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Effects of Temperature in Different Initial Duration Time for Soft Clay Stabilized by Fly Ash Based Geopolymer

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Abstract

When soft clay soils are included in engineering projects, it's stabilized usually with some kinds of admixtures named as stabilizers. The common stabilizers that highly practiced are OPC, lime, high calcium fly ash (FA), etc. Each one of these stabilizers has its shortcomings. Geopolymers are the product of alkali activated aluminosilicate sources that excelled as an alternative to ordinary binders due to its sustainability, low cost and good mechanical properties. This study investigates the effects of some key elements like liquid over fly ash ratio (Liq/FA), initial duration curing time (D) and its temperature to soil – FA based Geopolymers samples characterized by its unconfined compressive strength testing (UCS), volumetric measurements, scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-Ray diffraction (XRD). The Liq/FA taken as 2.71, 3.167, 3.8 and 4.75 respectively and the duration time taken were 1, 6, 18 and 24 hrs. respectively. The tests results showed that the maximum peak strength gain when Liq/FA is 3.8 at 90 °C with 24 hrs. D. It was observed that Young's Modulus increased with increasing curing temperature for certain D. Volumetric strain increased by increasing D and its temperature. SEM and XRD analyses confirmed the Geopolymers gels formation for a selective precursor while EDX analyses showed that silicon over aluminium ratio is 1.38 for selective spectrum within the gel to the same mixture.

Keywords: Geopolymers; Soil Stabilization; Scanning Electron Microscopy (SEM); Energy Dispersive Spectroscopy (EDS); X-Ray Diffraction (XRD).

1. Introduction

Soils can be categorized according to its particles size usually into cohesive and cohesion less. Cohesive soils have a small particle sizes which causes general tendency to illustrate sticky properties and / or particle – water attraction, furthermore, physical disturbances, wetting exposure may dictate such soils to possess low shear strength, high plasticity and high compressibility [1]. Soft clay is a term refers to soils that exhibit low undrained shear strength (less than 40 kPa) and high compressibility (Cc between 0.19 to 0.44) at specified moisture contents (45 to 65%) [2, 3].

When soft clay soils are encountered in any engineering projects, some kind of ground improvement is essentially needed to overcome its defects. Techniques like pre loading, electro osmoses, stone column were highly examined in the literature. The chemical stabilization represents a suitable method to treat soils under consideration. This method can be done by addition of chemical admixtures (stabilizers) to soils to render some geo technical properties like strength, volume and moisture change) less sensitive to fluctuations [4].

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Ordinary Portland cement (OPC), lime, high calcium fly ash and bitumen are the most stabilizers used within this field. As a matter of fact, ordinary Portland cement is the second material used around the world [17] and the production of that material increases 3% every year [18], consequently, green house emission of this material is about 1.35 billion annually [19], so the control of such greenhouse gas emission is imperative and major issue [20]. Because all of these, several efforts are now in the progress to reduce the use of Portland cement in civil engineering applications. In the other hand, lime and other calcium based aluminosilicate materials like class CFA have a long term performance deterioration due to the possibility of ettringite and thaumasite formation.

It is believed that many of flexible and rigid pavement failure cases that was recently reported because of the underlying subgrade failure, using soil stabilization techniques is very cost effective since the required thickness of each treated layer can be decreased to a great extent, another profits of such method can be gained resulted by eliminating the required maintenance and the deterioration rate of the top surface layers, due to above, authors and research organizations are highly motivated to seek for new motivate materials that can play this turn of improvement. Geopolymers are the product of alkali activation of some amorphous aluminosilicate sources such as low calcium FA, rice husk ash and Meta kaolin. Common binding gel results from hydration of cement is calcium silicate hydrate (C-S-H) and aluminium silicate hydrate (A-S-H) or both, while in Geopolymers, the main product usually are sodium aluminosilicate hydrate (N-A-S-H) [9], and/or potassium aluminosilicate hydrate (K-A-S-H) [10]. Many information are now available about using Geopolymers concrete and mortar in civil engineering projects [6], furthermore, researchers widely used fly ash as a source material in this broad field [21]. Actually, there are a great agreement that degree of heating plays an important role in accelerating strength [22], that fact was robustly confirmed by series of research contributions through the literature [23]. In general sense, heating changes the formation of the crystals of the resulted Geopolymers gel which lead to many mechanical properties of Geopolymers to be enhanced [24], the most interesting enhanced mechanical properties comprise compressive strength [25] and even tensile strength [26],[27]. It was clearly noticed that soil – Geopolymers applications are a current issue [28] and this dictates another efforts to improve the knowledge about this effect of heating temperature to paralyze the good experience that was acquired in Geopolymers mortar and concrete and that what is aimed throughout this paper. However, it was reported that the feasibility of using Meta kaolin based Geopolymers as lean clay stabilizer was examined and confirmed [7], and FA based was also used to treat granular soil [8]. This study tries to improve the knowledge the effect of heating temperature for soil- *FA* based Geopolymers mix taking into account some Geopolymers key elements such as *Liq/FA*, curing temperature and *D*. The present study tries to investigate that effect by an experimental program.

1.2. Nomenclatures

A-S-H	Aluminium silicate hydrate.	Liq/FA	Liquid over fly ash ratio.
CO_2	Carbon dioxide.	N-A-S-H	Sodium aluminosilicate hydrate.
C-S-H	Calcium silicate hydrate.	OPC	Ordinary Portland cement
D	Initial duration time in hrs.	SEM	Scanning electron microscopy.
EDS	Energy dispersive spectroscopy.	UCS	Unconfined compression strength in MPa.
FA	Fly ash.	XRD	X-Ray diffraction.
K-A-S-H	Potassium aluminosilicate hydrate.	Liq/FA	Liquid over fly ash ratio.

2. Methodology

2.1. Materials Used

2.1.1. Soil

Table 1 lists some geotechnical properties of soft clay soil recovered at Albawya suburb near Baqubah, Iraq. Figure 1 shows XRD analysis for that soil while Table 2 illustrates elements composition by energy dispersive spectroscopy “EDS”.

Table 1. Some geotechnical properties of soil used

Item	Property	Value	Specification
1	Specific gravity	2.71	ASTM D 854 - 2
2	Liquid limit	33.6	ASTM D 4318 -00
3	Plastic limit	21.6	ASTM D 4318 -00
4	Plasticity Index	12	/
5	Passing No.200	100%	/
6	Percent of sand	0%	ASTM D 422, D 1140
7	Percent of clay	59%	ASTM D 422
8	Percent of silt	41%	ASTM D 422
9	USCS classification	CL	ASTM D-2487
10	pH	8.7	ASTM D-2472

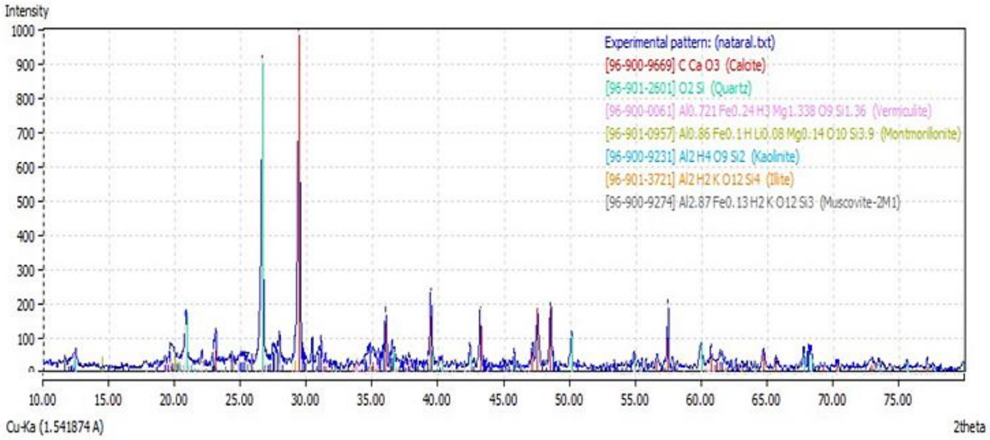


Figure 1. XRD analyses for soil used

Table 2. EDS analyses of soil used

Element	Weight%	Atomic%
O	55.44	72.36
Mg	2.65	2.27
Al	4.41	3.41
Si	15.36	11.42
K	1.46	0.78
Ca	14.12	7.36
Cr	0.11	0.05
Fe	5.91	2.21
Ni	0.35	0.12
Cu	0.01	0.00
Zn	0.02	0.01
Pb	0.16	0.02

2.1.2. Fly As

Low calcium FA “Class F” produced by Deyana Construction Projects Company used in the present study as aluminosilicate source. Table 3 lists elements composition by EDS.

Table 3. EDS analyses of soil used

Element	Weight (%)	Atomic (%)
O	51.41	67.46
Na	0.49	0.45
Mg	0.91	0.79
Al	12.71	9.89
Si	20.89	15.61
K	1.15	0.62
Ca	3.00	1.57
Ti	1.41	0.62
Fe	7.55	2.84
Co	0.15	0.05
Ni	0.12	0.04
Zn	0.14	0.05
Pb	0.05	0.01

2.1.3. Sodium Silicate Na_2SiO_3

Sodium silicate was manufactured in United Arab Emirates. Table 4 lists the properties of used sodium silicate.

Table 4. Properties of used sodium silicate

Item	Description	Value
1	Ratio of SiO_2 to Na_2O	2.4 ± 0.05
2	Na_2O percent by weight	13.10– 13.70
3	SiO_2 percent by weight	32.00 – 33.00
4	Density - 20° Baumé	51 ± 0.5
5	Specific Gravity	1.534 – 1.551
6	Viscosity (CPS) 20°C	600 – 1200
7	Appearance	Hazy

*According to the manufacturer.

2.1.4. Sodium Hydroxide NaOH

The sodium hydroxide used during the present study is commercially manufactured in Kuwait in flakes form. That flakes should be dissolved in water at specific weight to achieve the desired molar concentration. Table 5 lists some properties of used sodium hydroxide.

Table 5. Sodium hydroxide properties

Property	Unit Measuring	Specification ASTM E291-09	Results
Sodium hydroxide (NaOH), min.	Percent	97.5≥	98.14
Sodium carbonate (Na_2CO_3), max.	Percent	0.40	0.36
Sodium chloride (NaCl), max.	Percent	0.15	0.07
Iron oxides (Fe_2O_3), max.	Percent	0.01	0.005
Sulphate as Na_2SO_4	Ppm	200≤	70
Copper as Cu^{+2}	Ppm	4.0≤	0.1
Nickel as Ni^{+2}	Ppm	5.0≤	2.42
Manganese as Mn	Ppm	4.0≤	0.02
Silicate as SiO_2	Ppm	20≤	14
Water Insoluble	Ppm	200≤	60

*According to the manufacturer.

2.2. Solution Preparation

In order to synthesize good performance Geopolymers gels, adequate activator must be available. Alkali hydroxide or silicate used to initiate Geopolymerization, furthermore, alkali hydroxide solutions are the most common activators used due to its simplicity [6]. During the present study, the activator used are consist of sodium hydroxide and sodium silicate. Many authors studied the effect of the silicate to hydroxide ratio effects to Geopolymers concrete who established that ratio at 2 to get best strength gain. It was observed after many trails that this ratio dictated to be 0.5 due to sodium silicate viscosity. Another series of trails showed that the most effective total activator liquid to total solids (fly ash+ dried soil) ranged between 0.35 to 0.4. However, this ratio established to 0.38 in this study. A reasonable value of 10 Molar of sodium hydroxide was also used to prepare its solution.

2.3. Laboratory Tests and Sample Preparation

2.3.1. Unconfined compressive strength (UCS)

Nominal dimensions of the UCS molds used are 44 mm diameter and 100 mm height. The dried soil prepared and pulverized first, the activator was prepared at the specified recipe. The FA and soil then mixed before activator adding, the precursor mixed to about 3 minutes to achieve reasonable homogeneity. The resulted mixture poured in the molds and compacted at five layers by using adequate tamper and compaction efforts. Sample ejector used to extrude samples. Finally each sample was put in drying oven to conduct D (1hrs, 6hrs., 18hrs. and 24 hrs. respectively) with specified temperature (50, 60, 70, 80, 90, 100, 110 °C and 120 °C respectively) for each D, then after, the samples was taken to curing chamber to complete 7 day curing at 23 ± 3 °C. The samples tested at loading rate 2% per minute. The Young's Modulus of each sample was evaluated as the slope of the stress strain curve [13].

2.3.2. Microstructural Characterization

The micro structure of the stabilized soil was observed by SEM. Tescan VEGA 3 SB apparatus was used for untreated and for 3.8 Liq/FA cured at 70 °C for D = 6 hrs. The tests was conducted at University of Technology/ Nanotechnology and advanced Material Research Centre.

2.3.3. Mineralogical Analyses

The mineralogical analyses was done by XRD, the basic aim is to characterize the mineralogical changes and to confirm the resulting gels. The same broken samples of UCS tests crushed and grounded to do the test at University of Baghdad / Central laboratory of Ibn Alhaytham College for 3.8 Liq/FA cured at 70 °C for D = 6 hrs. Minerals matching was conducted using Match software.

2.3.4. Soil PH

Soil PH was measured according to ASTM D 4972 [15]. The basic aim is to monitor the available alkalinity testing since PH level influences Geopolymerization [16].

The crushed UCS samples pulverized to pass No 10. Sieve then added to the same weight of water. PH level observed after 10 minutes by pH meter.

2.4. Study Layout

Figure 2 shows the general layout of this study.

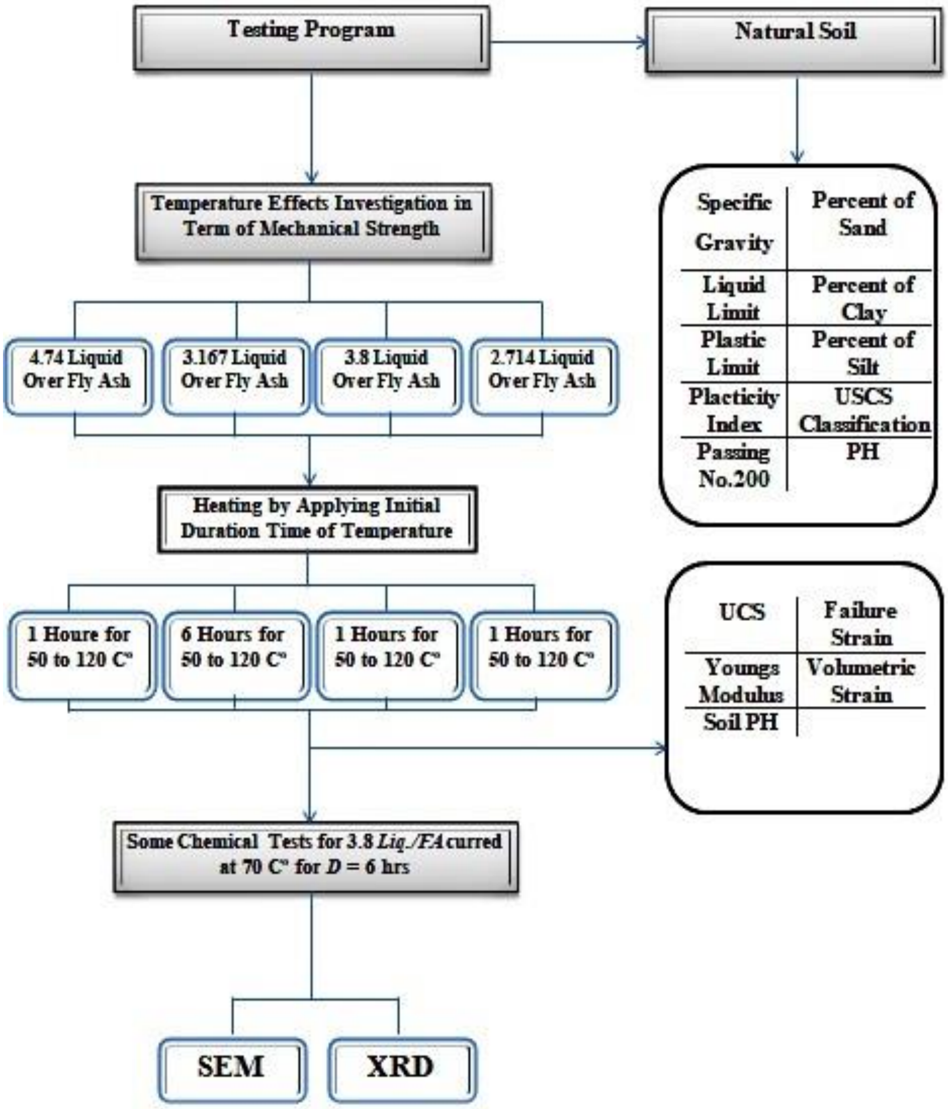


Figure 2. Study layout

3. Results and Discussion

3.1. Mechanical Properties of Stabilized Soil

Variation of peak UCS due Liq/FA for different temperatures for D are shown in Figure 3.

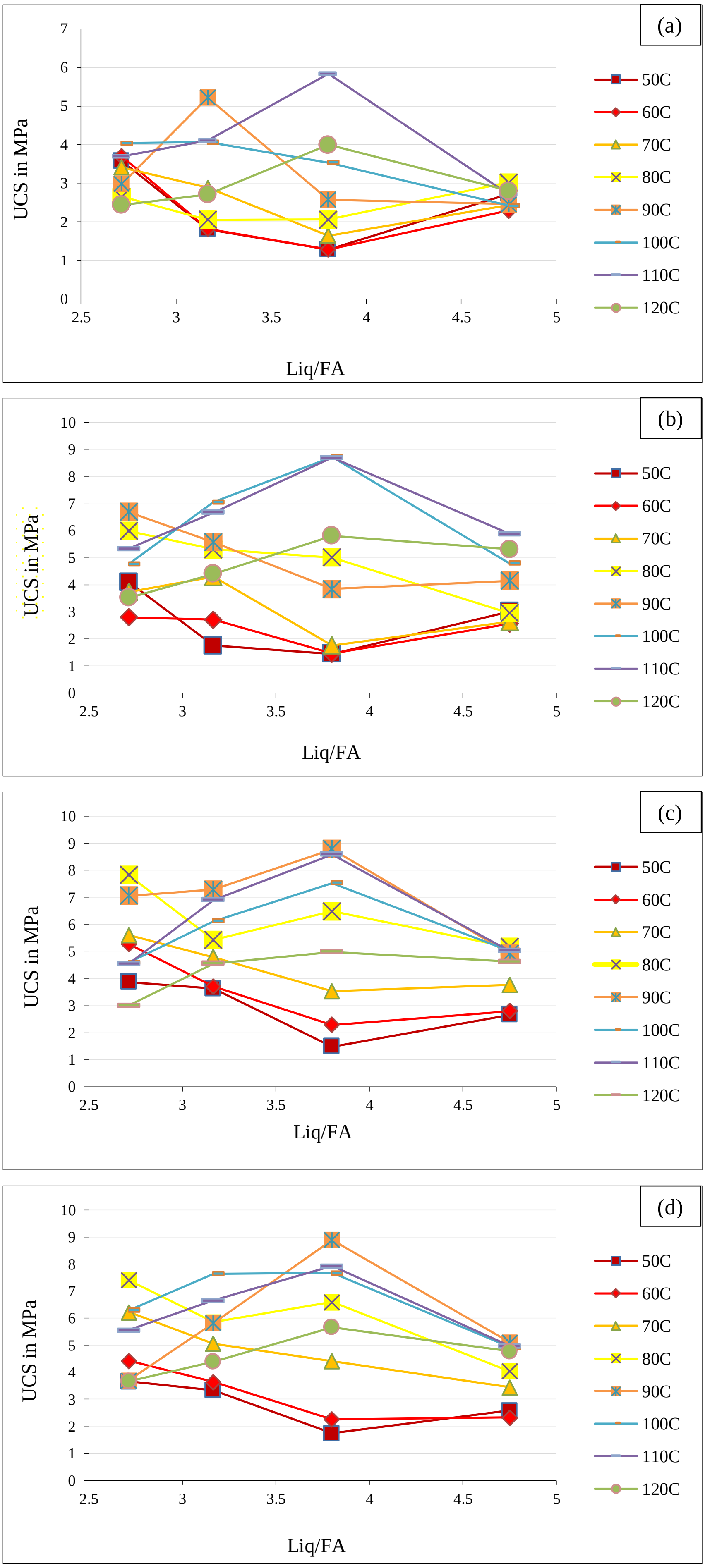
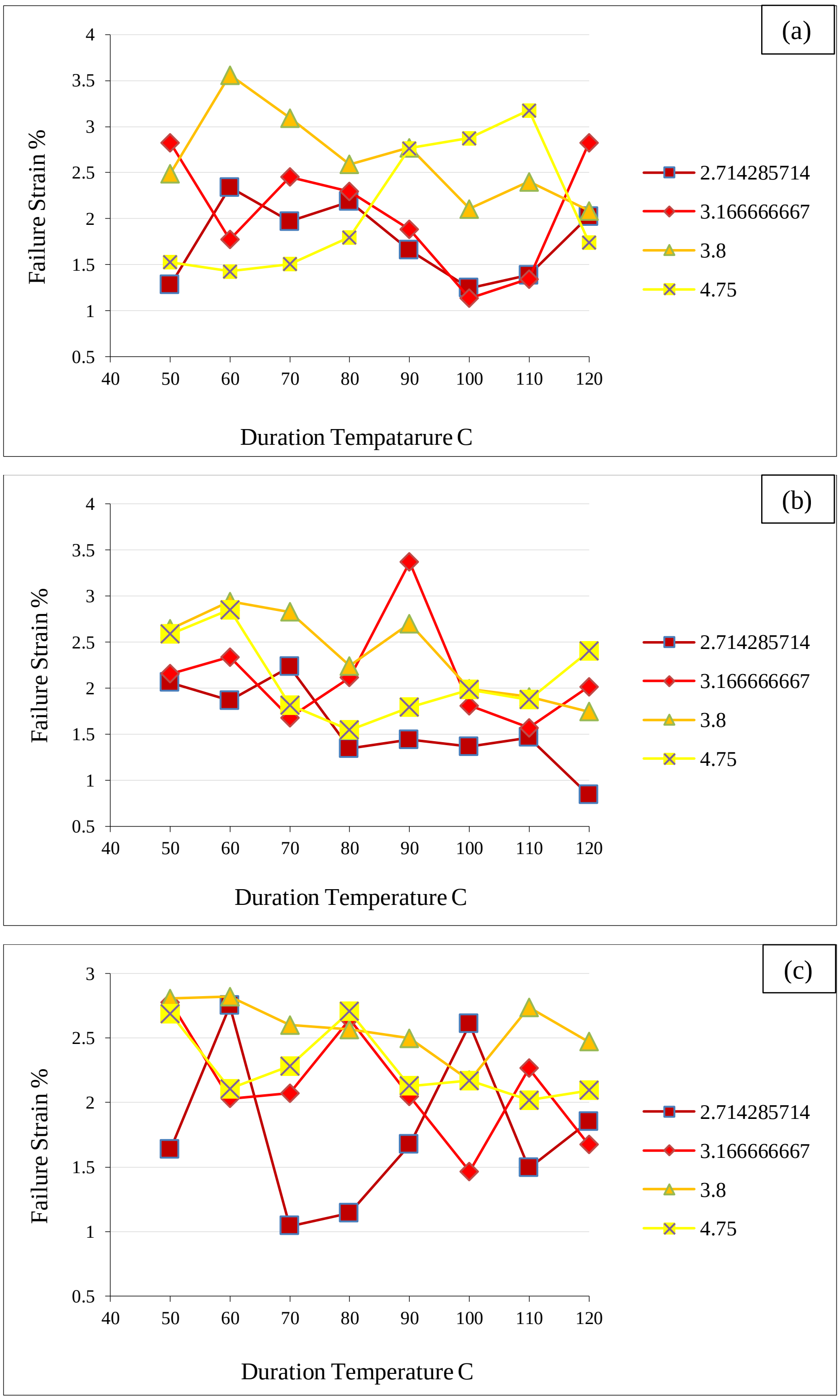


Figure 3. Variation of peak UCS due to different Liq/FA at 7 days for (a) D =1hr (b) D= 6hrs, (c) D=18hrs, (d) D= 24hrs

It is obvious that the UCS depends upon the degree of geo polymerization which depends in turn upon heat energy consumed. For that reason, when D, are 1hr and 6 hrs, the optimum Liq/FA appears obviously at 100 °C and 110 °C, while in 18hrs and 24hrs, Liq/FA appears in 80 and 90 °C. Increasing temperature for FA based Geopolymers enhanced UCS up to 70 °C at D = 48 hrs [14]. However, the optimum temperatures for soil Geopolymers mixtures are higher than in concrete because of the thermal properties of stabilized soil. Reduction of strength in high temperatures can be attributed to dehydration of free water above 100 °C [12]. Optimum Liq/FA is obvious at 3.8 when a considerable degree of geo polymerization was achieved.

It is clearly obvious that the UCS of the stabilized soil is much more than the corresponding untreated soil at its optimum moisture content value (0.433 MPa), the tested samples at all Liq/FA and heating conditions used confirmed the fact that fly ash based Geopolymers is effective soil stabilizer because increasing UCS of 345 kPa or more is considered effective according to ASTM D4609 [29]. However, the strength gain is uneven especially for the 4.75 Liq/FA for D=1 hr at low temperatures, this fact is expected in low dosages which analogous with meta kaolin experience [7], Figure 4 shows variation of failure strain due to the applied temperatures of D.



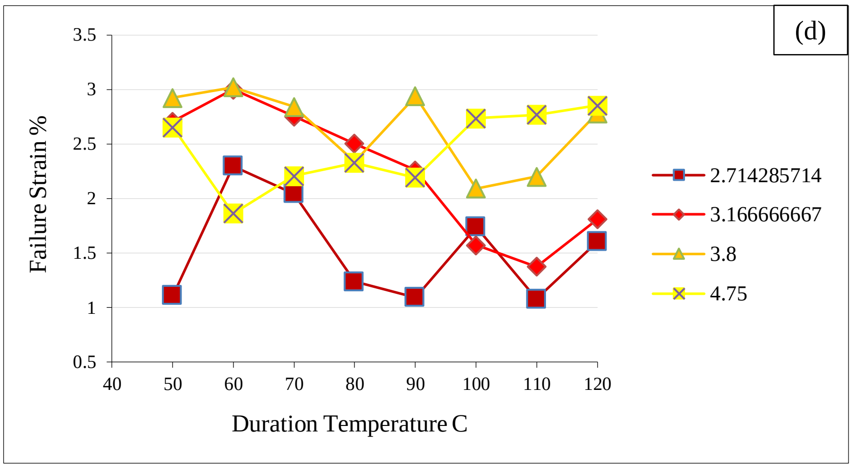
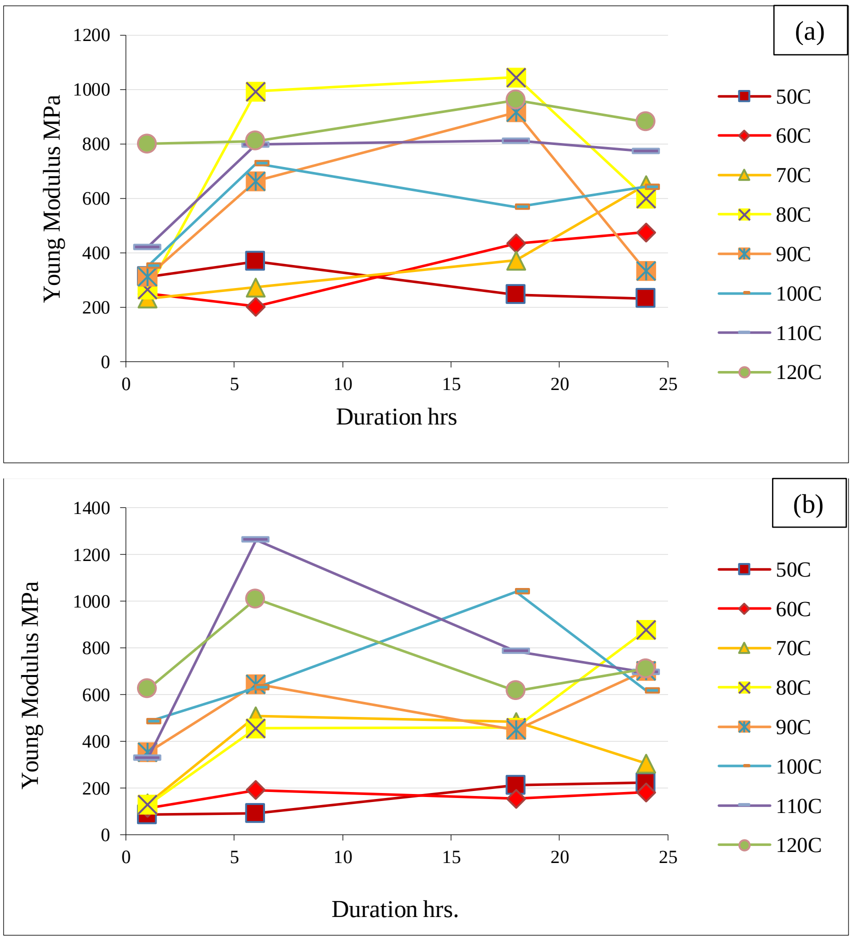


Figure 4.Variation of failure strain due to different temperatures at 7 days curing (a) D = 1hr (b) D =6hrs, (c) D =18hrs, (d) D = 24hrs

Enhancing ductility of soil by increasing failure strain is beneficial for flexible pavement, stabilizing clayey soils with Meta kaolin based Geopolymers at considerable concentration enhanced failure strain to about 75% [7]. The same degree of improvement was achieved at higher Liq/FA as shown in Figure 4. (b), (c) and (d). For Figure 4 (a) failure strain appears to be erratic due to low level of geo polymerization progress. Figure 5 shows the variation of Young's modulus due to different D for certain Liq/FA.



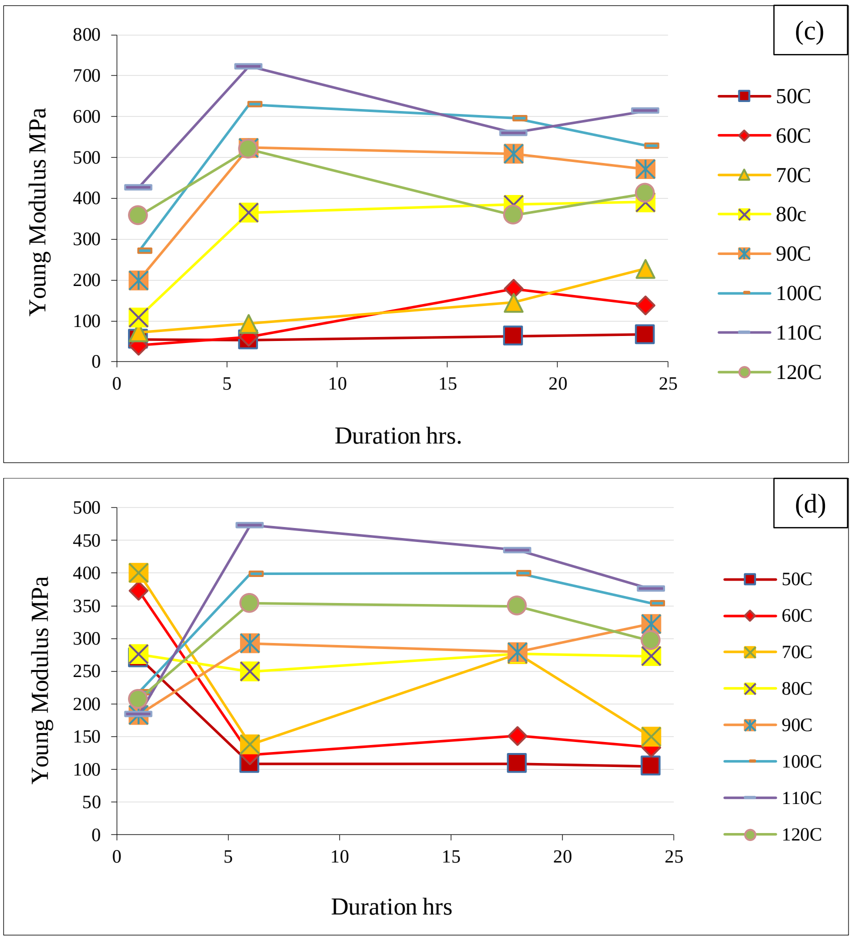
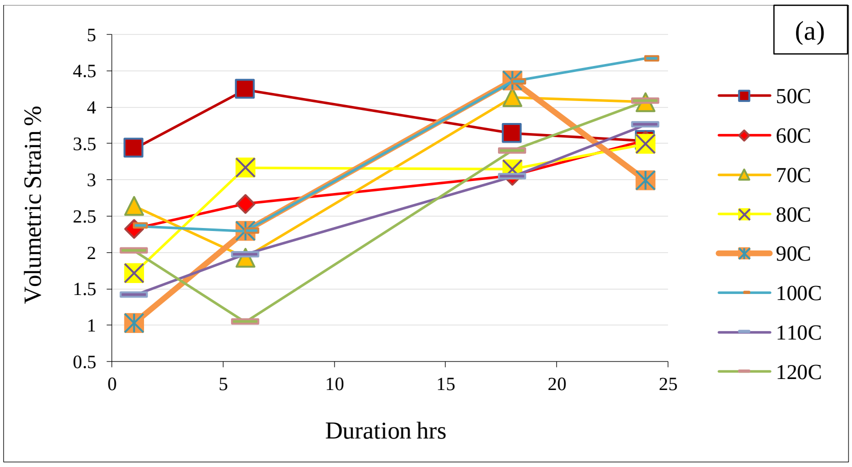


Figure 5. Variation of Young's modulus due to initial duration time at 7 days curing (a) 2.714 Liq/FA, (b) 3.167 Liq/FA, (c) 3.8 Liq/FA, (d) 4.75 Liq/FA

It can be seen from Figure 5 that Young's modulus increased as degree of geo polymerization progressed. The maximum value was appeared at 110 ºC. Enhancing gain value when compared with the reference untreated soil (120 MPa) is not as UCS, but this gain still takes lower values for the 4.75 Liq/FA. This behavior leads to thought that related mechanisms of failure are different between stiffness and strength for soil – Geopolymers mix which dictates other efforts in the future studies.

3.2. Volumetric Strain of Stabilized Soil

The volumetric strain is the change of volume to the original volume, the samples dimensions was measured just before UCS testing. Figure 6 shows variation of volumetric strain due to the applied temperatures of D.



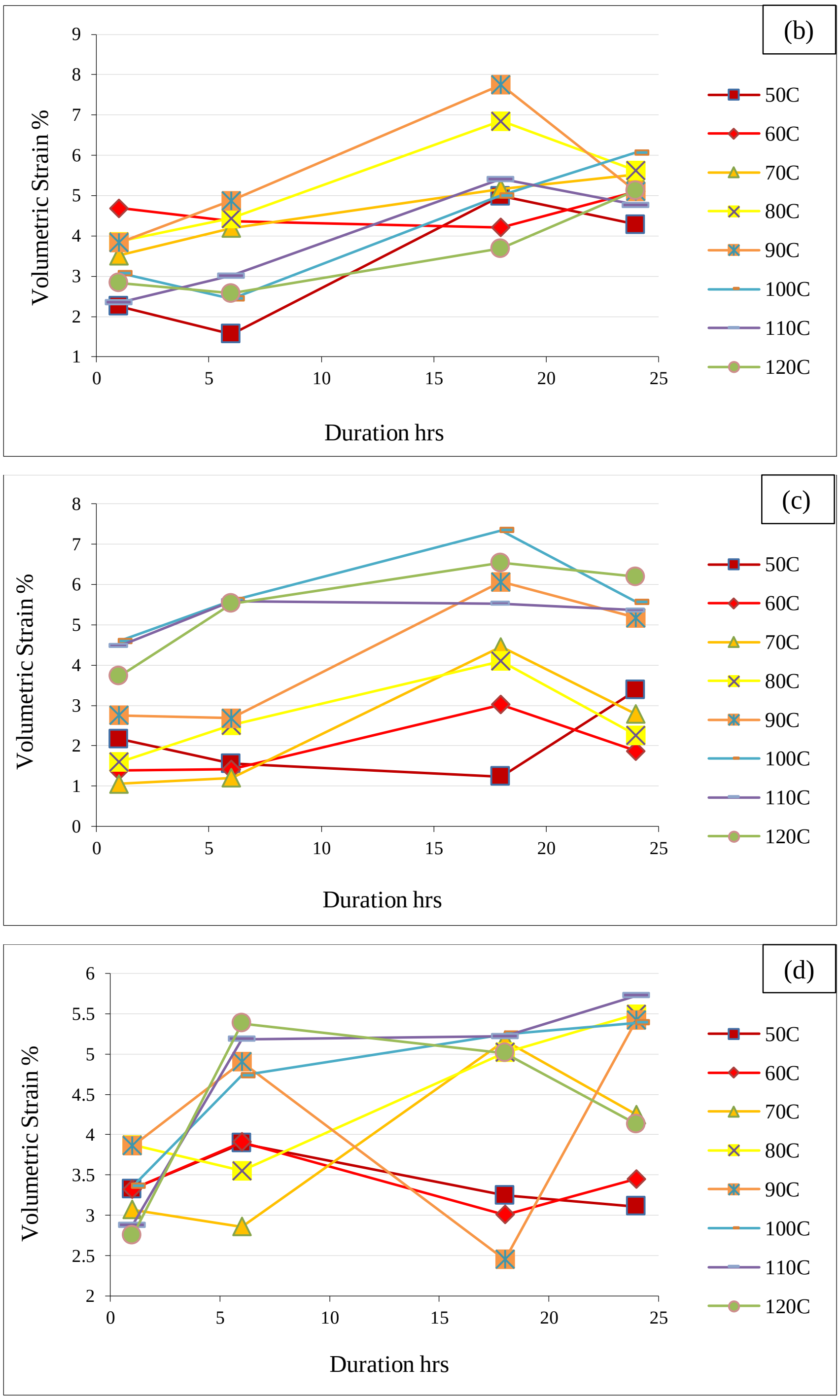


Figure 6. Variation of volumetric strain due to initial duration time at 7 days curing (a) 2.714 Liq/FA, (b) 3.167 Liq/FA, (c) 3.8 Liq/FA, (d) 4.75 Liq/FA

For lower Liq/FA, the volumetric strain was high as shown in Figure 6(a) and (b), while when that ratio reached 3.8 the resulting strain was enhanced due to the presence of high amount of fly ash. In general sense, the recorded shrinkage levels during this study is higher than those for Meta kaolin based Geopolymers cured at ambient temperature. This can be attributed to the enforcing nature of applying heat during this study, however, further research is needed to compare ambient temperature and sun light curing with the above results.

3.3. Soil pH Monitoring

Figure 7 shows variations of pH levels due to different temperature of initial duration time.

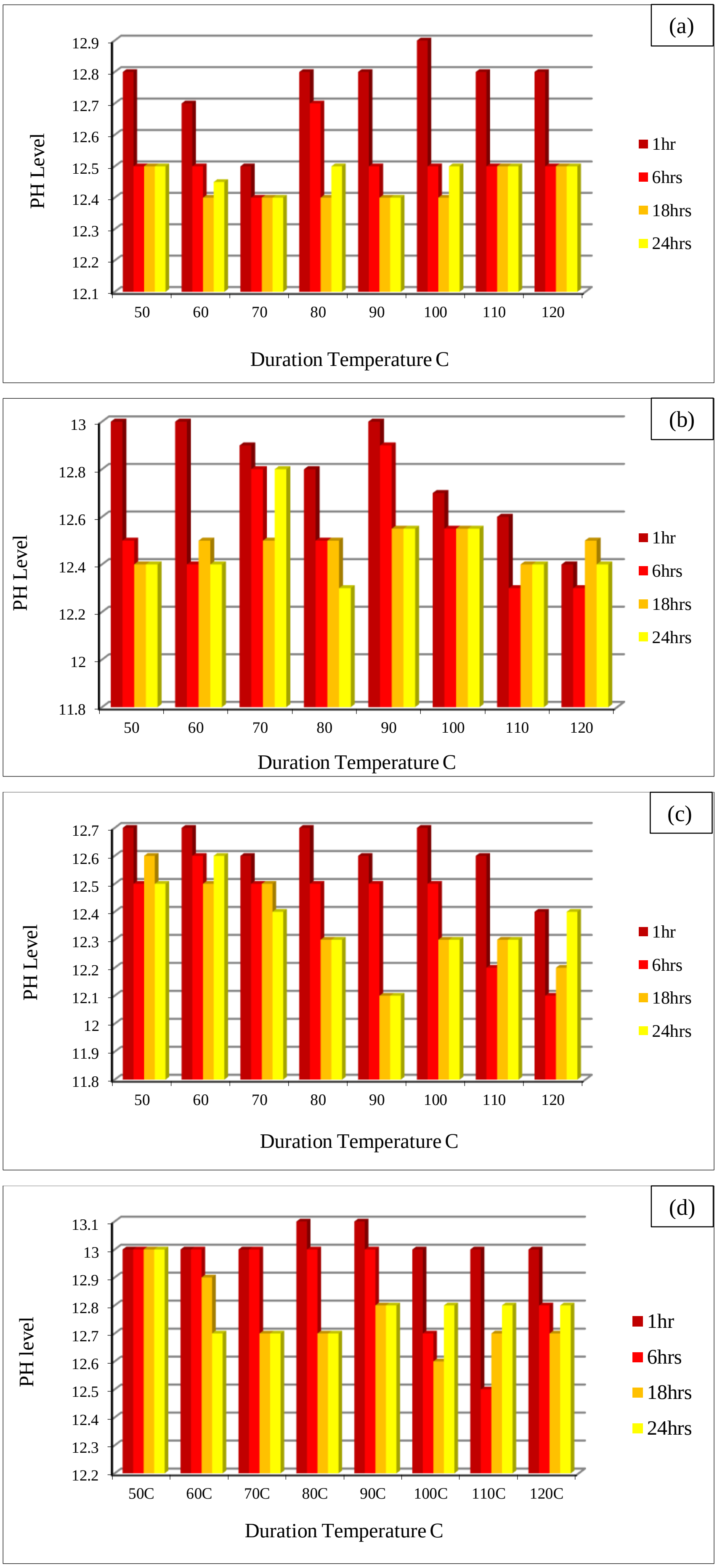


Figure 7. Variation of soil pH due to different temperatures at 7 days curing (a) 2.714 Liq/FA, (b) 3.167 Liq/FA, (c) 3.8 Liq/FA, (d) 4.75 Liq/FA

A considerable gap of pH level between 1hr and others D pH levels in low Liq/FA, it’s also obvious that this gap tends to be decreased as that ratio increased especially for early durations. This can be attributed to the high quantity of sodium hydroxide solution and low consumption of alkalinity due to the low presence of fly ash.

3.4. SEM-EDS Characterization of Stabilized Soil

The formation gel was observed to 3.8 Liq/FA for *D* = 6 hrs. Figure 8 shows *SEM* images for this soil Geopolymers recipe.

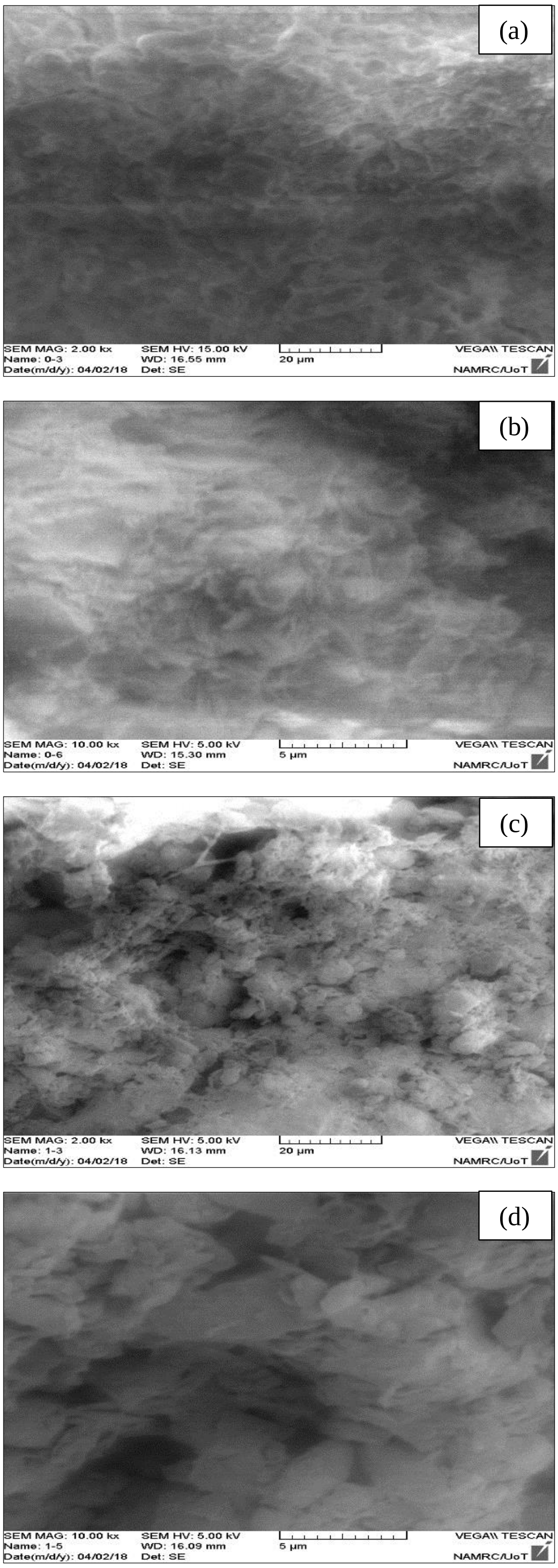


Figure 8. SEM images (a) Un stabilized soil 20 µm, (b) Un stabilized soil 5 µm, (c) 0.38 Liq/FA for D = 6 hrs 20 µm, (d) 3.8 Liq/FA for D = 6 hrs 5 µm

It can be seen from Figure 8 (c) and (d) that crumpled foil-like microstructure of the reacted binding phase was confirmed EDX spectra of that area is shown in Table 6 which indicates that gels area is rich in calcium, silicon, sodium and aluminium, some unreacted FA particles are evident.

Table 6. EDX Spectra of the Stabilized Soil

Element	Weight (%)	Atomic (%)
O	55.52	70.68
Na	5.79	5.13
Mg	1.51	1.26
Al	5.39	4.07
Si	16.17	11.73
Cl	0.18	0.10
K	1.04	0.54
Ca	8.71	4.42
Ti	0.47	0.20
Fe	4.93	1.80
Co	0.04	0.01
Zn	0.08	0.02
Hg	0.13	0.01
Pb	0.03	0.00

3.5. XRD Characterization of Geopolymers Gels

XRD pattern to 3.8 Liq/FA for D = 6 hrs, is shown in Figure 9.

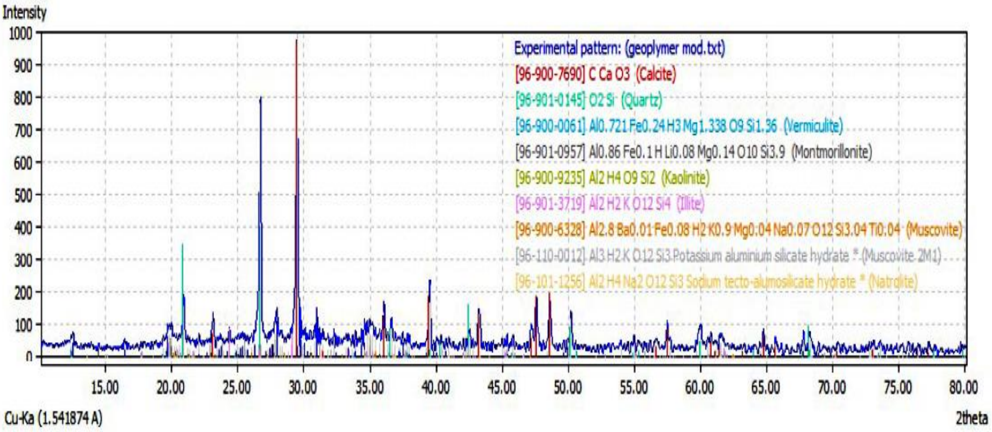


Figure 9. XRD pattern of 3.8 Liq/FA for D = 6hrs

Phase evolution was done by using Match software. The analyses results confirms formation of potassium aluminosilicate hydrate (K-A-S-H) (18.46%) and sodium aluminosilicate hydrate (N-A-S-H) (1%). no new peaks are evident which means no reaction happens between soil and Geopolymers.

4. Conclusions

- In this study, it can be concluded that applying initial duration time with different temperatures highly affect the mechanical properties of soft clay soil – fly ash based Geopolymers.
- Presence of sodium silicate highly affects soil-Geopolymers mixture due to its viscosity.
- Using sodium silicate and sodium hydroxide alone for activation needs further research.
- A clear hesitation is observed in the mechanical strength components when FA content is 8% by weight especially in 1 hour D.
- Optimum Liq/FA (with respect to peak UCS) needs a considerable degree of Geopolymerization level to be distinguished.
- It is believed by the author that the optimum curing temperature depends upon the consumed heat energy, in this way, the optimum value of temperature and its duration may change according to the thermal properties of the bonded aggregates and the percent and nature of source material.

- Further research is needed regarding the effect of NaOH molar concentration to soft soil - Geopolymers mix mechanical behaviour.
- Volumetric strain of heated soft soil – Geopolymers mixes is very high because of enforcing heating rate, this dictates further research to examine the intermittent heating periods and sun light effects.
- Similar to Meta kaolin based Geopolymers, fly ash based Geopolymers enhanced ductility and stiffness.

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