing temperature. Elevated temperatures rise the short-term compressive strength, which can even achieve higher values in 7 days. Nevertheless, the long-term (28 days) strengths were obviously fairly lower or similar in comparison with those saw for short-term (7 days) samples that were cured for 2 or 3 hours at 100°C temperature due to the rapid development of the tough structure perhaps does not consequence in such a good quality get product. Furthermore, the time of curing affects the effect of temperature during curing. Curing for just 1 hour at a high temperature did not cause significant changes in strengths improvement but longer curing was accountable for a significant quickening of the reaction degree and rise in short-term strengths.
2. Porosimetry analysis presented that the pore size distribution reduced with high temperature, and this was true for both soils. On the other hand, the main differences in pore structure came into view between specimens treated at diverse temperatures (50 and 100°C). The elevated temperature of hardening progression led to a decrease larger pores which has a positive consequence on the ending mechanical characteristics of alkali-activated treated soil.

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