Chapter 6

Chemical Stabilization using Bench Scale Model through Electrokinetics

6.1 General

This study explores stabilizing effect of three chemical additives i) Enzymatic induced calcite precipitate (EICP), ii) Sodium Carbonate with Calcium Chloride, and iii) Sodium Silicate with calcium chloride on alkali interacted soil using electrokinetic (EK) technique. It is well known that EK is a new and innovative method for the treatment of problematic soil. EK is based on the phenomenon that the movement and action of charged particles take place under the action of a low direct current or a low potential gradient. EK involves electrolysis, electro-osmosis, electro-migration, and electrophoresis mechanism for the transportation of charged species across the soil. The combined effect of these mechanisms together with various geochemical reactions alters the chemical composition of the soil porous medium and thereby alters the physicochemical properties of the soil. The main advantage of the EK technique is that it creates a minimum disturbance to the existing soil structure and the ability to control the zone of treatment. In this chapter, the whole EK experimental programme is divided into two sections which includes the monitoring of data such as pH of electrolyte, electroosmotic flow, current and voltage during the EK test, and the investigation of geotechnical properties Atterberg limits, unconfined compressive strength, triaxial test, consolidation test after the completion of EK test. Mineralogical and morphological studies have also been performed to analyse the physiochemical change in the soil properties after the EK treatment. Apart from these, the stabilizing effect of these three chemical stabilizers on the heaving behaviour of soil due to inundation with 16M NaOH solution is also investigated. The test mechanism, monitoring of data during the test, test results, and summary of observation are discussed below.

6.2 Test Procedure and Conditions

The different concentrations of three chemicals, sample preparation, test procedures, test mechanism involved in this study are discussed in details in this section.

6.2.1 Test Procedure and Conditions

The EK test to assess the stabilizing effect of chemical stabilizers on the alkali interacted soil was carried out in a bench scale electrokinetic model. The fabrication detail, model dimension and image of the bench scale electrokinetic model are discussed in chapter 3. The influence of all three chemical stabilizers on the enhancement of geotechnical parameters of alkali interacted soil was investigated using five test combinations summarized in Table 6.1. The concentration of the constituent chemicals was selected based on the basis of literature survey. A series of 15 EK tests were conducted extensively in this study. The effect of different concentrations of these chemical stabilizers on the geotechnical properties of alkali interacted soil have been discussed in detail. The preparation of sample plays an important role in any of the EK test due to the reason that the minor variations in the density and moisture content of the specimen may impact the test results. To prepare the sample for EK test, the alkali interacted soil specimen was compacted in the soil chamber at the density corresponding to 85% of MDD and water content corresponding to saturated density. Filter papers were used at both ends of the specimen to avoid the movement of soil into the electrolyte chamber. Following this, the solution of EICP, sodium carbonate, sodium silicate, and calcium carbonate of specific molarity were filled in their respective chambers. A voltage gradient of 1 volt per cm was applied using a DC voltage supplier across the electrode. The whole EK experiment continues for 7 days. After the electrokinetic test, the sample was left for additional 7 days to allow for the completion of the reaction between chemicals and soil. Further, after the completion of electrolysis and electromigration of the solution, the treated soil samples were extracted

Table 6.1: Summary of different combinations of chemical used in EK tests

<table>
<tr><th rowspan="2">Chemical Stabilizers</th><th rowspan="2">Test Combination</th><th colspan="4">Chemical Proportions</th></tr>
<tr><th>Urea (M)</th><th>$CaCl_2$ (M)</th><th colspan="2">Enzyme (g/L)</th></tr>
<tr><td rowspan="5">EICP</td><td>C1</td><td>1</td><td>0.83</td><td colspan="2">6</td></tr>
<tr><td>C2</td><td>1</td><td>0.67</td><td colspan="2">5</td></tr>
<tr><td>C3</td><td>1</td><td>0.57</td><td colspan="2">3</td></tr>
<tr><td>C4</td><td>1</td><td>0.83</td><td colspan="2">5</td></tr>
<tr><td>C5</td><td>1</td><td>0.57</td><td colspan="2">5</td></tr>
<tr><td rowspan="6">Sodium Carbonate</td><td></td><td colspan="3">Sodium Carbonate (M)</td><td>Calcium Chloride (M)</td></tr>
<tr><td>M1</td><td colspan="3">0.5</td><td>1</td></tr>
<tr><td>M2</td><td colspan="3">0.8</td><td>1</td></tr>
<tr><td>M3</td><td colspan="3">1</td><td>1</td></tr>
<tr><td>M4</td><td colspan="3">1.2</td><td>1</td></tr>
<tr><td>M5</td><td colspan="3">1.5</td><td>1</td></tr>
<tr><td rowspan="6">Sodium Silicate</td><td></td><td colspan="3">Sodium Silicate (%)</td><td>Calcium Chloride (M)</td></tr>
<tr><td>T1</td><td colspan="3">10</td><td>1</td></tr>
<tr><td>T2</td><td colspan="3">20</td><td>1</td></tr>
<tr><td>T3</td><td colspan="3">30</td><td>1</td></tr>
<tr><td>T4</td><td colspan="3">40</td><td>1</td></tr>
<tr><td>T5</td><td colspan="3">50</td><td>1</td></tr>
</table>

and tested for Atterberg limits, consolidation test, UCS test and triaxial test.

6.2.2 Electrokinetic Mechanism and Procedure

The entire EK tests were conducted in three phases with five combinations of chemicals in each phase. In the first phase, the alkali interacted soil is treated with EICP. The EICP process utilizes the enzyme as a catalyst for the decomposition of urea into ammonium and carbonate ions. The formation of calcite precipitation from urea and calcium chloride solution necessitates specific chemical reactions in a systematic manner. The involved chemical reactions are shown in equations 6.1, 6.2 and 6.3 as follows:

$$CO(NH_2)_2 + H_2O \rightarrow NH_4^+ + CO_3^{2-} \tag{6.1}$$

$$CaCl_2 \rightarrow Ca^{2+} + 2Cl^- \tag{6.2}$$

$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3(Precipitate) \tag{6.3}$$

In this experiment, calcium chloride solution was filled in an anode chamber and the urea solution with enzyme was injected into the cathode chamber. The concentration and ratio of calcium chloride, enzyme and urea solution are mentioned in Table 6.1. for all five test combinations. When the experiment starts and voltage gradient is applied, electrolysis occurs which decomposes the urea and enzyme solution into ammonium ions ($2NH_4{}^+$) and carbonate ions (CO_3^{2-}) (Eq 6.1) and calcium chloride solution into calcium ions (Ca^{2+}) and chloride ions ($2Cl^-$) (Eq 6.2). After hydrolysis of the solution, the resultant ions move towards their respective electrode through the electromigration process. During the movement of calcium ion towards the cathode, it combines with carbonate ion which was travelling towards the anode, forming calcium carbonate precipitation (Eq 6.3). This calcium carbonate precipitation gets deposited into the soil due to its low solubility in the solution. In second phase the EK was conducted with the combination of five different moralities of sodium carbonate and calcium chloride as shown in Table 6.1 to stabilize the alkali interacted soil. The main purpose of using sodium carbonate and calcium chloride is to allow the deposition of calcium carbonate in the voids of soil. The test is performed using calcium chloride solution fed at the anode chamber and sodium carbonate solution at the cathode chamber and a voltage gradient is supplied continuously for 7 days. As the EK process start, the electrolysis of calcium chloride solution produces calcium ion and chloride ion in anode chamber whereas the electrolysis of sodium carbonate produces carbonate ion and sodium ion in cathode chamber. After the production of resultant ions move towards their respective electrode through the electromigration process. During the movement of calcium ion towards the cathode, it combines with carbonate ion which was travelling towards the anode, forming calcium carbonate precipitation. In third phase the efficiency of five different concentration of sodium silicate and calcium chloride solution as shown in Table 6.1 on the geotechnical properties of alkali interacted soil using EK technique is investigated. The main purpose of use of sodium silicate and calcium chloride is to analyze the combine effect of calcium ion and silicate ion on the alkali interacted soil. The test was carried out by feeding calcium chloride and sodium silicate at the anode and cathode respectively to know the effects of cation and anion of species in the system. After feeding the chemical solution in their chamber, a voltage gradient was applied across the soil which leads to the generation of calcium ions and silicate ions in the anode chamber and cathode chamber respectively. The generated calcium ion is transported towards the cathode which reacts with a large amount of silicate ion which is transported towards the anode through electromigration of ions. The ingression of calcium ion and silicate ion induces good cementation property which enhances the properties of alkali interacted soil.

6.2.3 Quantification of Precipitated Calcite (Acid Leaching test)

After the completion of EK test of phase 1 and phase 2, the quantity of $CaCO_3$ precipitation in the soil specimen had been measured using the gravimetric acid digestion technique (Mortensen et al., 2011). The procedure for the acid digestion experiment is illustrated in Fig 6.1. In this method, 10-15 gm of treated soil specimens were chipped off from three different locations (near the anode, middle, and cathode of the soil chamber). The collected sample had been oven-dried and weighed. Then the oven-dried sample was soaked in 4M hydrochloric acid to dissolve the precipitated $CaCO_3$. After the application of acid washing, the soil specimen was risen and washed properly with distilled water. The washed sample was oven-dried to measure the loss in weight due to acid digestion. The difference in mass before and after the acid digestion experiment was considered as the net mass of $CaCO_3$. Table 6.2 shows the precipitation percentage corresponding to each test combination. The value of precipitation ratio has been averaged for three sam-

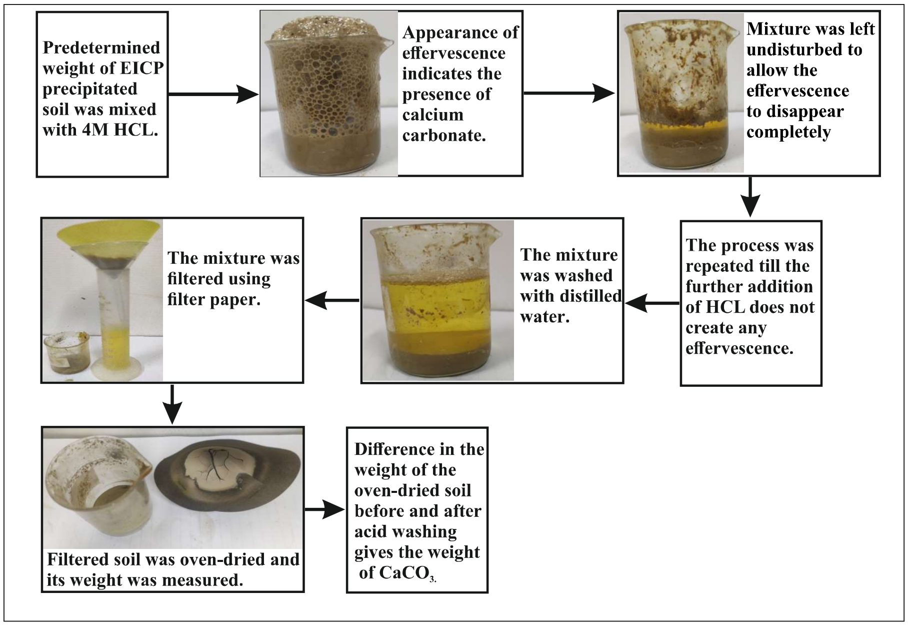

Figure 6.1: Step-by-step procedure for acid leaching test

Table 6.2: Variation in $CaCO_3$ with different combination of chemicals

Test Combinations	$CaCO_3$ Percentage	Test Combinations	$CaCO_3$ Percentage
C1	5.6	M1	4.7
C2	6.3	M2	5.2
C3	4.2	M3	6.4
C4	4.8	M4	7.8
C5	6	M5	8.4

ples collected from three different locations along the specimen length. The acid leaching test was also performed on the uninteracted soil and alkali interacted soil. No significant change in weights was observed after the acid leaching test on both the soil.

6.3 Monitoring of Data During EK Test

The variation in pH of electrolyte, electroosmotic flow, current, and voltage with time during EK test was monitored carefully. The change in all these parameters is discussed in detail in this section.

6.3.1 Variation of pH of Electrolytes and Soil

The variation in the value of pH depends on the types of electrolytes used at their respective chambers. The electrolyte solutions were extracted from both electrolyte compartments at regular intervals for measuring the pH value of the electrolyte. The pH value of anolyte and catholyte for EICP, calcium chloride-sodium carbonate, and calcium chloride-sodium silicate are shown in Fig. 6.2, Fig. 6.3, and Fig. 6.4 respectively. The initial value of pH for $CaCl_2$ solution in the anode chamber and urea-enzyme solution in the cathode chamber was nearly the same for all the combinations of EICP. While a slight change in the initial pH value of the catholyte solution has been observed in the other two cases due to a change in concentration. During the EK test, with time, a significant change in pH was observed in both chambers. Fig. 6.2 shows that the pH of the cathode chamber increased to a maximum value of 13.96 while the pH of the electrolyte in the anode chamber decreases continuously up to a value of 2.3. The maximum and minimum pH value of catholyte and anolyte solution from all different combinations of calcium chloride and sodium carbonate was observed as 12.58 and 2.53 respectively as shown in Fig. 6.3.

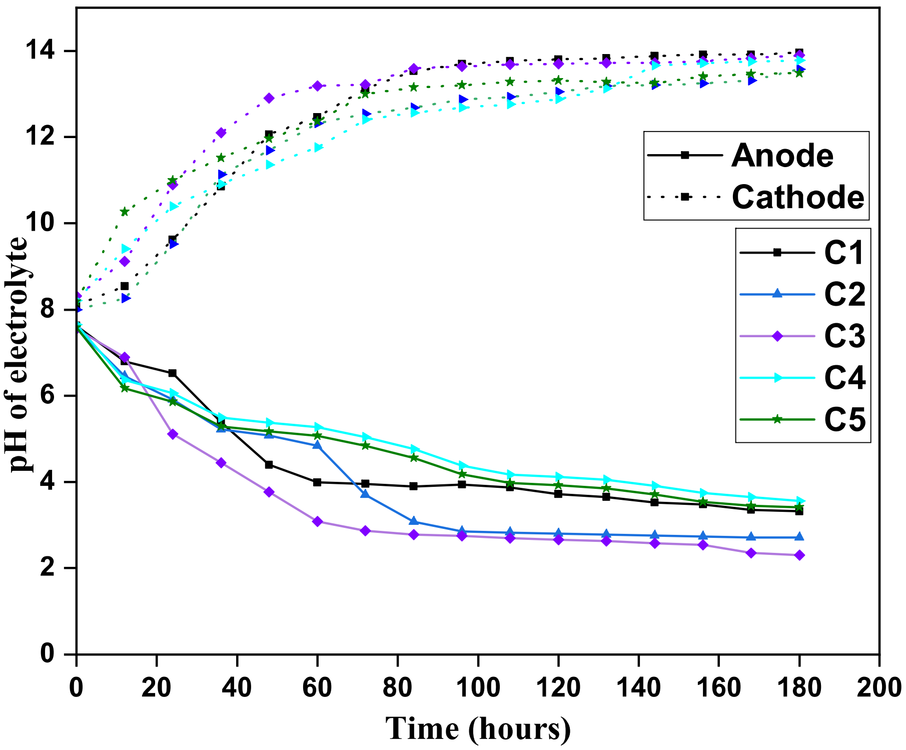

Figure 6.2: Variation of pH of anolyte and catholyte with EICP

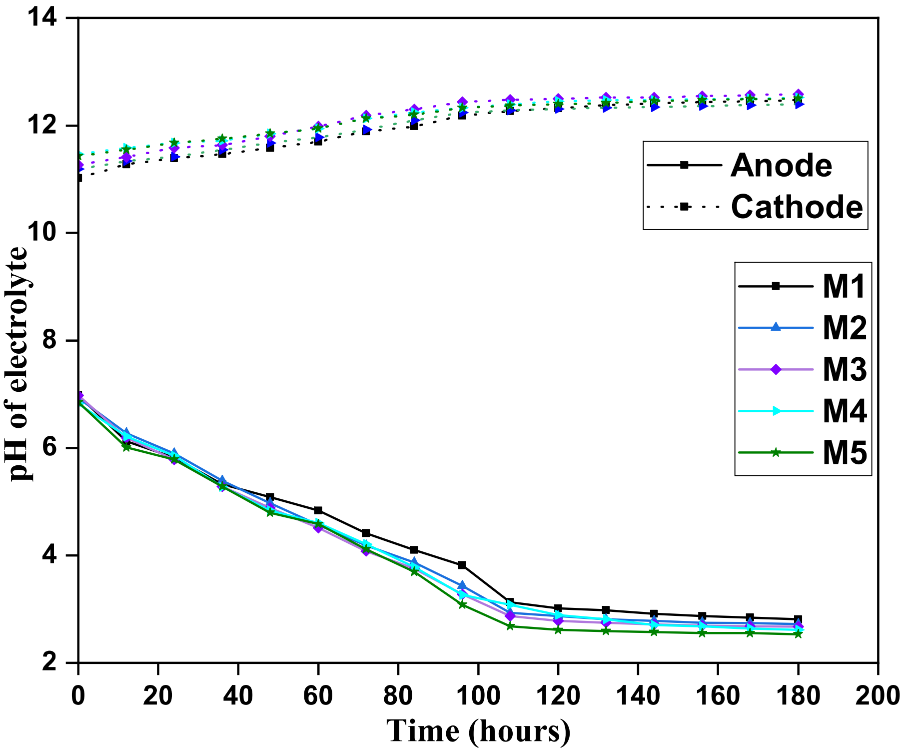

Figure 6.3: Variation of pH of anolyte and catholyte with sodium carbonate

The pH values of the catholyte in Fig. 6.4 increased up to 11.93 in contrast the minimum pH value of an anolyte was observed as 2.33.

The variation in pH in catholyte and anolyte solution can be attributed to the generation of the acid front at the anode and the base front at the cathode. The applied voltage gradient leads to the electrolysis of ionic solutions that initiated EK redox reactions, which generated H^+ and OH^- at the cathodic end to produce the base front and generated O_2 and H^+ at the anodic end, to produce the acid front. The furthermore reduction in pH of the anolyte solution in all combinations of all three phases of the EK test is due to the dissociation of $CaCl_2$ into calcium and chloride ions. The positively charged carbonate ions travel towards the cathode while the production of chloride ions reduces the pH in the anode chamber (Yasuhara et al., 2012). However, in the case of EICP, the application of direct current induces the electrolysis of urea-enzyme solution in the cathode chamber releasing thereby ammonia and carbonate ions. The negatively charged carbonate ions travel towards the anode while the ammonia ion increases the pH in the cathode chamber which in turn enhances the EICP process.

After the completion of the EK test, the pH test was conducted to investigate the

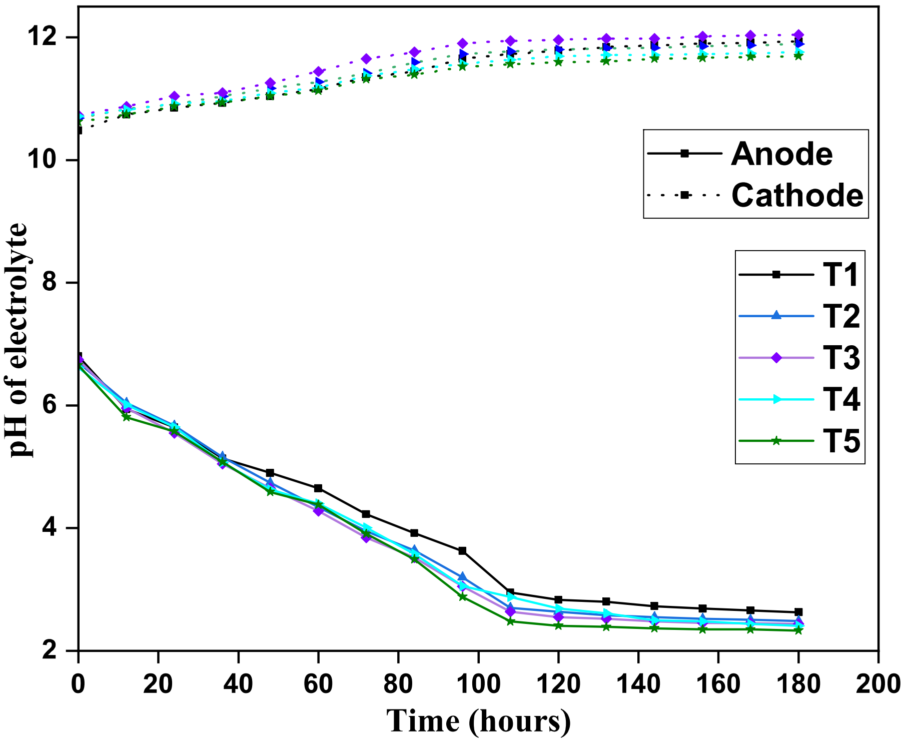

Figure 6.4: Variation of pH of anolyte and catholyte with sodium silicate

effect of ingression of different combinations of all three chemical through EK on the alkali interacted soil. For this the treated soil sample was collected at three different locations and pH test was conducted on that sample according to respective ASTM code. Fig. 6.5, Fig. 6.6 and Fig. 6.7 present the pH value of EK treated soil measured at three different locations by using all different combinations of ionic solutions of all three chemicals. The figures show that, after the EK treatment with all different combination of three chemicals, the pH value of the soil specimen increases at the middle portion and near the cathode whereas the pH value of soil decreases near the anode. The pH value of the soil specimen treated with EICP increases at the middle portion and near the cathode with the values being 12.86 and 13.4 respectively whereas the pH value of soil decreases near the anode with the value of 11.22.

A similar change in pH value of the soil specimen was observed in Fig. 6.6 for alkali interacted soil treated with calcium chloride and sodium carbonate. The pH value increases to 12.68 near the cathode and 10.15 at the middle portion respectively whereas the pH value of soil decreases near the anode with the value of 8.22.

A similar trend was also depicted in Fig. 6.7 which shows that the pH value of soil

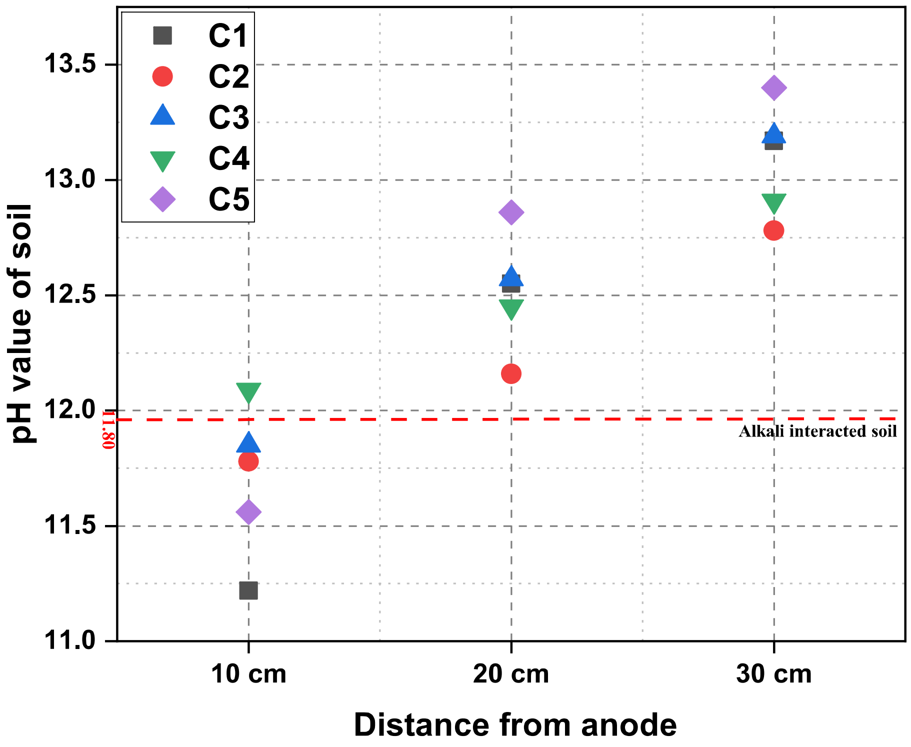

Figure 6.5: Variation in pH of soil at different locations treated with EICP

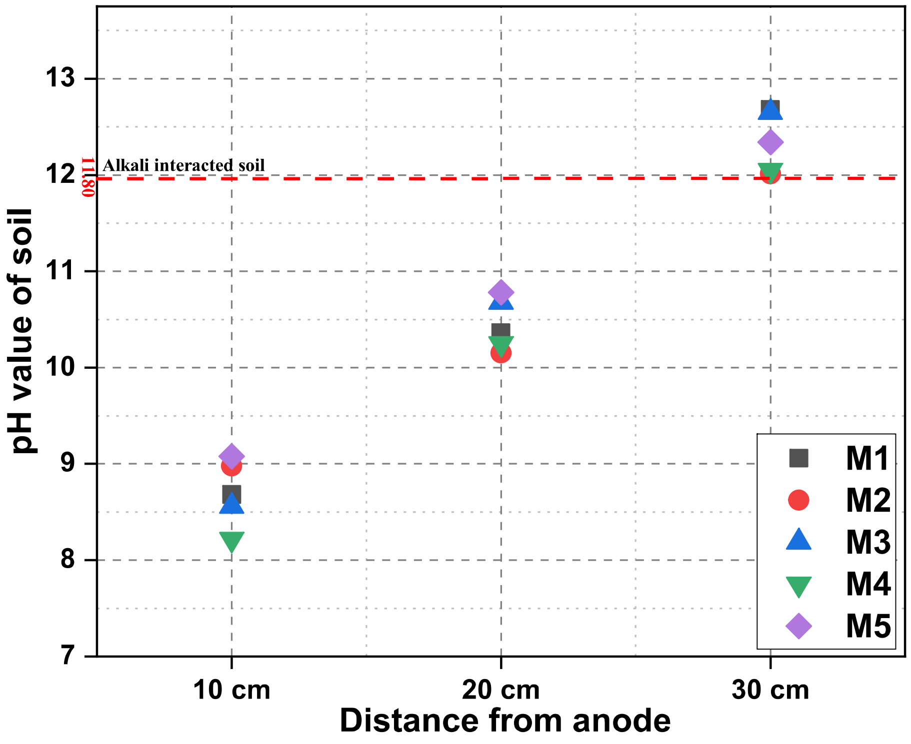

Figure 6.6: Variation in pH of soil at different locations treated with sodium carbonate

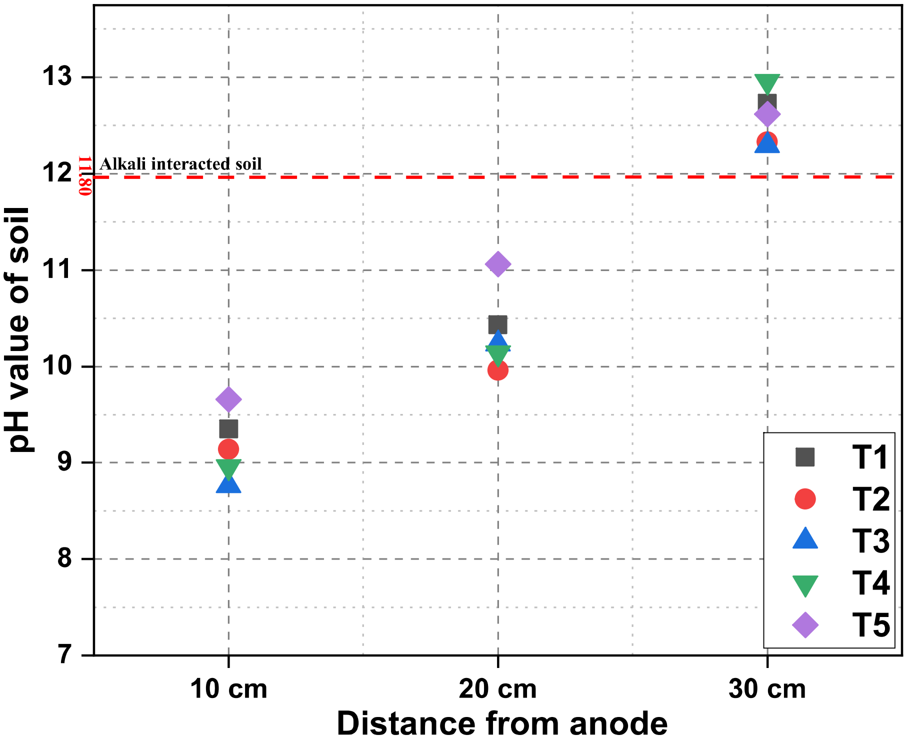

Figure 6.7: Variation in pH of soil at different locations treated with sodium silicate

increases at the middle portion and near the cathode with the values being 11.06 and 12.96 respectively, whereas, the pH value of soil decreases near the anode with the value of 8.76 after the EK treatment of alkali interacted soil with calcium chloride and sodium silicate solution. The reduction in pH values of soil near the anode after the EK test was due to the release of hydrogen ions from the electrolysis process, which then contribute to the acidity environment near the anode. While the increment of pH values near the cathode after EK is due to the release of hydroxide ions that contribute to the alkalinity environment near the cathode. Apart from these, the change in pH value of chemically treated soil is also associated with the flow of the stabilizing ions from their respective reservoir into soil affecting the physicochemical behaviour of soil (Mosavat et al., 2013).

6.3.2 Variation in Electric Potential

Fig. 6.8, Fig. 6.9, and Fig. 6.10 shows the profile of electric potential with time for all three phases of EK test. From the figures, it can be seen that initially, electric potential increases with time then after reaching a peak it decreases. The electrical potential of soil is a function of the ionic strength of the soil pore fluid and the surface characteristics of the particles (Ozkan et al., 1999). Thereby, the higher electric potential at the beginning of the system is due to the increase of ionic strength of the soil pore fluid from the addition of chemical ions, electrolysis product and dissolution of chemical compounds and clay minerals. The increase in the electric potential of soil can also be attributed to

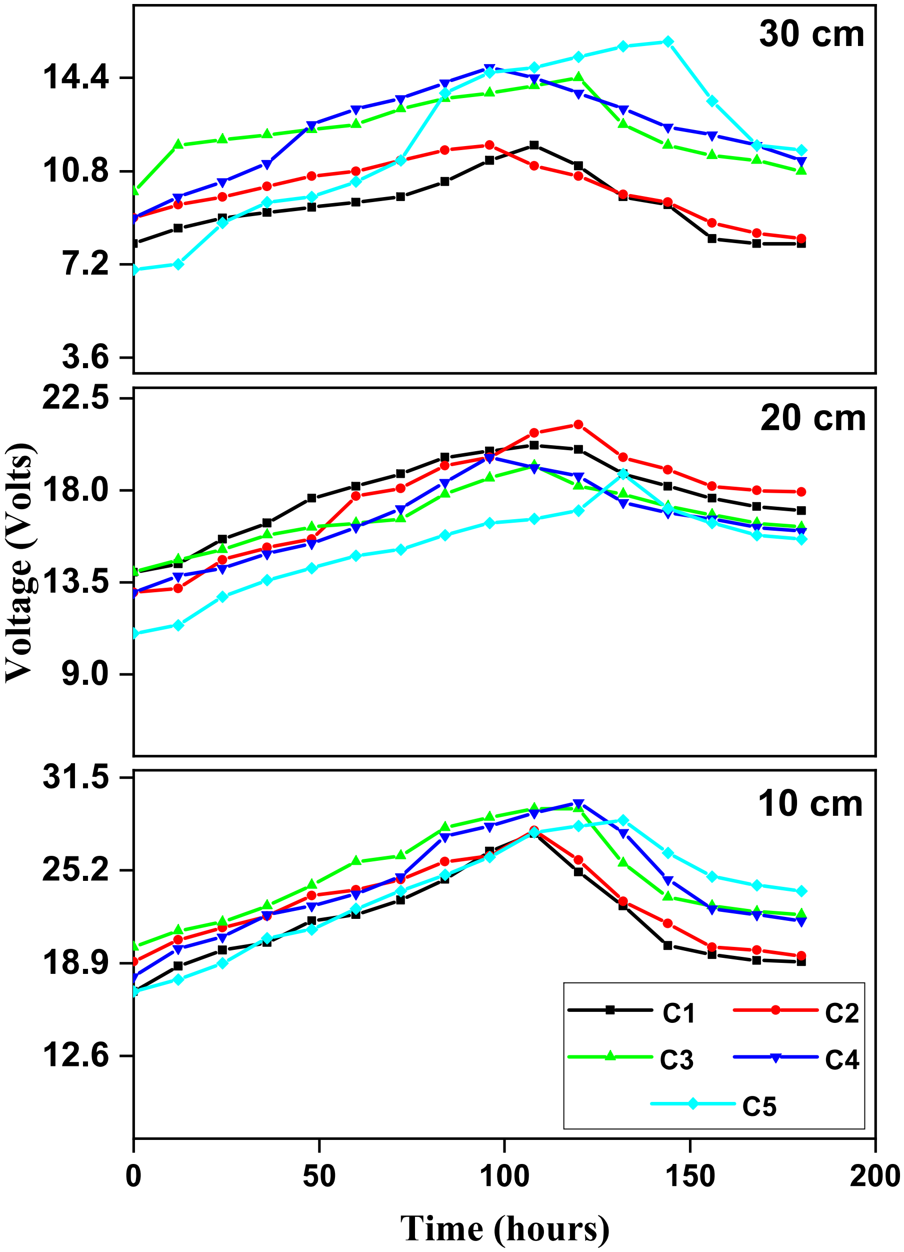

Figure 6.8: Variation of voltage at different locations after EICP treatment

the electromigration of ions between the electrode after the electrolysis of the chemical solution. The movement of ions resulted in a change in the soil conductivity which cause the variation in the electric potential. The higher value of electric potential was observed

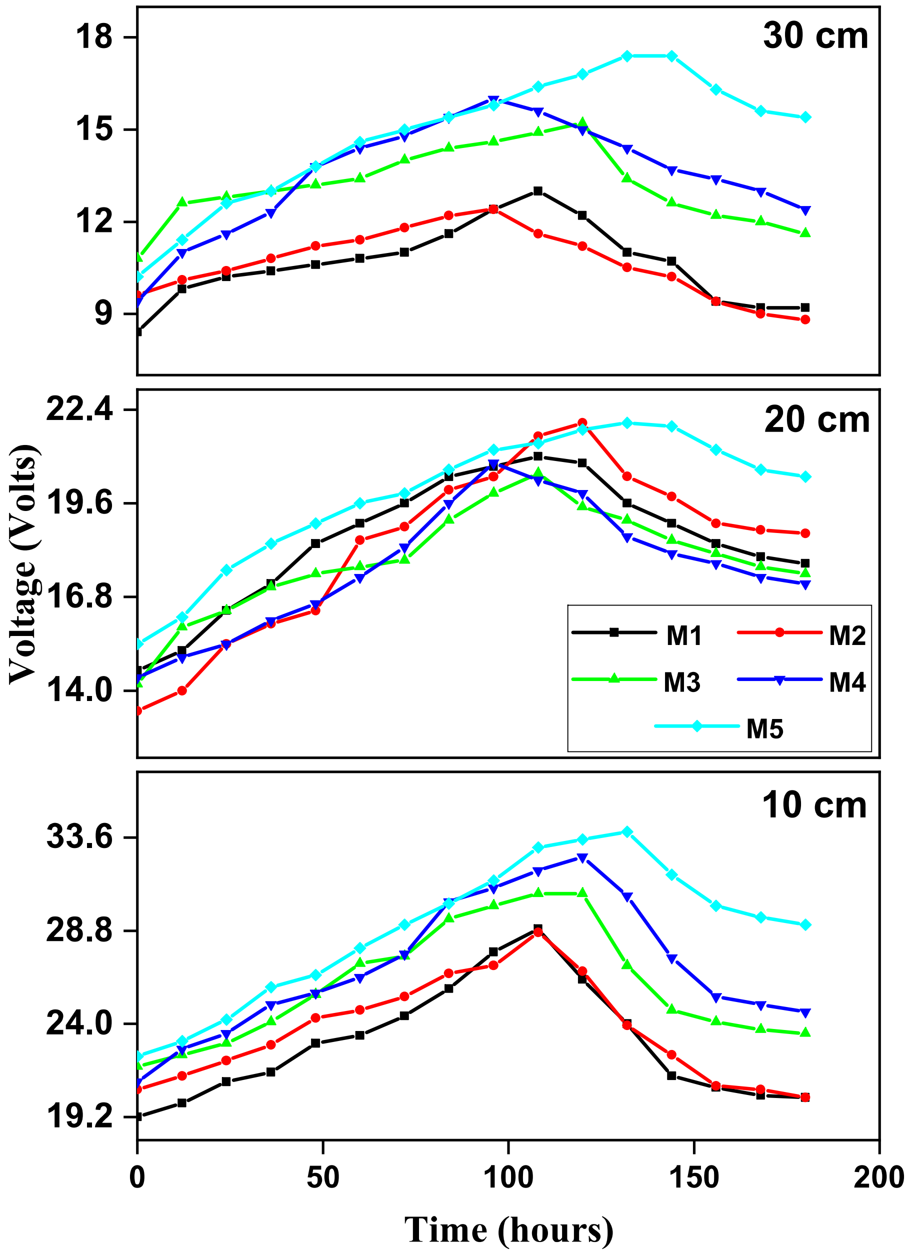

Figure 6.9: Variation of voltage at different locations after sodium carbonate treatment

near the anode and it gets decreases towards the cathode. The high value of the electrical potential at the anode area is mainly due to the higher solubility of stabilized ions under acid environments. Whereas, the decrease of electrical potential values at the cathode compared to the values at the anode was mainly due to the lower solubility of these ions under an alkaline environment which leads to the precipitation of ions near the cathode zone. A similar kind of behavior was also reported by Keykha et al. (2014). In the case of EK treatment using EICP and calcium chloride and sodium carbonate, the reduction

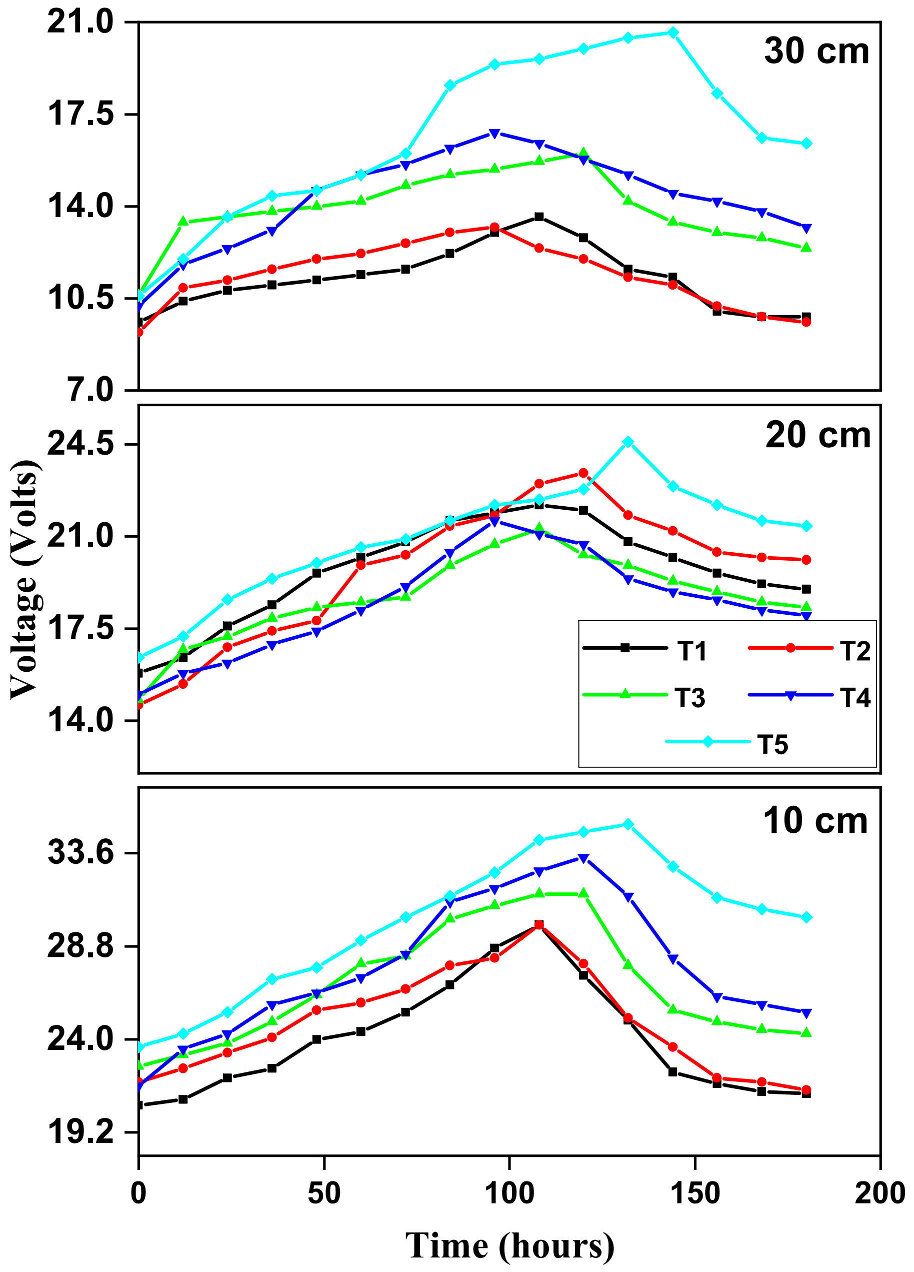

Figure 6.10: Variation of voltage at different locations after sodium silicate treatment

in electric potential after attaining the peak was due to the precipitation of $CaCO_3$ in the soil voids which causes obstruction in the movement of ions (Baveye et al., 1998). Whereas in the case of calcium chloride and sodium silicate the insoluble stabilized ion act as a cementations agent which is filled in the voids between soil particles and also reduces the electric potential.

6.3.3 Variation in Electroosmotic Flow During EK Process

The closed geometry of the EK setup allows water to enter at the anode and leave at the cathode only. The fluid flowing from the anode towards the cathode during electroosmosis was collected in the cathode chamber. During the EK test, it was observed that after certain time water travelled from anode to cathode through the soil. The amount of water entering the system (inflow) and leaving the system (outflow) was monitored continuously with time to check the efficiency and the balance of the system. The variation of cumulative discharge volume, as well as per day, drained volume with time during EK treatment with different chemicals are shown in Fig. 6.11, Fig. 6.12 and Fig. 6.13 respectively. The results of electroosmotic flow for all EK tests show similar pattern suggesting that the rate of discharge volume in electroosmotic flow was mainly controlled by ionic solutions, surface charge, charge ion concentration, and soil properties used in this study (Alshawabkeh and Bricka, 2000; Mosavat et al., 2013). It clearly shows that the electroosmotic flow of water was higher during the initial period of the EK test. After that period, the electroosmotic flow of water starts to decrease and became constant at the end of the experiment. This was attributed to the use of sodium silicate solution at

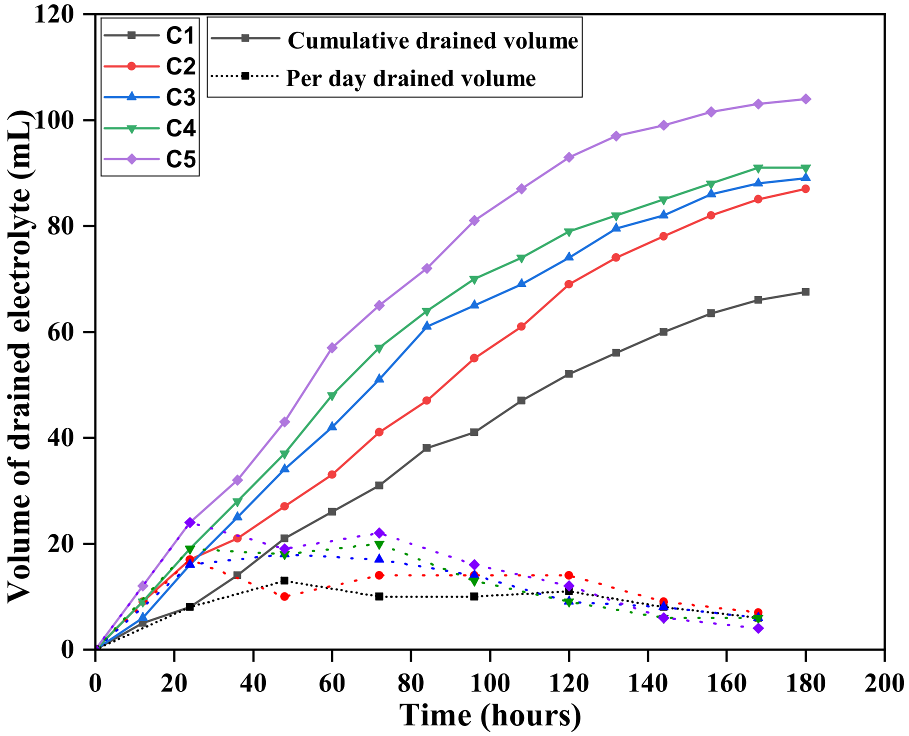

Figure 6.11: Variation of electroosmotic flow with time (EICP)

the cathode and the use of calcium chloride solution at the anode effect electroosmosis and electromigration processes in the system. It is evident from Fig. 6.11 that the total

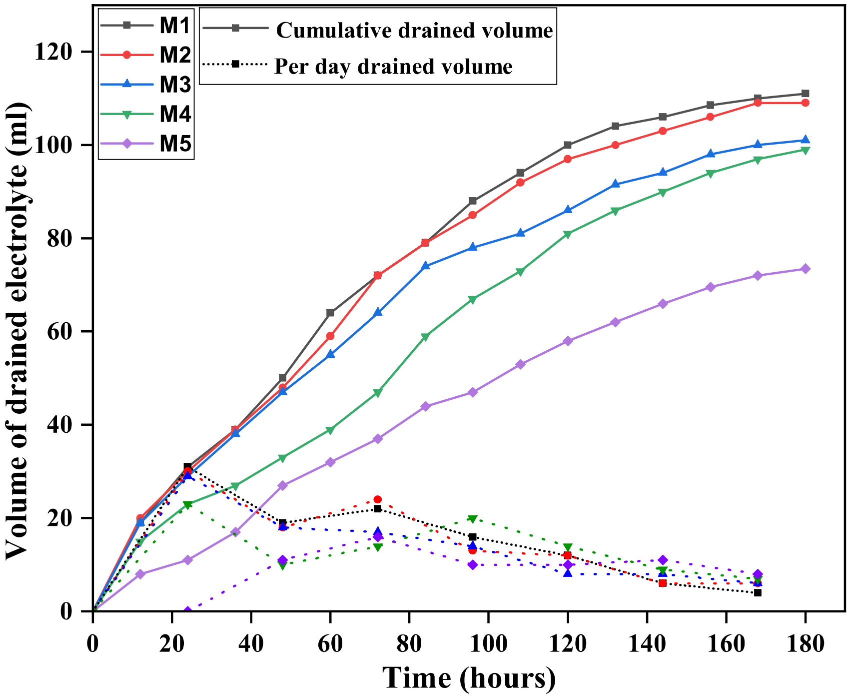

Figure 6.12: Variation of electroosmotic flow with time (sodium carbonate)

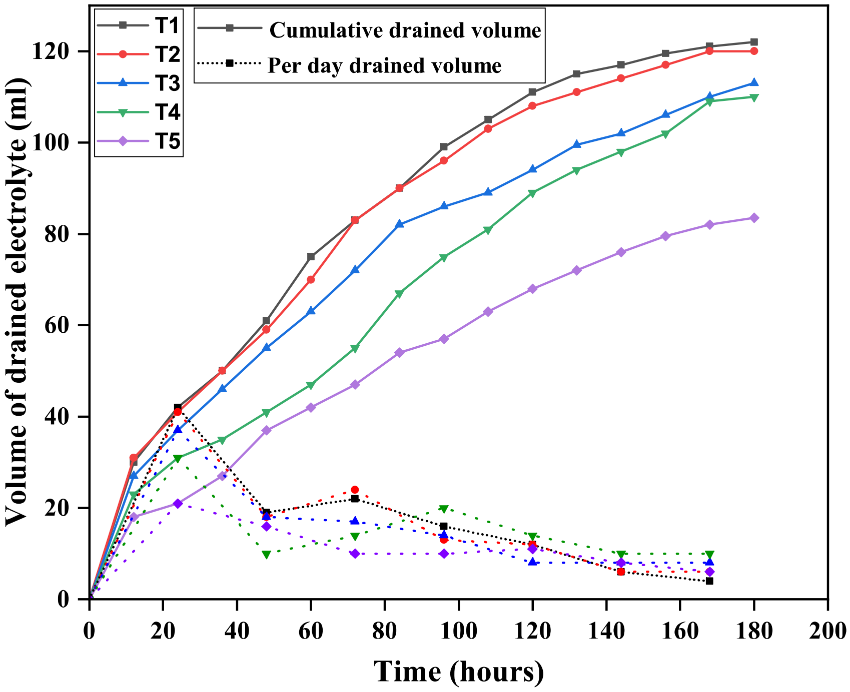

Figure 6.13: Variation of electroosmotic flow with time (sodium silicate)

volume of discharge at the end of the test was 140 mL for combination C5 and 67.5 mL for combination C1. It can also be seen that the amount of fluid collected in the cathode chamber is inversely proportional to the calcite precipitation ratio as a higher precipitation ratio tends to reduce the soil permeability which in turn hinders the electroosmotic flow. However, in the case of calcium chloride-sodium carbonate and calcium chloride-sodium silicate, the decrease in the total volume of discharge is observed with an increase in the concentration of chemicals used. The decrease in electroosmotic flow is mainly due to the precipitation of calcite and silicate salt in the voids of soil.

6.4 Results of Geotechnical Testing After EK Treatment

After the completion of the EK test for a 7-day interaction period, the samples were kept undisturbed for another 7-days to allow the residual reactions to take place. At the end of this period, the samples were tested for Atterberg limits, unconfined compressive strength, triaxial test, and consolidation test. For the consolidation test, the sample was extracted using a consolidation ring from the anode side, cathode side, and middle of the soil specimen. The cylindrical sample of size 38mm in diameter and 76mm in height were extracted from the middle portion of the soil specimen for unconfined compressive strength and triaxial test. The remaining treated soil was oven dried for Atterberg's limit test. The samples extraction technique is discussed in the flow diagram as shown in Fig. 6.14. The change in these geotechnical properties after the EK test was discussed in detail in the subsequent section.

6.4.1 Variation of Atterberg Limits of Post EK Treated Soil

The liquid limit test on alkali interacted soil after the EK treatment was conducted using the cone penetration method to assess the efficacy of a chemical stabilizer on alkali interacted soil. Whereas the plastic limit of soil was determined through the conventional rolling technique. Fig. 6.15, Fig. 6.16, Fig. 6.17, Fig. 6.17, Fig. 6.19, and Fig. 6.20 shows the variation in liquid limit and plastic limit of alkali interacted soil after EK treatment with all five different combinations of all three chemical stabilizers respectively.

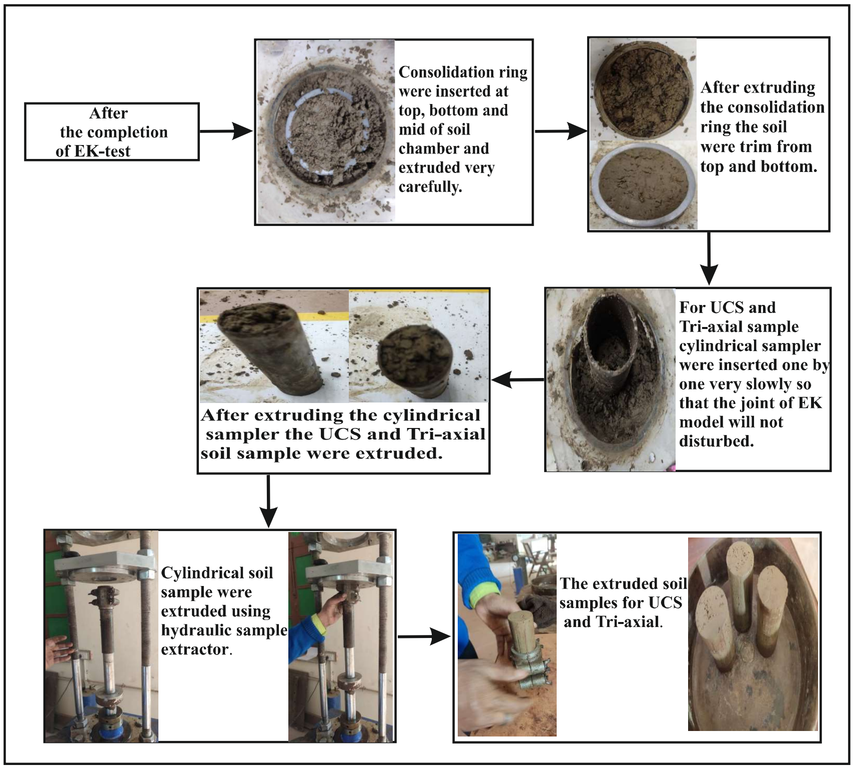

Figure 6.14: Flow diagram of sample extraction

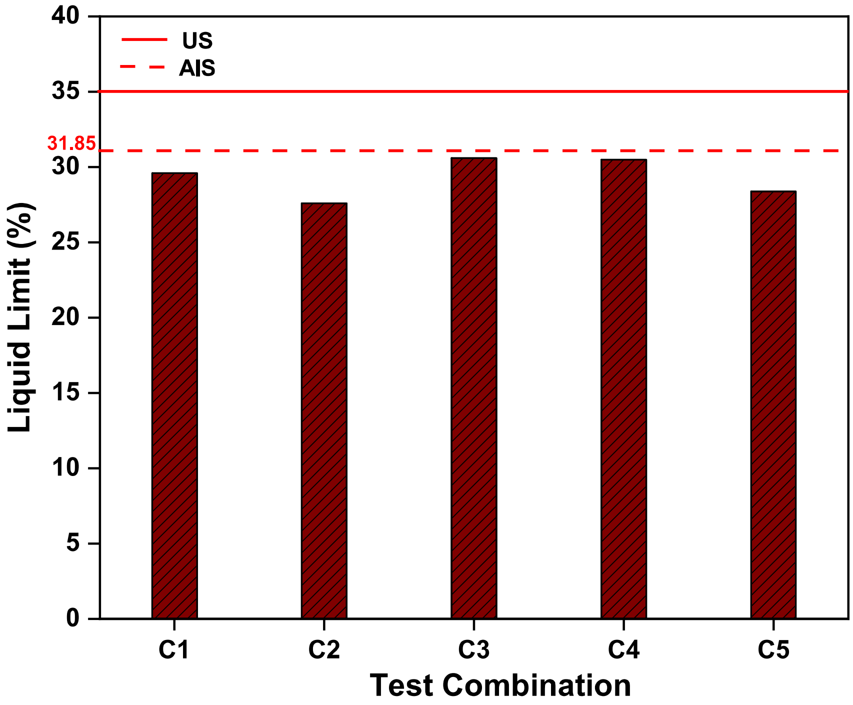

Figure 6.15: Variation in Liquid Limit values for various test combinations of EICP

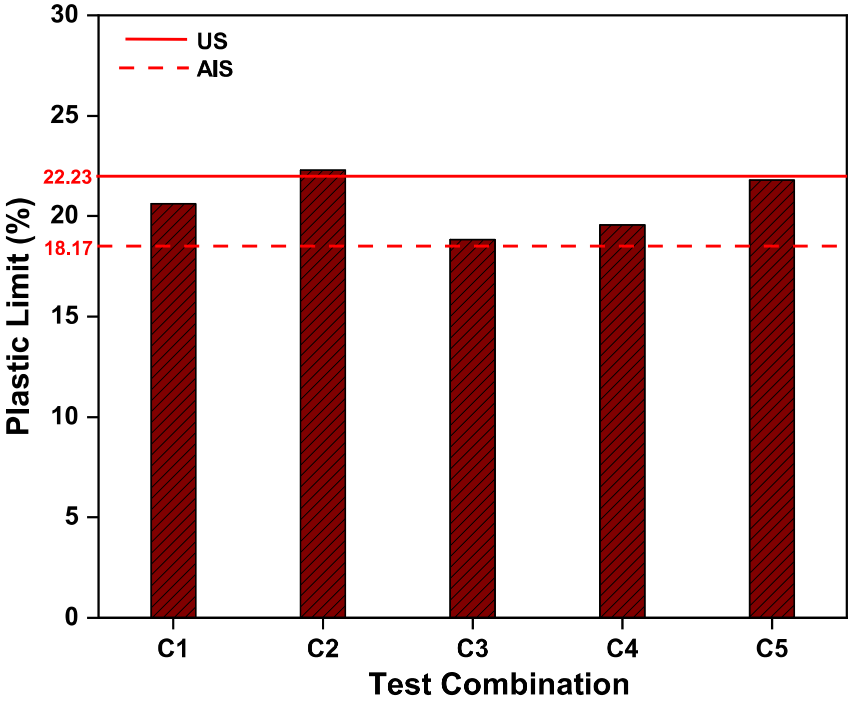

Figure 6.16: Variation in Plastic Limit values for various test combinations of sodium carbonate

The soil plasticity is a function of the physicochemical properties of the clay-ionic solutions and the moisture content of the soil. The changes in soil chemistry affected its moisture content during the EK process and resulted in changes in the liquid limit (LL), and plastic limit (PL) of the EK-treated soils. Fig. 6.15 shows the reduction in liquid limit and Fig. 6.16 increase in plastic limit value of alkali interacted soil with EICP. The reduction in liquid limit and plastic limit after EICP treatment can be attributed to the associated enzymatic activities. The enzymatic process supports the calcium carbonate precipitation and in turn the clogging of the soil voids. The formation of calcium carbonate gives the cementitious effect which improves the cohesive and adhesive force between the soil particle resulting reduction in liquid limit value (Tiwari et al., 2021). The calcite mineral formed in the soil causes the aggregation of soil particles which increases the binding of soil particles resulting in a more stabilized soil matrix (Muguda and Nagaraj, 2019). Furthermore, the movement of calcium ions during the EK process starts replacing the sodium and hydrogen ions which causes the reduction in diffuse double layer. Hence, the decrement in the soil plasticity can be anticipated (Choobbasti et al., 2019).

A similar trend has been also observed in the case of EK-treated soil with calcium

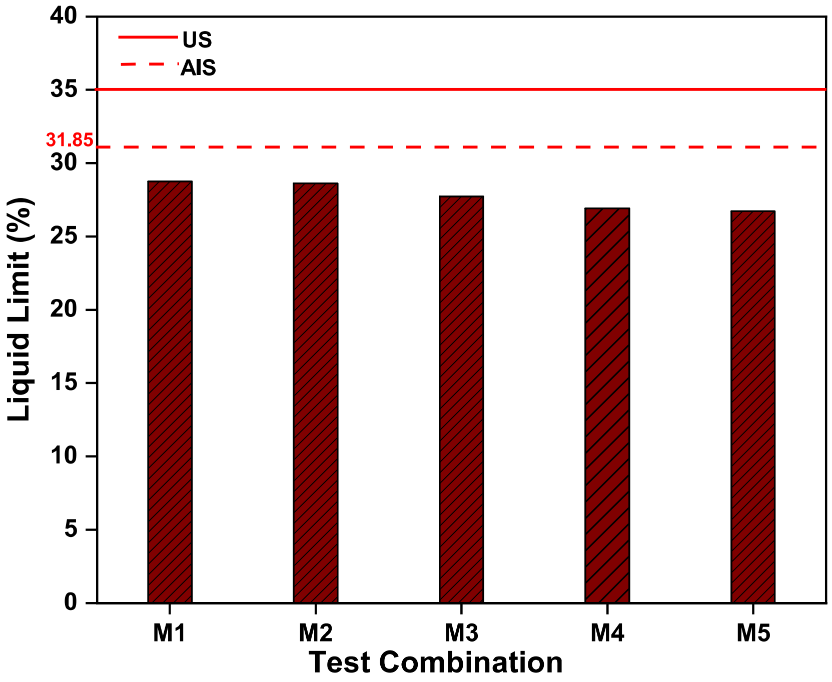

Figure 6.17: Variation in Liquid Limit values for various test combinations of sodium carbonate

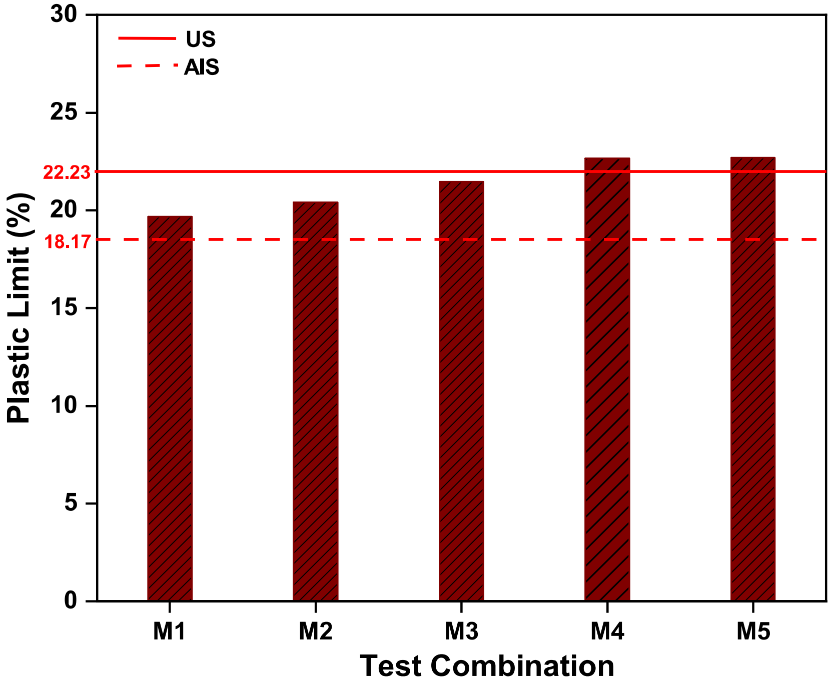

Figure 6.18: Variation in plastic Limit values for various test combinations of sodium carbonate

chloride and sodium carbonate as shown in Fig. 6.17 and Fig. 6.18. The supply of

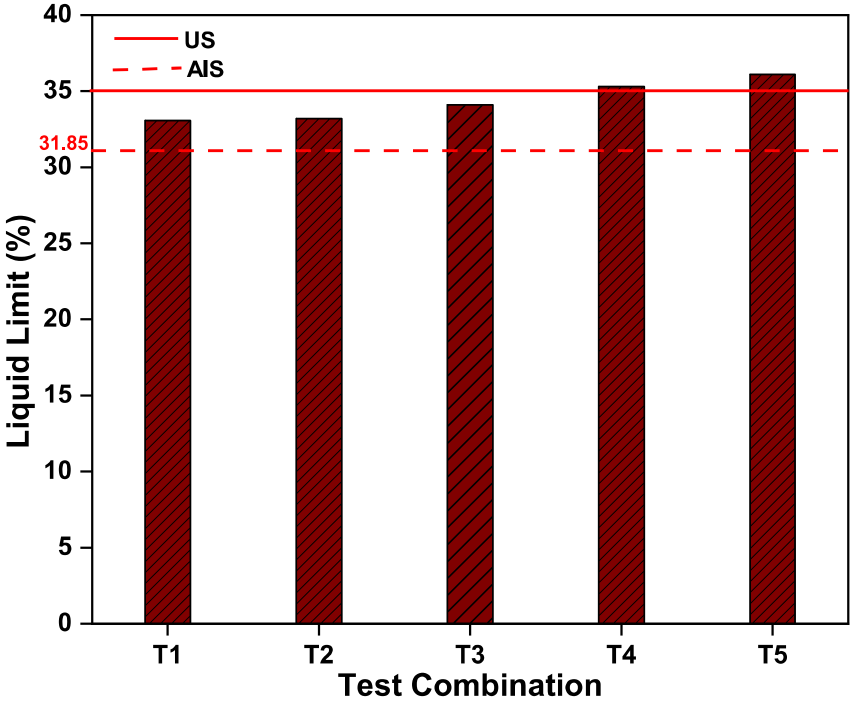

Figure 6.19: Variation in Liquid Limit values for various test combinations of sodium silicate

calcium ionic solution into the soil voids decreased the soil plasticity of the treated soil and a reduction in soil plasticity was observed at the cathode region due to carbonate solution ingression. The reaction between calcium ions and carbonate ions also leads to the precipitation of calcium carbonate in the voids of soil which shows the binding of soil particles leads to a change in liquid limit and the plastic limit value of alkali interacted soil. The change is predominant with the concentration of sodium carbonate.

However, a completely different trend has been observed as shown in Fig. 6.19, and Fig. 6.20 for the EK treated alkali interacted soil with calcium chloride and sodium silicate. The liquid limit value of alkali interacted soil increases and the plastic limit value decreases after EK treatment. The liquid limit and plastic value vary from 33.08% to 36.1% and 17.51% to 14.49% respectively with different concentrations of sodium silicate.

The introduction of calcium ion and silicate ion due to the EK test alters the clay structure after the cation exchange reaction which results in the variation in Atterberg limit values. The thickening of the diffuse double layer of clay particles due to the attachment of multivalent cations on the clay surface supports the change in the Atterberg

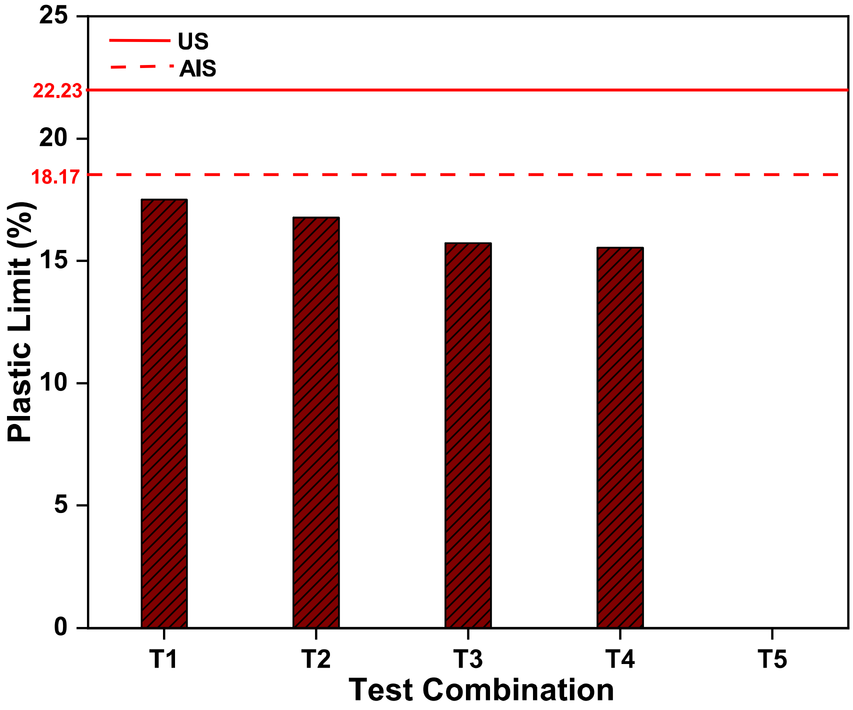

Figure 6.20: Variation in plastic Limit values for various test combinations of sodium silicate

limit value of alkali interacted soil after the EK test. The reaction between calcium ion and silicate ion leads to precipitation of insoluble salt between soil voids which alters the soil plasticity of alkali interacted soil. The significant change of liquid limit in this system confirmed the changes in clay structure due to the electrochemical effects, which in turn increase the soil shear strength as discussed in the subsequent section.

6.4.2 Variation in Unconfined Compressive Strength of Alkali Interacted Soil after EK Treatment

The unconfined compressive strength test was conducted on the cylindrical sample, extracted after EK treatment to assess the efficiency of different EK chemical stabilizers on strength property of alkali interacted soil. The variation in unconfined compressive strength of alkali interacted soil with different combinations of all EK chemical stabilizers are shown in Fig. 6.21, Fig. 6.22, and Fig. 6.23 respectively. The increase in unconfined compressive strength of alkali interacted soil after EK treatment with EICP and calcium chloride-sodium carbonate is mainly due to a double exchange reaction between ions which results in the $CaCO_3$ precipitation. The increase in strength of EK-treated soil can be associated with the amount of $CaCO_3$ precipitation into the soil voids. The variation in unconfined compressive strength with $CaCO_3$ percentage is also shown in Fig. 6.21 and Fig. 6.22. In comparison, the alkali interacted soil exhibits UCS value of 103 kPa, a significant improvement in the UCS of alkali interacted soil has been observed after EK treatment with EICP and calcium chloride-sodium carbonate.

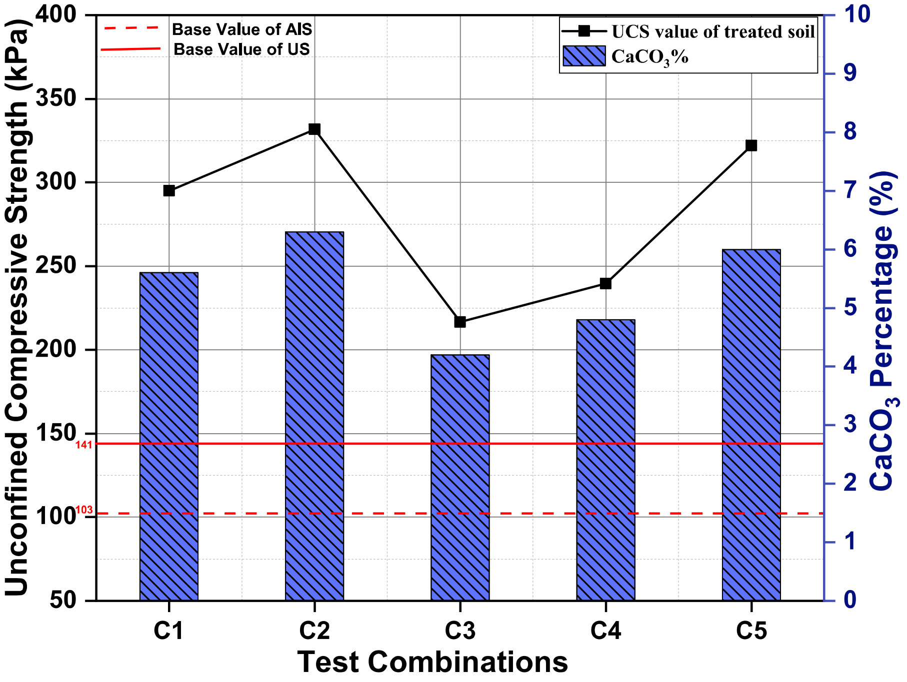

Figure 6.21: Variation of UCS for different test combinations of EICP

The increase in UCS value is primarily attributed to the strong short-term reaction between clay and stabilizers, allowing for flocculation. The electrolysis of ionic solution generates Ca^{2+} and CO^{3-} ions in their respective electrolytic chamber and electromigration allows the movement of these ion into the soil leads to the deposition of $CaCO_3$ in the soil voids. The $CaCO_3$ precipitation in the voids of clay particles form a package between clay particles, thus generating cementation or bond between clay particles. Moreover, the particle void filled with precipitated $CaCO_3$ may increase the soil density in terms of increasing the UCS. The formation of $CaCO_3$ facilities a dense soil matrix and bring the soil particle together which could be a reason for the increment in UCS value. In addition to this, the large specific surface area of clayey particles provides greater particle contact for the calcite bond formation to improve the UCS of soil (Sharma and Ramkrishnan,

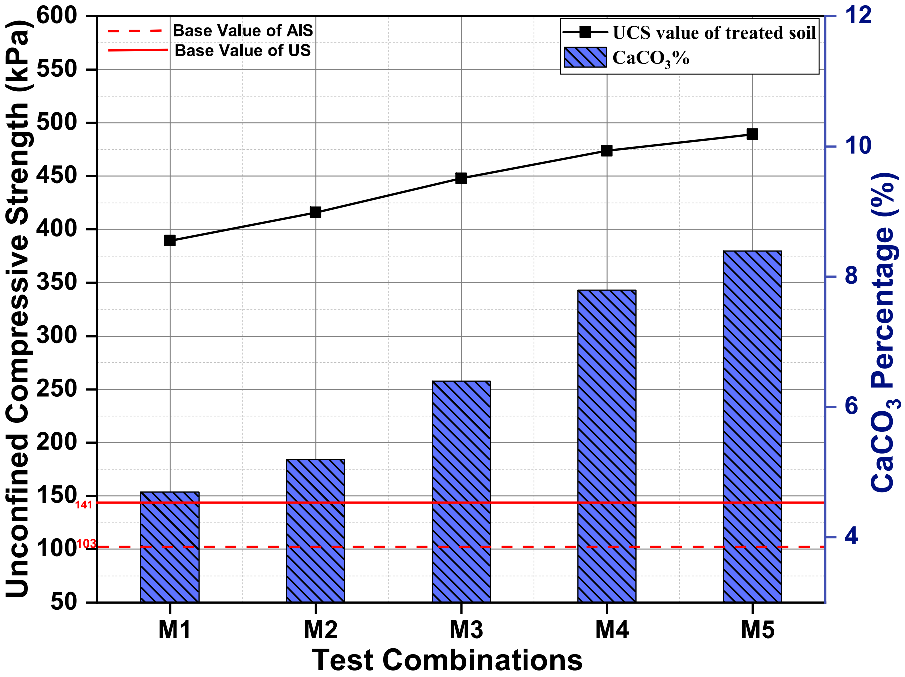

Figure 6.22: Variation of UCS for different test combinations of Sodium carbonate

2016). The deposition of $CaCO_3$ precipitation leads to adhere the soil particle to each other which form a better particle parking, which in turn led to the strong bonding between the soil particles and enhanced the unconfined compressive strength of soil (Moghal et al., 2020; Muhammed et al., 2021). The increasing values of UCS also attributed with the precipitation of $CaCO_3$ crystal that provides more contact area between soil particles which increases the shearing resistance of soil (Mujah et al., 2018). The increase in strength due to the production of calcite precipitation can also be explained by the formation of an integrated matrix that binds the soil particles together.

The unconfined compressive strength profile of the EK treated alkali interacted soil with calcium chloride-sodium silicate solution shows a clear pattern of improvement with regards to the concentration of sodium silicate as shown in Fig. 6.23. An increase in UCS value has been observed from 103 kPa to 464.37 kPa after the EK treatment. The increase in UCS value is mainly due the formation of good cementation between the particles after interaction with calcium chloride and sodium silicate solution. The migration of Ca^{2+} and SiO_3^{2-} present in calcium chloride and sodium silicate solution in the soil under an electric field leads to the formation of cementing agents consisting of calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH) after the pozzolanic reaction. The

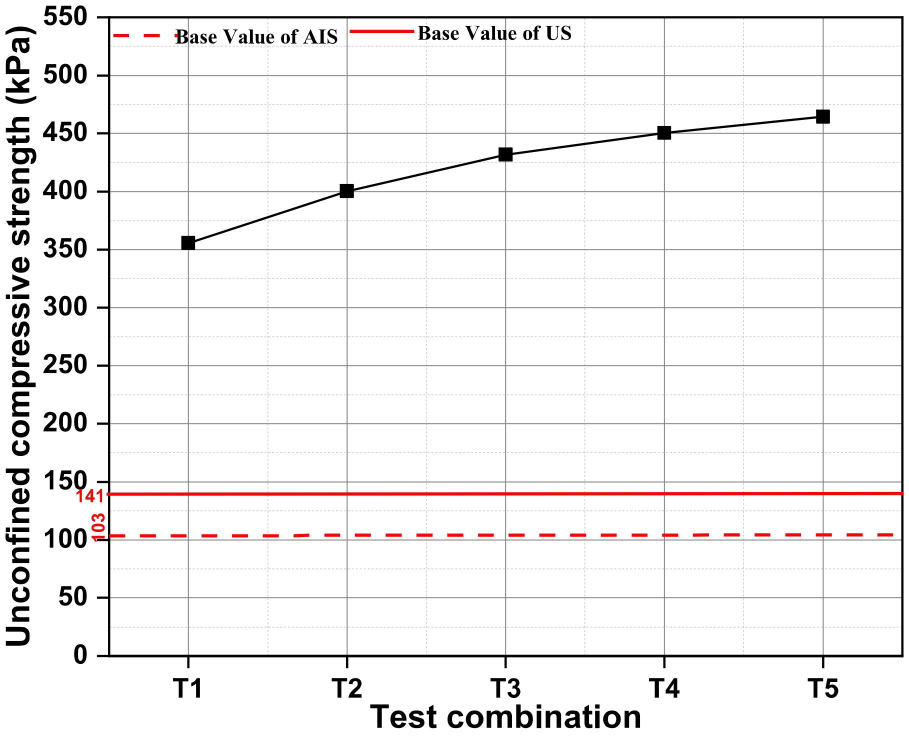

Figure 6.23: Variation of UCS for different test combinations of Sodium carbonate

formation of these cementing agents CSH and CAH produces a heavy cementation effect between the soil particles which resulting the enhancement of the UCS value of alkali interacted soil. Fig. 6.23 also shows that the stiffness of soil increases with higher dosages of sodium silicate solution. The quantity of silicate ions is important to promote the precipitation of the calcium silicate hydrate (CSH) and subsequently crystallising in the void spaces. Apart from these, the alkali nature of soil due to interaction with sodium hydroxide also affects the formation of CSH gel (Jawed and Skalny, 1978). The alkaline nature of soil enhanced and accelerate the formation of CSH gel in the presence of silicate ions which can instantaneously bind the soil particles resulting increase in strength of alkali interacted soil. The interaction of NaOH solution with the soil produces a highly alkaline environment that can be responsible for degradation of the clay mineral lattices leaching the reactive silica and alumina (Ingles and Metcalf, 1972). The reaction between calcium ions and the reactive silica and alumina leads to the formation of CSH, which contributes to the increase in overall strength of alkali interacted soil after EK treatment with calcium chloride and sodium silicate solution (Suganya and Sivapullaiah, 2016).

6.4.3 Variation in Shear Strength Parameters of Alkali Interacted Soil after EK Treatment

The shear strength is an important parameter that governs the behaviour of soil in field under loading conditions. After the completion of the EK test UU triaxial test have been conducted on cylindrical samples under three confining pressures (50kPa, 100kPa, 150kPa) to examine the stabilizing effect of the different chemicals on variation in shear strength parameter of alkali interacted soil. The different confining pressures replicate the field stress condition on the soil. The increase in strength parameter after EK treatment with chemicals was due to the changes in water content and the electrochemical effect caused by the electrokinetic process occurring concurrently. The improvement in cohesion and angle of internal friction value after EK treatment with different chemical stabilizers are depicted in Fig. 6.24, Fig. 6.25, Fig. 6.26, Fig. 6.27, Fig. 6.28 and Fig. 6.29 respectively.

The chemical reactions between stabilizers and soil particles lead to the formation of cementitious materials that bind the particles together, thus increasing the strength of the sample. In fact, the chemical reactions between the stabilizer and soil particles facilitate

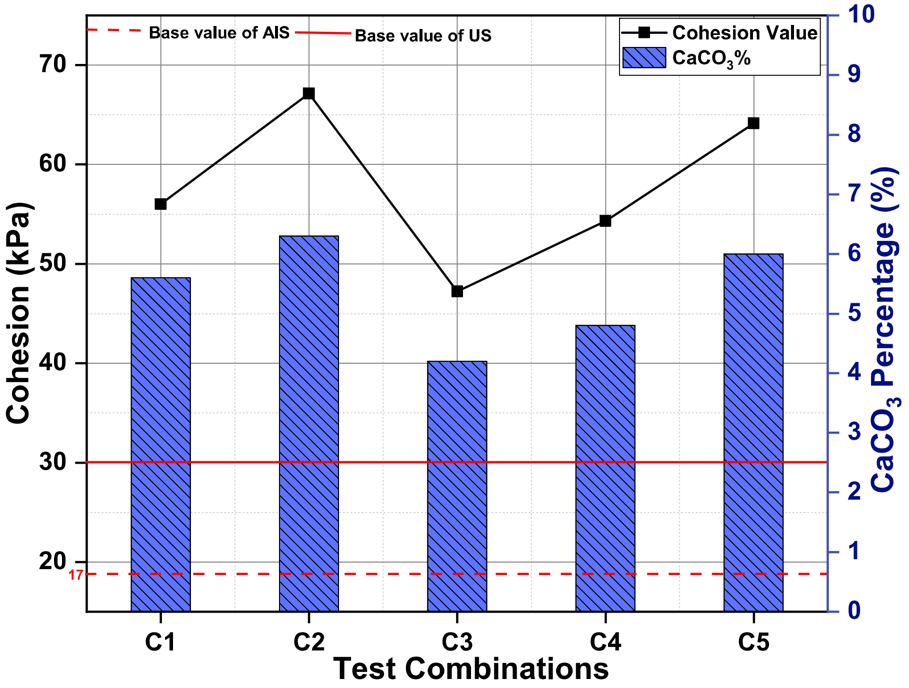

Figure 6.24: Variation in cohesion of treated soil for different test combinations of EK-EICP

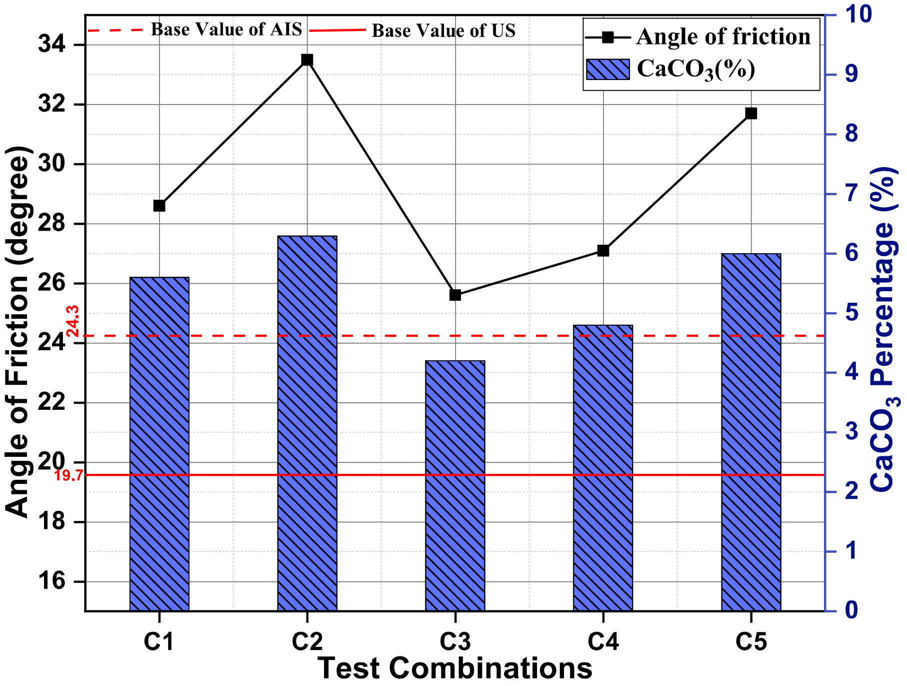

Figure 6.25: Variation in angle of internal friction of treated soil for different test combinations of EK-EICP

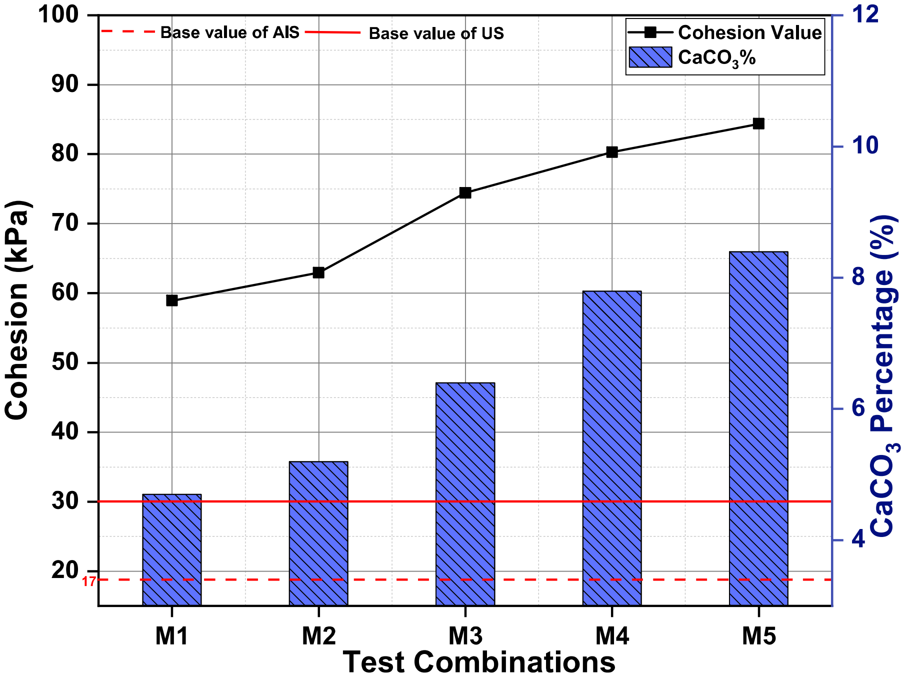

Figure 6.26: Variation in cohesion of treated soil for different test combinations of sodium carbonate

the filling of inter-particle void spaces and thereby increase the bonding between the particles. The cohesion value of alkali interacted soil increases from 17 kPa to 67.12 kPa and 84.37 kPa and the angle of internal friction increases from 24.3^o to 33.50^o and 27.32^o after EK treatment with EICP and calcium chloride-sodium carbonate respectively. The improvement in the shear strength parameter can be anticipated with the amount of calcite precipitation in the voids of soil after EK treatment with EICP and calcium chloride-sodium carbonate. The relation between cohesion and angle of internal friction with $CaCO_3$ percentage is also shown in Fig. 6.24, Fig. 6.25, Fig. 6.26 and Fig. 6.27. The maximum cohesion of 67.12 kPa and 84.37 kPa was obtained from the treated sample containing 6.3% and 8.4% precipitated carbonate. The angle of internal friction value of treated sample increases to 33.50^o and 27.32^o respectively. The increase in cohesion value is possibly due to more $CaCO_3$ precipitations formed lead to particle–particle cementation and provide a significant improvement in soil properties. The increases in friction angle with the calculated percentage of precipitation is mainly due to the double-layer water is like lubrication between particles, and the strong short-term reaction may decrease the thickness of the diffused double layer, which weakens the lubricating effect and results in the increase in friction angle. The calcite precipitation formation form bridges between the soil particle, thereby increasing the strength and stiffness of soil. Similar results have also been reported in different studies which reveals that the precipitation of $CaCO_3$ in the soil promotes the bonds among the soil particle, thus improving the cohesion and angle of internal friction (Almajed et al., 2020; Oliveira et al., 2017; Rohy et al., 2019). The EK treatment of alkali interacted soil with calcium chloride- sodium silicate system shows the similar trend with the rest of the system as seen in Fig. 6.28, and 6.29 . This shows that shear strength parameter values improved significantly with the concentration of sodium silicate. The improvement in shear strength parameter is observed due to migration of calcium ion and silicate ion which leads to reducing the double layer thickness due to cations exchange process which allowed a more densely packing of the particles. The soil strengthening is also attributed to the precipitation of calcium and silicate ions form cementing agent that bind the soil particles together. The reaction between silicate ions with of calcium ions at the other side is important to promote the precipitation of the calcium silicate hydrate (CSH) and subsequently crystallising in the void spaces. The precipitation of insoluble salt has greater influence on the shear strength parameter of the

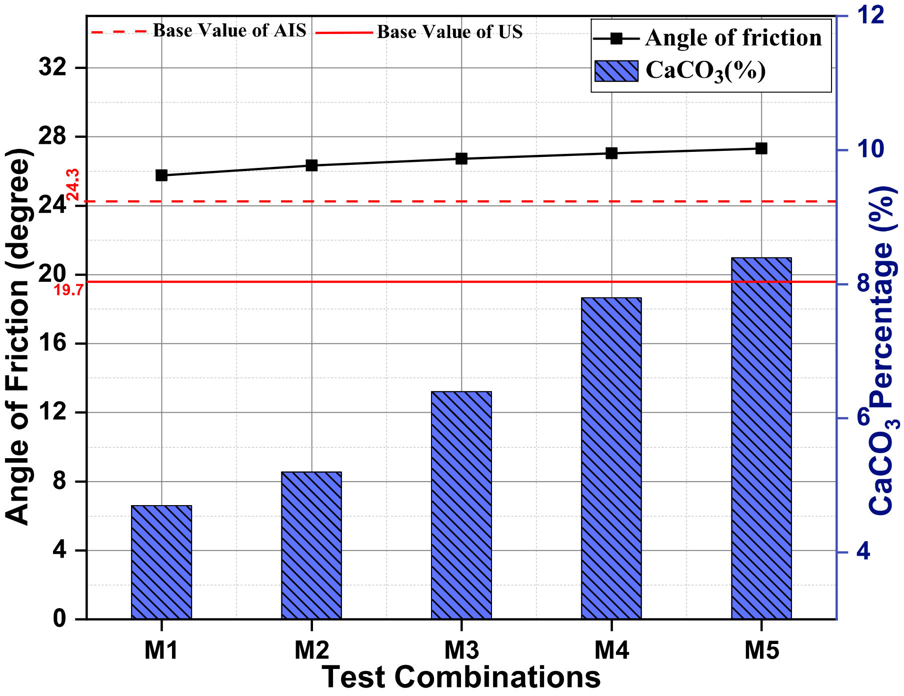

Figure 6.27: Variation in angle of internal friction of treated soil for different test combinations of sodium carbonate

soil under electrokinetic stabilisation treatment.

The increase in strength is also due to reaction between free calcium ions in the system with silica and alumina present in the soil resulting in additional CSH and/or CASH gel. The existence of CSH and CASH gel lead to improvement in shear strength parameter of soil. The strength of alkali interacted soil increases improves with increasing dosage of sodium silicate additive. The development of CSH is abundant and more prominently seen in the case of soil samples treated with 40% sodium silicate solution. The cohesion value increases from 17 kPa to 77.93 kPa after treatment. Whereas the angle of internal friction increases with increases in the concentration of sodium silicate. The angle of internal friction value increases from 24.3^o to 28.13^o after EK treatment. CSH contributes greatly to the cementation of soil particles and hence enhanced the strength characteristics of alkali interacted soil. On further addition of sodium silicate, the cohesion value of alkali interacted soil decreased. This progression of cohesion reduction due to an increase in sodium silicate is similar to the report by (Sina and Bujang, 2011) where it was inferred that due to solubility of Sodium Silicate in water resulting the lack of the intermolecular bonding of Sodium Silicate with alkali interacted soil.

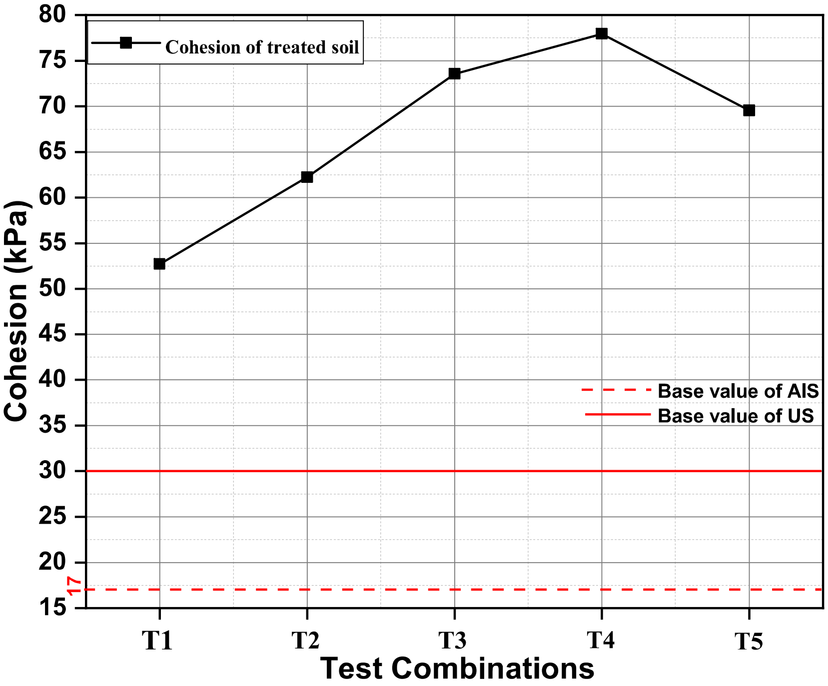

Figure 6.28: Variation in cohesion of treated soil for different test combinations of sodium silicate

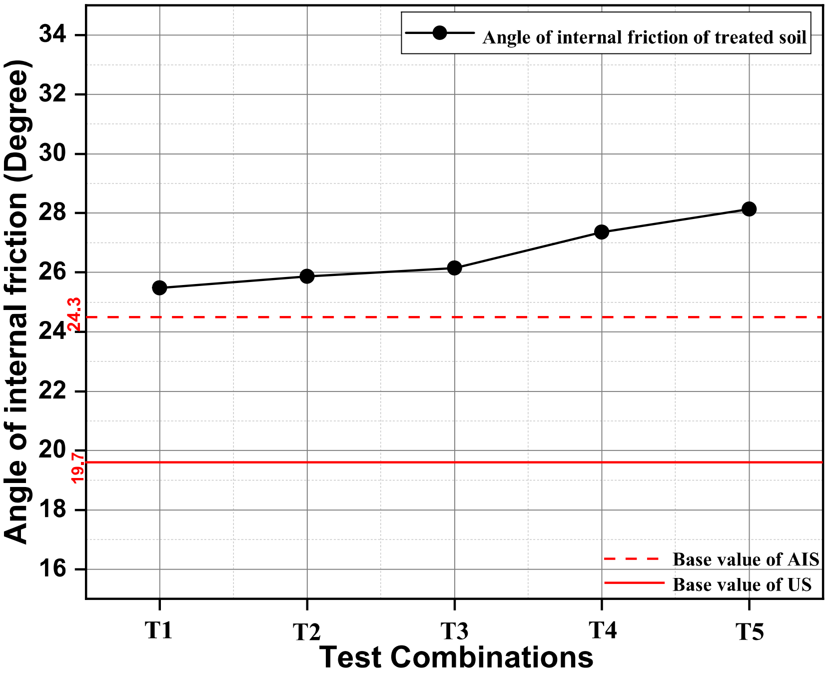

Figure 6.29: Variation in angle of internal friction of treated soil for different test combinations of sodium silicate

6.4.4 Variation in Coefficient of Compression of Alkali Interacted Soil after EK Treatment

The effect of different concentrations of all three chemicals on the compressibility characteristics of alkali interacted soil was determined by conducting a one-dimensional consolidation test (oedometer test). The purpose of the consolidation test is to determine the magnitude and rate of volume decrease that a laterally confined soil specimen undergoes when subjected to different vertical pressures. To conduct the consolidation test, soil samples were extracted from three different locations (near the anode, at the middle and near the cathode) after completion of the EK test. For determining the compression index of treated soil, the soil sample was restrained laterally and drained axially while being subjected to incrementally applied stress loading (25, 50, 100, 200, 400 and 800 kPa) and then unloading (400, 200, 100, 50, 25 and 0 kPa). The apparent difference in compressibility behaviour between the alkali interacted soil and the soil sample treated with different combinations of all three chemicals are shown in Fig.6.30, Fig. 6.31, and Fig. 6.32 respectively. The compression index of the treated samples was less than that

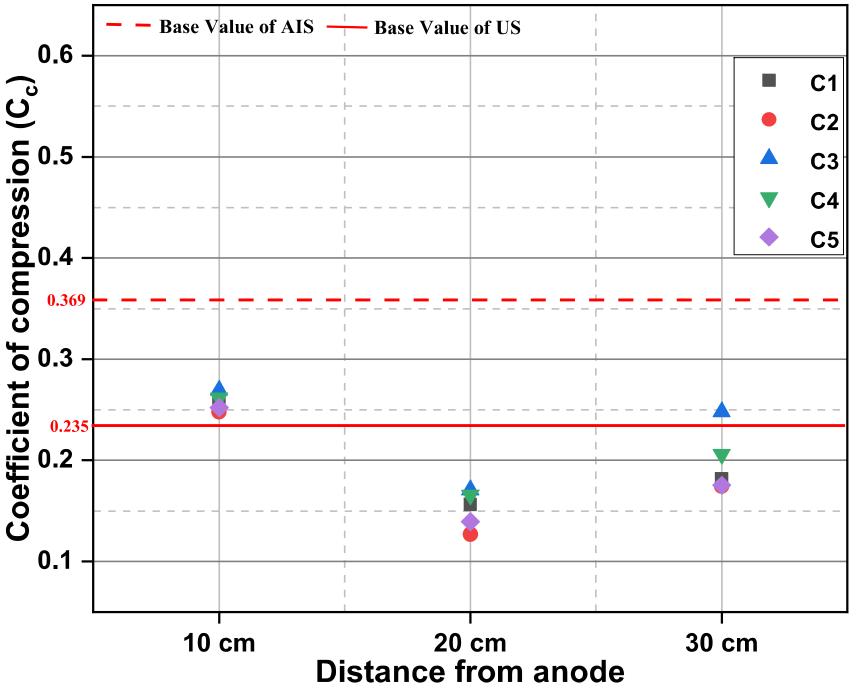

Figure 6.30: Variation in coefficient of compression along the specimen length treated with EICP

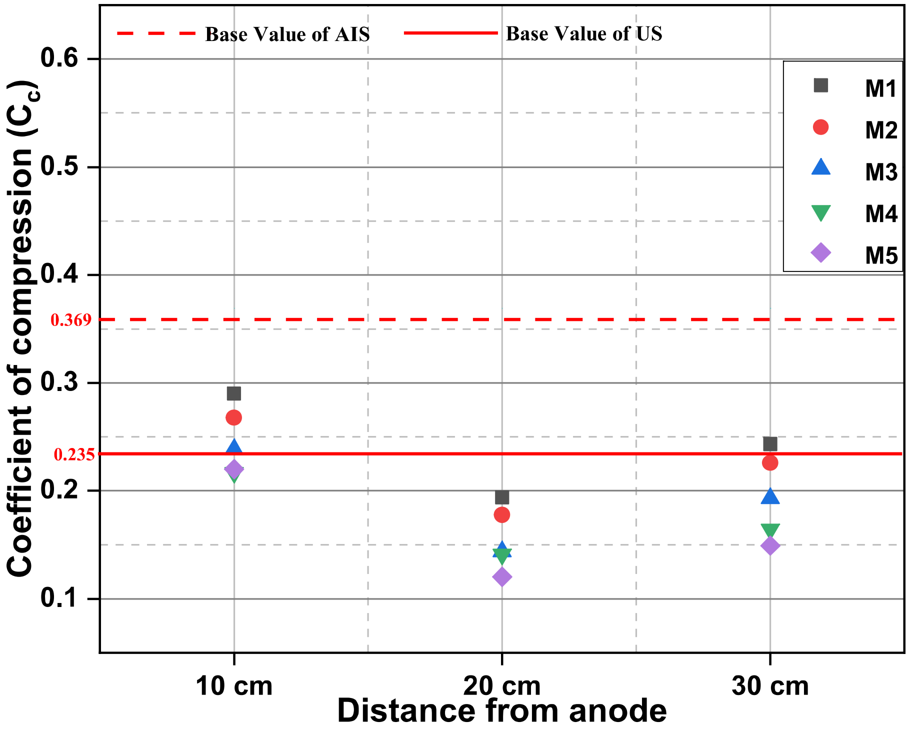

Figure 6.31: Variation of coefficient of compression along the specimen length treated with Sodium Carbonate

for the untreated soil sample. Interestingly, a remarkable change in compression index value can be observed at three different locations along the specimen length. The maximum reduction in compression index value was observed at the middle of soil specimen in all three EK test with chemicals. The minimum value of compression index of treated soil extracted from middle is probably due to the movement of the ionic solution from anode towards the cathode and met with the ionic solution migrate form cathode at the mid-point of the sample. The migration of ions leads to changes of clay structure due to the electrochemical effects, which in turn reduces in compressibility of treated soil. The reduction in compression index value indicates that ingression of chemicals using EK technique improves the compressibility behaviour of the alkali interacted soil. The reduction in compressibility of soil after EK treatment with EICP and calcium chloride-sodium carbonate solution can be attributed to the clogging effect of the calcite precipitation. The clogging of the pore spaces with the calcite particles reduced the accumulation of the fluid into the soil. The calcite particles deposited in the voids of alkali interacted soil generate a cementation effect between soil particles. The cementitious effect of soil particles reduces the compressibility of the treated samples. The difference in compressibility of treated soil and alkali interacted soil can also be correlated to the change in Atterberg's limits. Moreover, several correlations have also been developed in past which show that the change in the plasticity index affects the compression index value (Ibrahim et al., 2012; Muguda and Nagaraj, 2019).

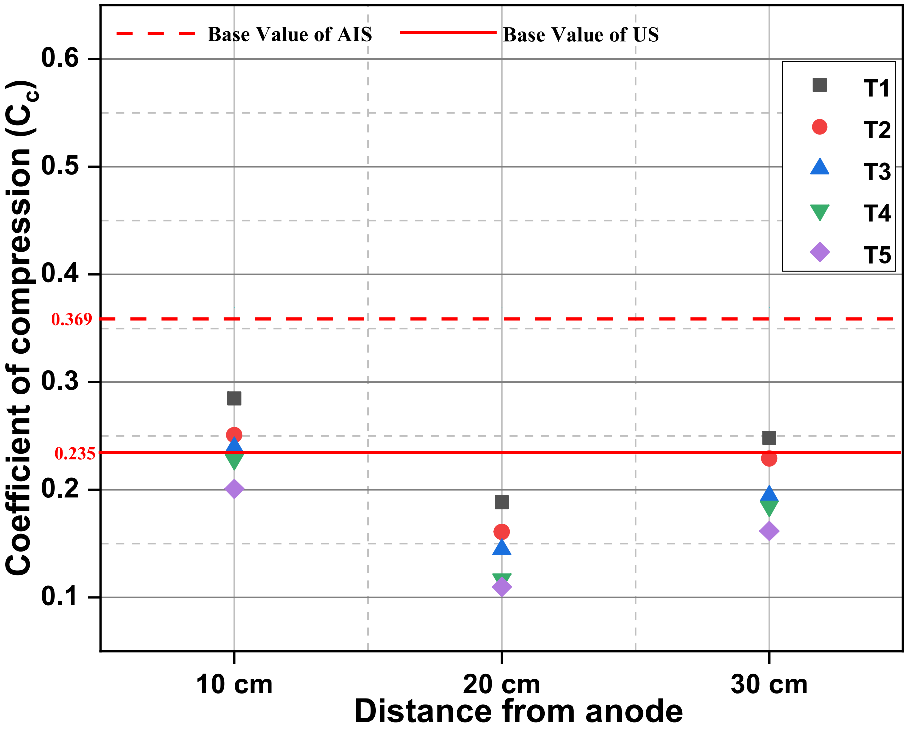

Figure 6.32: Variation of coefficient of compression along the specimen length treated with Sodium Silicate

A similar trend has been observed in the case of EK treatment with different combinations of calcium chloride-sodium silicate as shown in Fig. 6.32. The reduction in compressibility of alkali interacted soil is mainly due to the precipitation of insoluble salt between the soil particles blocking any further movement of water through the soil. After the hydrolysis of calcium chloride and sodium silicate solution Ca^{2+} was transported to the cathode, a large amount of SiO_3^{2-} reacted with Ca^{2+} which causes a pozzolanic reaction between these ions resulting formation of cementing agents including calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH). The formation of these bonds improved the binding of the soil particles, leading to a reduction in compressibility of alkali-interacted soil. It has been also observed that the reduction in compressibility of alkali interacted soil increases with an increase in the concentration of sodium silicate in the soil, as it enhanced the formation of calcium silicate hydrate. It is also well known that the reduction in compressibility is associated with the content and the variety of binding agents because the growth of cementitious products is mainly controlled by the pozzolanic reactions (Cong et al., 2014). The high pH value of soil also enhanced the Pozzolanic reactions between calcium ions and silicate ions to form soluble calcium silicate which polymerizes further to form gels that bind clay or sediment particles together and fill the voids of alkali interacted soil (Brykov et al., 2002; Huter, 1981).

6.4.5 Heaving Analysis on Soil Stabilised with Chemicals

The study of the percentage heaving of soil and soil mixed with all five different combinations of all three-chemical stabilizer under 16M NaOH solution was carried out using a one-dimensional oedometer test. The main intent was to investigate the stabilizing effect of chemical stabilizer on alkali induced heaving. For this, different combinations of three chemicals (as shown in Table 6.1) were batch mixed with the soil. The prepared mix was

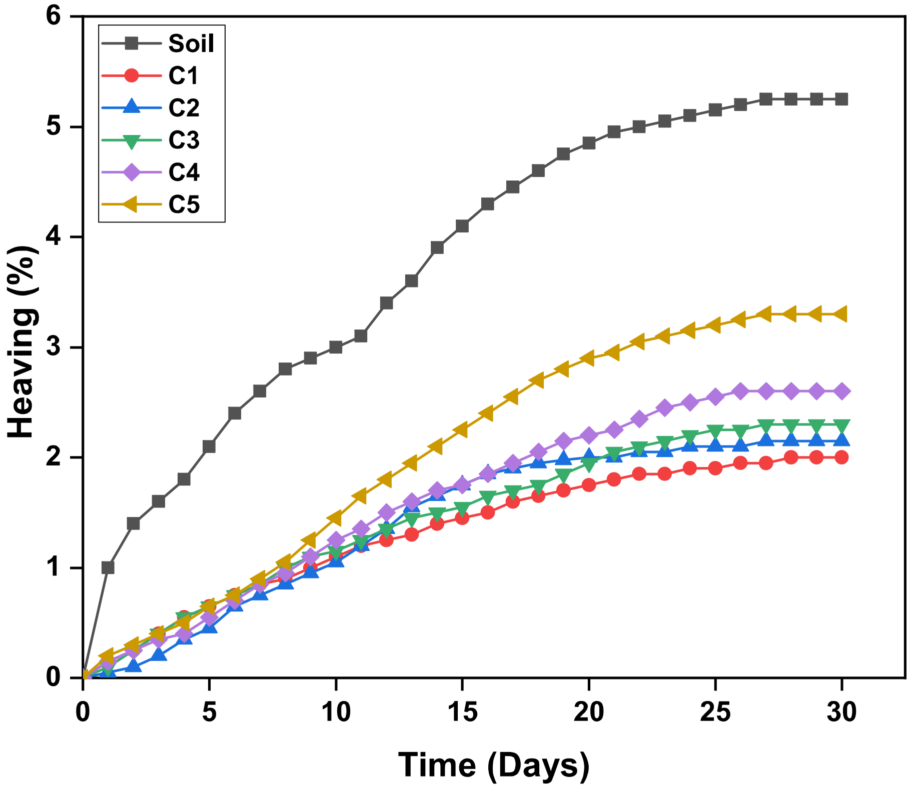

Figure 6.33: Variation in percentage heaving with time in soil and EICP treated soil

then compacted in an oedometer ring and left for 7 days interaction period. After the interaction, the ring was placed in the consolidation apparatus and the whole assembly was kept in the consolidation set up. Further, 16M NaOH solution was inundated from the bottom and the heaving was measured using a dial gauge at the top. The percentage heaving was calculated by dividing the amount of heaving by the total height of the soil sample. The heaving behaviour of all combination on treated soil under 16M NaOH solution are shown in Fig. 6.33, Fig. 6.34 and Fig. 6.35 respectively. The monotonic decrease in percentage heaving of EICP and calcium chloride-sodium carbonated treated soil can be attributed to the calcite precipitation. The produced $CaCO_3$ crystals precipitated in soils reduce the void ratio (or porosity) of the soils which creates a hindrance in the inundation of NaOH solution in to soil voids. As a results, the percentage heaving of soil get reduced. The reduction in the percentage heaving of the EICP and calcium chloride-sodium carbonate treated samples was mainly controlled by the amount of $CaCO_3$ precipitation. The maximum reduction in percentage heaving was observed in case of EICP treated

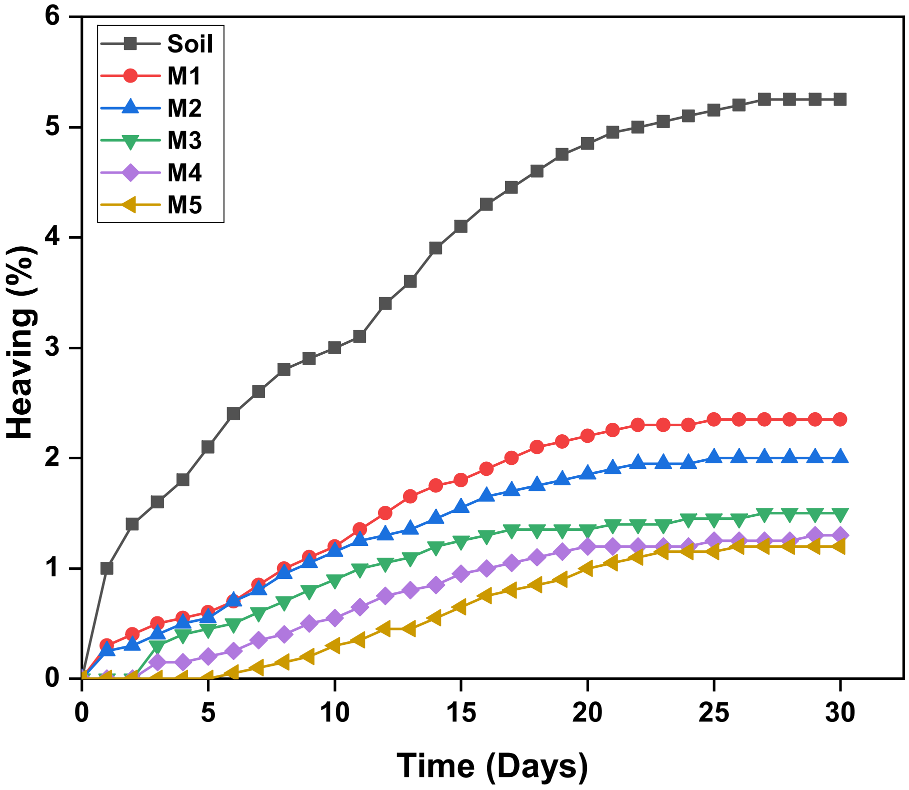

Figure 6.34: Variation in percentage heaving with time in soil and Sodium Carbonate treated soil

soil from 5.55% to 2% and in case of calcium chloride-sodium carbonate from 5.55% to 1.2% for the combination of chemical concentration that yields maximum calcite precipitation. The quantity of $CaCO_3$ in soil governs the rate and amount of flow of NaOH solution in soil leads to variation in percentage heaving in soil. However, in case of soil sample with relatively lower precipitation ratio, NaOH solution leads to the development of new preferential liquid flow path resulting into interaction of NaOH solution with the soil and causing some heaving. Apart from reduction in flow of NaOH solution due to precipitation of $CaCO_3$, the cementitious property of $CaCO_3$ also cause the reduction in percentage heaving. The cementitious nature of the calcite binds the soil particle together which in turns reduces the percentage heaving of soil. From Fig. 6.35 it can be clearly seen that with different concentration of calcium chloride-sodium silicate solution significantly reduces the alkali induced heaving in the soil samples. The reduction in heaving of sodium silicate stabilized soil in the short term is mainly due to the reduced affinity of clay particles for water absorption, and over the long term, to the formation of cementitious

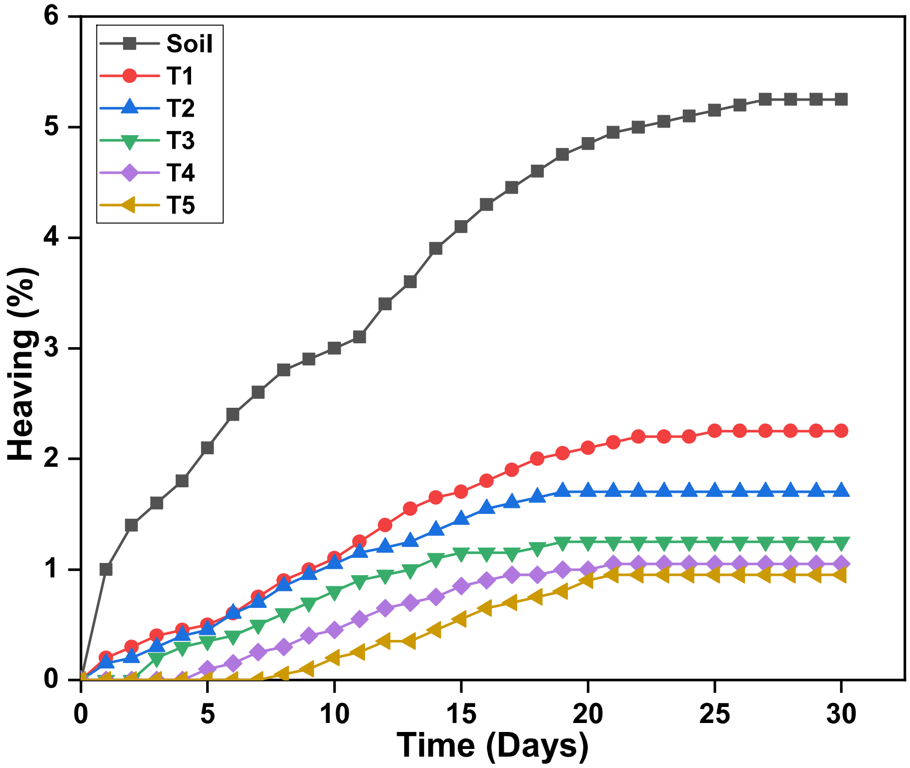

Figure 6.35: Variation in percentage heaving with time in soil and Sodium silicate treated soil

compounds (i.e., CSH, CAH) which bind soil particles and resist heaving behaviour with NaOH solution. The uninteracted soil sample with no sodium silicate content recorded a maximum heaving of 5.55%. The percentage heaving reduced to 0.95% after the soil is mixed with sodium silicate solution. The addition of calcium chloride and sodium silicate solution leads to the significant change in the modified soil micro structure as a result of formation of cementing compounds.

The reaction between sodium silicate and calcium chloride solution generates insoluble calcium silicate which polymerizes further to form a gel that binds soil particles together and fills voids of soil (Yonekura and Kaga, 1992). The crystallising of precipitate of calcium silicate hydrate (CSH) into the voids of soil retards the permeation of NaOH solution into the soil as a result of which the percentage heaving reduces with increases in the concentration of sodium silicate. Apart from these, the ingression of NaOH solution into the soil accelerate the formation of CSH, which can instantaneously bind the soil particles. The sodium hydroxide solution produces an alkali environment which accelerate the hydration of calcium silicates producing more cementitious compounds (Jawed and Skalny, 1978). Hence, the reaction between sodium hydroxide and sodium silicate enhanced and accelerated production of CSH gel.

6.4.6 Effect of EK Treatment with Chemicals on Mineral Composition of Alkali Interacted Soil

The change in geotechnical properties of alkali interacted soil after the EK treatment with different chemicals can be attributed to change in mineralogical compositions and formation of new compound after treatment. In view of this X-ray diffraction (XRD) test has been conducted to investigate the change in mineralogical compositions after EK treatment with chemicals. The X- ray diffractograms of soil, alkali interacted soil and change in mineralogical compositions of alkali interacted soil after stabilized with different chemical are shown in Fig. 6.36, Fig. 6.37 and Fig. 6.38 respectively. XRD pattern of soil inundated with water shows the peak of quartz. However, soil inundated with sodium hydroxide shows new peaks of sodium aluminium silicate hydroxide hydrate (NASH). The formation of this new compound is due to the precipitation of a combination of dissolved silica with sodium hydroxide. The precipitation of these minerals induced heaving in nonexpansive soil. Fig. 6.36 and Fig. 6.37 shows the peak intensities for calcite are observed on addition of EICP and Calcium chloride-sodium carbonate which is the most stable polymorph of calcium carbonate. The presence of calcite minerals between soil particles reduced its cavities and improved soil strength and stiffness. However, a clear reduction in the intensities of peak of quartz has been observed after the EK treatment with EICP and calcium chloride-sodium carbonate.

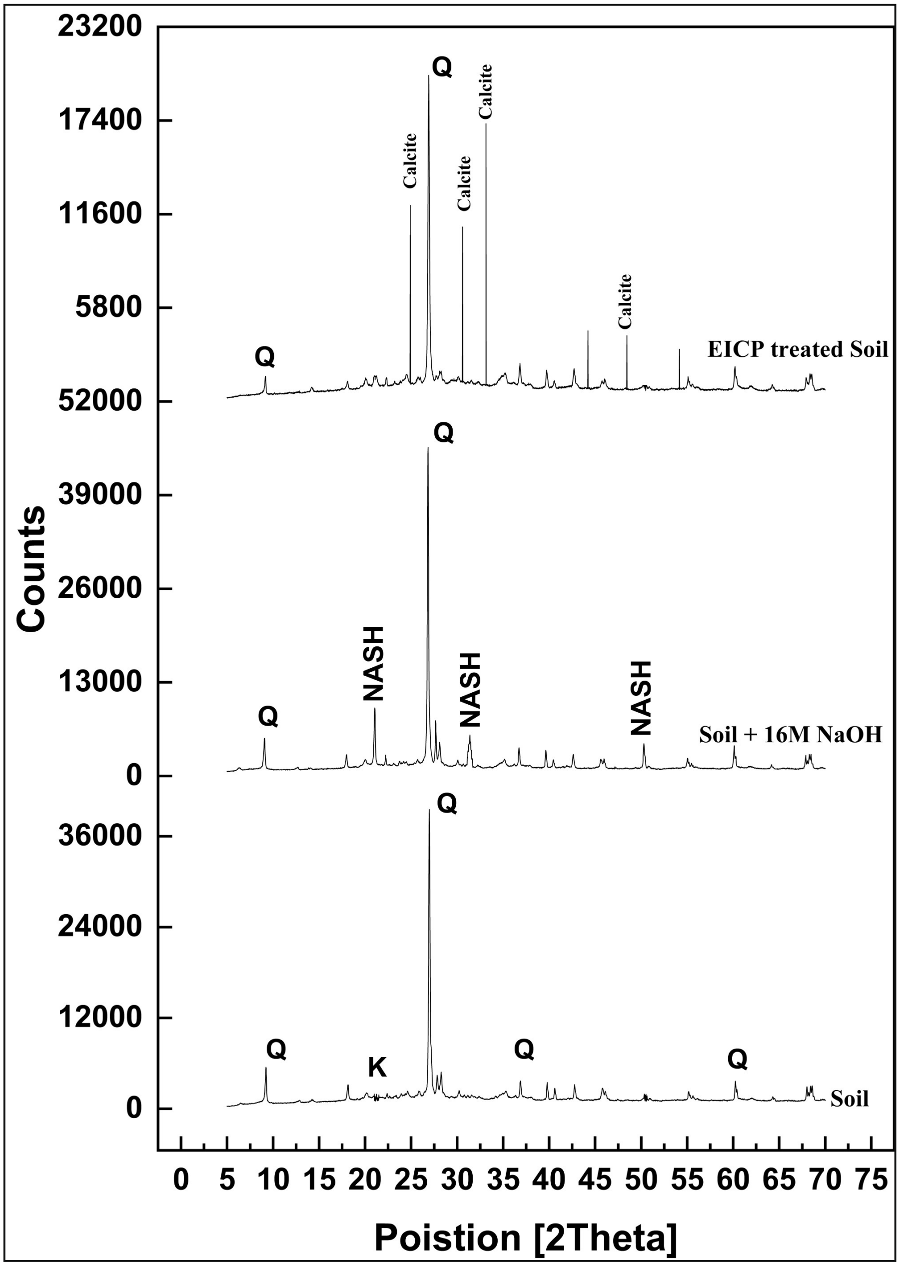

Figure 6.36: XRD pattern of uninteracted soil, alkali interacted soil and EICP treated soil

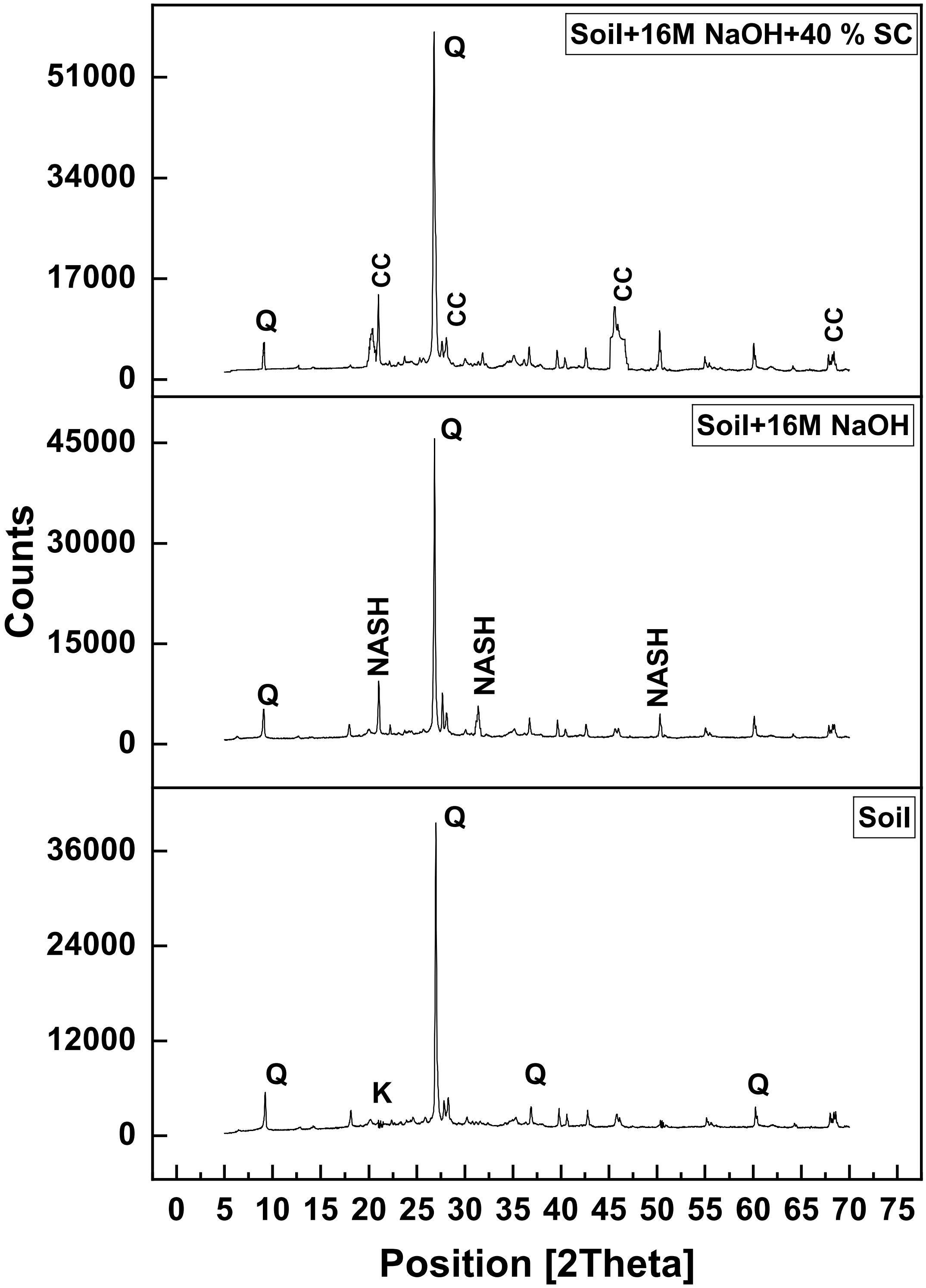

Figure 6.37: XRD pattern of uninteracted soil, alkali interacted soil and sodium carbonate treated soil

X-ray diffraction (XRD) pattern of change in crystal structure of alkali interacted

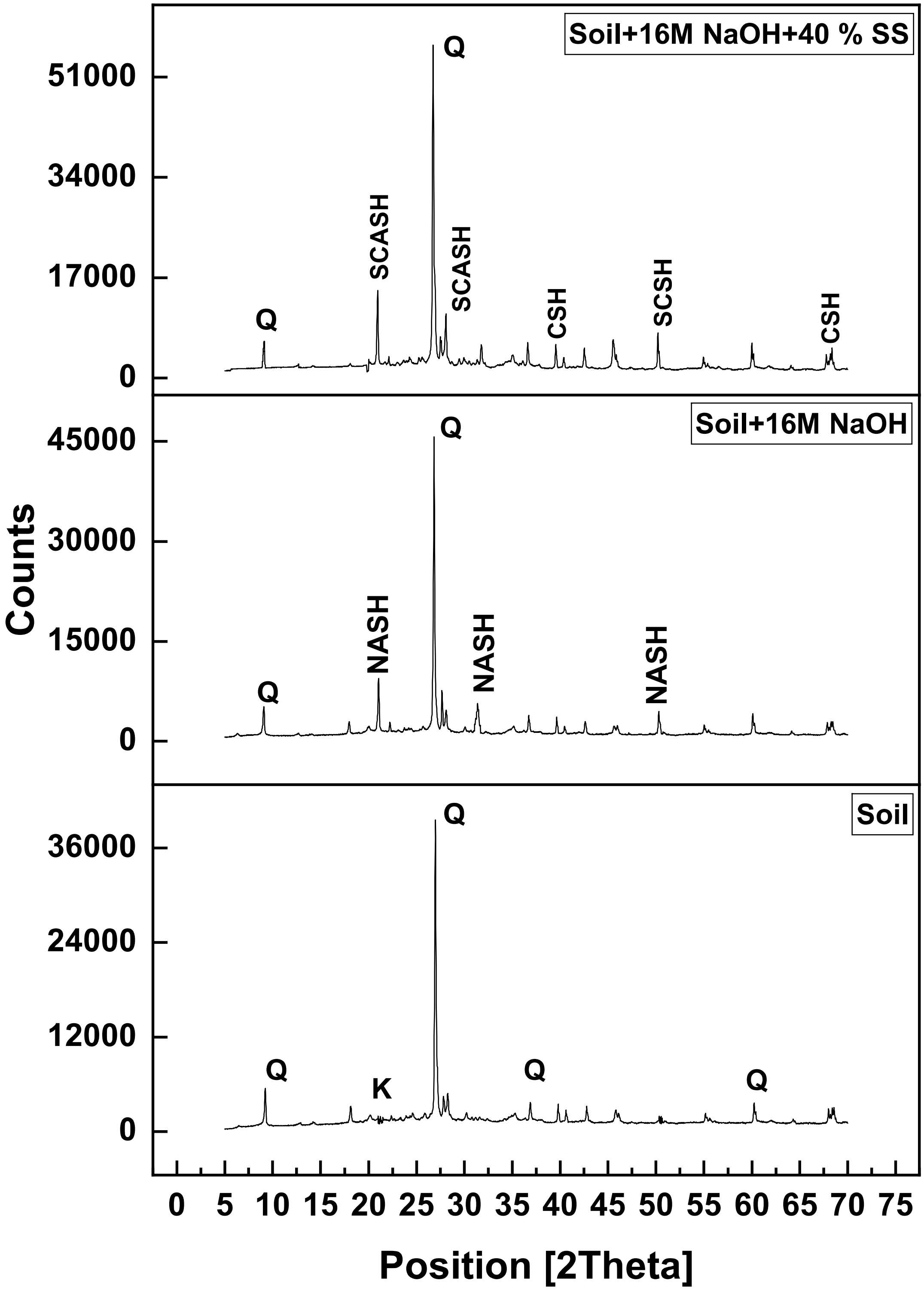

Figure 6.38: XRD pattern of uninteracted soil, alkali interacted soil and sodium silicate treated soil

soil treated with sodium silicate are shown in Fig. 6.38. Comparing the XRD pattern of alkali interacted soil and alkali interacted soil treated with sodium silicate shows changes in the mineralcomposition after treatment. The figure reveals the formation of peaks of cementitious compound like calcium silicate hydrate (CSH). In addition to the peak corresponding to CSH some additional peaks of sodium calcium silicate hydrate (SCSH) and sodium calcium aluminium silicate hydrate (SCASH) was identified in the alkali interacted soil with higher percentage of Sodium silicate solution. The formation of these cementitious compound improved binding of the soil particles which leads to substantial increase in strength of alkali interacted soil after treated with sodium silicate.

6.4.7 Microstructural Study of Alkali Interacted Soil after EK Treatment with Different Chemicals

The change in fabric of alkali interacted soil after EK treatment with different chemicals also governs the change in geotechnical properties of soil. To examine the microstructural transformation of soil interacted with NaOH solution and alkali interacted soil treated with different chemicals, a series of scanning electron microscopy (SEM) test has been conducted on the treated soil. The SEM image of soil, alkali interacted soil and alkali interacted soil treated with different chemicals are shown in Fig.6.39. The SEM image of untreated soil with water as shown in chapter 4 indicates flaky and stable structure. It can be observed that the soil particle changes into dispersive and hollow structure due to weathering effect after alkali interaction as shown in chapter 4. The looseness and hollowness in structure of soil after alkali interaction are evident of formation of new compound which alters the geotechnical properties of soil. In contrast, the SEM image of chemically treated alkali interacted soil shows modification in soil texture. SEM image of alkali interacted soil treated with EICP and sodium carbonate are shown in Fig. 6.39a, Fig. 6.39b, Fig. 6.39c, and Fig. 6.39d. These SEM images clearly shows deposition of $CaCO_3$ on the surface and voids of alkali interacted soil after treatment. The amount of calcite precipitation in the voids of soil particle has a significant impact on the strength of treated soil. The presence of rhombohedral $CaCO_3$ between the soil voids causes aggregation of soil minerals after EICP treatment. The calcite precipitation increases particle -particle contact which results a dense microstructure. The densification and binding of particle

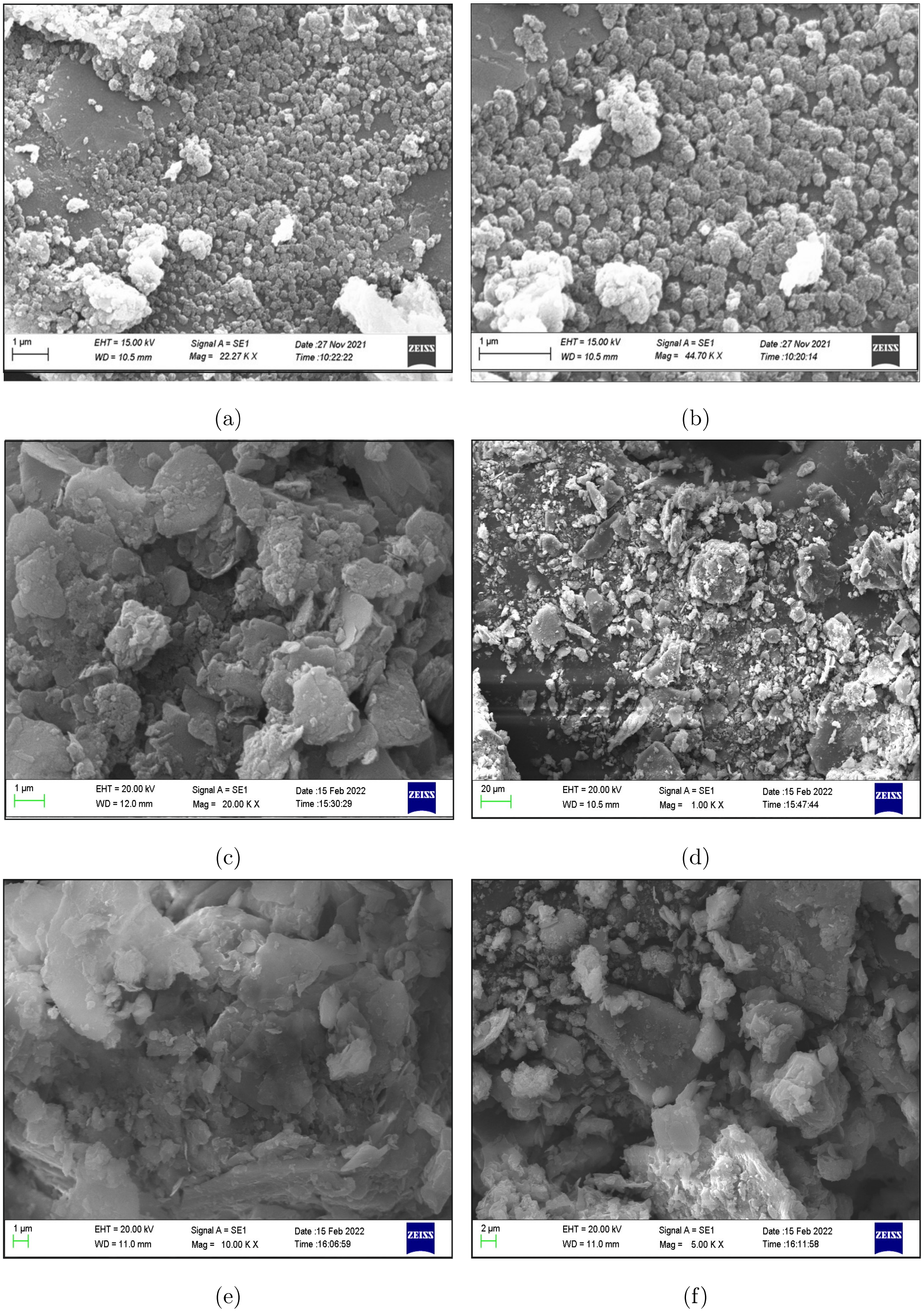

Figure 6.39: SEM image of alkali interacted soil, (a), (b) treated with EICP, (c), (d) treated with sodium carbonate, and (e), (f) treated with sodium silicate

due to calcite precipitation increases the strength and stiffness. However, the peaks of CSH, CASH and SCASH has been observed in the XRD analysis. Further for the visual examination of formation of these compounds, SEM analysis has been conducted on the alkali interacted soil treated with sodium silicate. The transformation of discrete particles into a close bonded structure and filling effect of CSH gel in the voids of the particles has been observed in SEM images as shown in Fig. 6.39e and Fig. 6.39f. Formation of these new compound confirms the occurrence of pozzolanic reactions after the treatment of soil with sodium silicate. The development of CSH, CASH and SCASH gel accompanied by the pozzolanic reaction linked the soil particle with each other, thus contributing to a stronger filling effect of sodium silicate. The cementitious effect of these gel enhanced the bonding between the soil particle and developed a more compact microstructure due to agglomeration of soil particles. These change in soil structure after treatment with sodium silicate contribute to increase in strength of the stabilized material.

6.5 Summary

In this chapter, the efficiency of EK technique with three different chemicals for the treatment of alkali interacted soil has been analysed. Five different combinations of three different chemicals as shown in table 6.1 has been used as anolyte and catholyte solution in EK test. The change in physicochemical change in soil after EK test has been analysed and discussed in detail. The change in voltage, EO flow, change in pH of electrolytic solution has also been closely monitored during the test. After the EK treatment different geotechnical test has been conducted on stabilized soil. It has been observed that the unconfined compressive strength and shear strength parameter has been improved after EK treatment with different chemicals. Further, the effect of these chemicals on alkali induced behaviour of soil also examined. The heaving results shows that these chemicals effectively control the heaving of soil caused by the inundation of 16M NaOH solution. XRD and SEM test has been also conducted on the treated soil to examine the change in mineralogical compositions and microstructure after treatment. The change in peak of untreated soil and peak of new compound has been observed after treatment with chemicals. The change in microstructure of soil also confirms the reaction between the soil and chemical which improved the geotechnical properties of alkali interacted soil.

The successful demonstration of bench scale electrokinetic model study in enhancing the strength of alkali-interacted soil reduction in alkali-induced heaving in soil using these chemicals additives makes it worth utilising these chemicals as a stabiliser on alkali-contaminated sites.