Laboratory study of a kaolinitic soil and sodium hydroxide interaction mechanisms and resulting swelling stresses

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ABSTRACT

The study of prolonged exposure of natural soils to alkaline solutions has been conducted to analyze and control the local soil swelling behavior observed in industrial sites. The occurrence of foundation heave in a Brazilian industrial site prompted a laboratory investigation aimed at understanding the mechanism of interaction between soil and alkali that induced swelling behavior. The study was extended to determine the corresponding swelling stress to be used in reinforcement design, and to investigate the soil response to chemical stabilization with 5% ferric chloride solution. Previously, a comprehensive mineralogical and physical-chemical investigation was accomplished using industrial caustic liquor and four natural soils from local stratigraphy. All soils are mainly constituted by kaolinite and quartz, and one also presents some montmorillonite. Both kaolinite and montmorillonite were degraded by NaOH liquor, and newly formed minerals were identified. The expansion is related to the mechanism of the silica-NaOH reaction. In the study reported here, the local soil with the highest volume change in sedimentation tests with NaOH solutions was subjected to quasi-constant volume oedometer tests with water and with 4N NaOH solution to determine the swelling stresses resulting from the reactions until stabilization. The alternative of chemical treatment with 5% ferric chloride solution was investigated through the same testing procedure. X-Ray diffraction analyses were applied to evaluate the changes in mineralogy. The swelling vertical stress reached 40 kPa under confined conditions, and stabilization took up to 40 days. The chemical treatment was effective in controlling ongoing reactions, but not effective in prevention.

Keywords: Swelling; silica-NaOH reaction; swelling stresses; ferric chloride treatment.

1. Introduction

Prolonged exposure of natural soils to alkaline solutions has been the focus of a variety of studies aiming at analyzing and controlling local soil swelling behaviour that may occur in industrial sites (e.g., Rao and Rao 1994, Sivapullaiah and Manju 2006a, Paulose et al 2014). The main assumption consists of chemical and physical-chemical reactions that can occur between clay minerals and alkaline solutions that, under certain environmental conditions, end up forming new mineral compounds with strong expansive potential (Sokolovich 1995, Bauer and Berger 1998, Sivapullaiah and Manju 2005, Sivapullaiah and Manju 2006b, Chavali et al 2017, Reddy et al 2017, Alastair et al 2018).

In the present work, the occurrence of foundation heave in a Brazilian industrial site led to a laboratory study, under confidentiality agreement about the industry and site details, to understand the mechanism of interaction between soil and alkali that induced soil swelling behaviour. Soil samples of four local natural strata were tested with the industrial site caustic liquor in a comprehensive mineralogical and physical-chemical investigation. At natural condition, kaolinite and quartz dominates all soils mineralogy, and one stratum also presents some montmorillonite. Only the results from that investigation that are of direct scientific relevance for the study presented herein are briefly described.

The study comprises two phases. First, suspensions were prepared by mixing the soils with NaOH solutions at different concentrations. Then, the soil stratum that presented the highest volume change at the end of the sedimentation tests was selected for a complementary laboratory study. At the second phase, this soil was compacted at a defined density and exposed to NaOH solution under quasi-constant volume in oedometer tests to determine the swelling stresses until stabilization, and to X-Ray diffraction method to analyse the corresponding chemical and mineralogical effects.

Besides, a chemical treatment alternative was also applied. Rao and Rao (1994) have proposed a treatment consisting of introducing a solution of 5% ferric chloride into the impacted soil in the field. Undisturbed soil samples were submitted to laboratory tests and the results showed that in the soil treated with ferric chloride the reactions were neutralized and therefore the resulting expansion. However, the study reported by Sivapullaiah and Manju (2005) showed that soil treated with 5% $FeCl_3$ continued swelling if there was continuous exposure to alkaline solutions.

This chemical treatment was applied to the soil of the present study after contamination and expansion in the laboratory, and a similar behaviour was observed. The soil treated with 5% $FeCl_3$ stabilized at first, but later on presented expansion when submitted to new contamination.

2. Materials and Methods

2.1. Soils

Four major soil strata were identified at the site, all residual soils with low to moderate degree of weathering. For simplicity, they were identified as Type 1, Type 2, Type 3 and Type 4, based mainly on texture and visual features, and considering geological similarities. Soil Type 1 occurred close to the ground surface throughout the site and all the soil samples available were field contaminated. The other three types of soil presented both contaminated and non-contaminated samples. Only the uncontaminated samples could be used for the chemical reactions and swelling behaviour evaluation. Therefore, soil Type 1 could not be used in this testing programme.

According to the previous comprehensive investigation, soil Type 2 could be described as a yellowish sandy silt or silty sand (NP – non plastic), soil Type 3 as a grey silty sand (PI 14%), and soil Type 4 a clayey silt light brown and sometimes pink and brown (PI 12%– 36% depending on the content of fines). Soil Types 2 and 3 are composed mainly by kaolinite and quartz, with feldspars and micas. Soil Type 4 also presented some montmorillonite at natural condition.

The Type 2 non-contaminated soil samples available in the laboratory were not sufficient to accomplish the complete testing programme, therefore, the soil strata selected for the complementary study were soils Type 3 and Type 4. Field pH measured on non-contaminated samples collected along the Type 3 strata varied between 4.07 and 6.14, and along Type 4 strata between 4.08 and 6.19. The corresponding contaminated samples field pH varied between 11.62 and 13.63 for Type 3 soil, and between 7.26 and 10.55 for Type 4 soil.

2.2. Methods

The soil samples used in the laboratory tests were collected from disturbed SPT samplers during the site investigation. The condition of previous contamination was established by the natural pH in water of each sample measured just after collection and presented in the boreholes' logs. The local soils are naturally acidic (pH < 6.5), therefore all samples with pH > 8.0 (many attaining pH ≥ 10) were classified as contaminated in situ, and intermediate values were indicative of transition zones.

2.2.1. Soil physical characterization

The determination of the grain size distribution of the samples followed NBR-7181 Brazilian standard. The determination of the specific gravity G_s followed NBR-6508 and the Atterberg Limits were determined in accordance with NBR-6459 for liquid limit (ω_L) and NBR-7180 for plastic limit (ω_P). The plasticity index (PI) is given by (ω_{L} - ω_{P}).

2.2.2. Chemical analyses and test solutions

a) pH determination

Measurement of soil pH is made in distilled water using a digital pH meter (edge® Multiparameter pH Meter). The pH meter is benchmarked using standard buffer solutions.

b) NaOH solutions

The NaOH solutions were prepared at three different concentrations: 0.1N, 1N and 4N. The basic procedure is the same, varying the amount of NaOH pellets, i.e., 4g to 0.1N, 40g to 1N and 160g to 4N respectively in 1000mL of distilled water. The materials needed to prepare the solutions were: a dry-cleaned 1000mL volumetric flask and NaOH pellets.

c) 5% Ferric Chloride solution

5% of ferric chloride solution was prepared by adding 40g of $FeCl_3$ in 1000mL of distilled water. Material required: $FeCl_3$, glass rod, glass beaker and distilled water.

2.2.3. Mineralogical analyses

The X-Ray Diffraction (XRD) analyses were performed in a Bruker-AXS D8 Advance Eco equipment, Cu Kα radiation (40 kV/25 mA), with a step of 0.01° 2theta, counting time 92 seconds per step with LynxEye XE state-of-the-art silicon drift position-sensitive linear detector (with energy discrimination) collected from 5 to 80° 2theta. The qualitative interpretation of the spectrum was performed by comparison with patterns contained in the relational database PDF 4+ (ICDD, 2014) in Bruker Diffrac.EVA software.

2.2.4. Free swelling tests

Four uncontaminated samples were selected for the free swelling tests with NaOH solutions at different concentrations (0.1N, 1N and 4N). In these tests, 40g of air-dry soil were weighed from the most clayey samples (SR02 and SM07) and 60g of air-dry soil from the fine sand samples (SM05 and SM06), after sieving them through the ASTM#10 sieve (2.0mm). Each sample was placed inside a 200mL glass beaker, filled with 100mL of the selected NaOH test solution, and allowed to rest for 45 days. The suspensions were stirred weekly to ensure that all soil particles were in contact with the solution. The heights of the mixtures were periodically measured, and their apparent volumes calculated. After 45 days, the expansion stabilized, and the sample that exhibited the highest apparent volume change was selected for the oedometer test series.

2.2.5. Oedometer swelling stress tests

Reddy et al (2017) performed one-dimensional free swelling tests using conventional oedometer apparatus with special cells fabricated with materials resistant to both acid and alkali. The soil samples were inundated with the test solution as the pore fluid and allowed to swell under free loading condition. No swelling stress was measured.

The ASTM D-4546-14 standard describes three procedures using the conventional oedometer apparatus. The soil after-wetting strains (swell or collapse) at different initial vertical stresses are measured. After completion of this stage, in case of swelling occurrence, vertical compressive loads can be applied in steps and the final vertical load able to compensate the former swelling strain is used to calculate the corresponding vertical swelling stress.

The Indian Standard IS 2720 (Part 41) (2016) describes two swelling stress test procedures to be performed after soil wetting with water, both using an oedometer cell. One procedure is similar to Method C of ASTM D-4546, and the swelling stress is determined as the stress that induces a vertical compression strain that compensates for the previous swelling strain. The second procedure is called a "Constant Volume Method" and the loading applied is controlled in order to keep the soil sample at zero net strain.

In the present study, the swelling stress test utilized the constant volume method in a system adapted from the conventional oedometer apparatus. Two main adaptations were accomplished: (1) all the experimental cells and reservoirs that would be in contact with the caustic solutions were fabricated with Teflon® material instead of steel; (2) an automated loading control system was installed in order to maintain net zero strain throughout the time of the test, combined with a data acquisition system. Fig. 1 shows a schematic representation of the experimental setup specially designed and manufactured in the university laboratory.

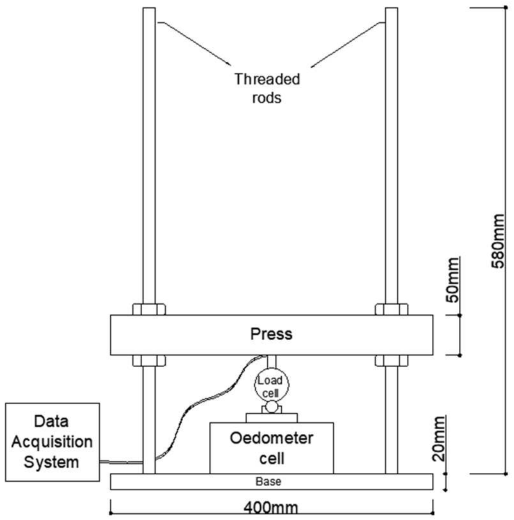

Figure 1. Scheme of the experimental setup of the constant volume swelling stress test in the adapted oedometer apparatus (Laboratory of Geotechnics, COPPE-UFRJ, 2021).

The main structure is composed of a steel press with two vertical threaded rods, where it is possible to set the press for different heights. A load cell is attached to the press and the load measured values are registered by the data acquisition system. The oedometer cell containing the soil specimen is positioned on the steel base, and the press height is adjusted in order to make contact between the load cell and the oedometer top cap. A level ruler is used to ensure the press remains horizontal during the test and the vertical displacement is prevented by tightening the locknuts on the vertical rods. This ensures that the initial load applied to the top cap of the oedometer cell is zero, and the data acquisition system is calibrated. After that, the specimen is completely inundated with the solution to be tested and the data acquisition system starts measuring the loads. The loads are the system response to keep the specimen at zero displacement or constant volume condition.

3. Results and Discussion

3.1. Soil characterization

Table 1 summarizes the physical properties and pH of the soil samples used in the free swelling tests.

Two samples (SR02 and SM07) had a very high content of fines, mostly clay (≥ 60%), and two samples (SM05 and SM06) were mainly constituted of fine sand (50-64%) with fines. All soils strata are heterogeneous, as usual in residual soil profiles.

3.2. Free swelling tests

The sedimentation columns resting on the bench are shown in Fig. 2, and the results are summarized in Table 2.

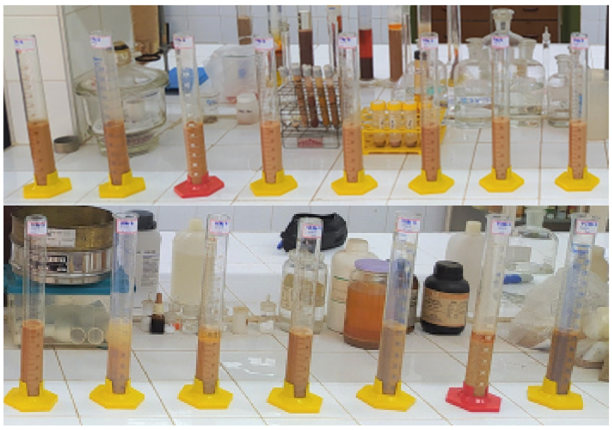

Figure 2. General view of the sedimentation columns on the bench.

Table 1. Soil samples characterization

Borehole	Depth (m)	Soil Type	pH	Gs	% Clay	% Silt	% Sand	Plastic Limit (ω_P)	Liquid Limit (ω_L)	Plasticity Index (PI)	Description	USCS classification
SM05	21.0-21.6	3	6.25	2.622	26	22	52	12.1	26.3	14	Clayey fine sand, yellowish grey	SC
SM07	17.1-18.0	3	8.28	2.633	61	21	18	9.9	21.4	11.5	Silty clay, pink, brown, grey	CL
SR02	14.45-15.0	4	5.37	2.513	63	21	16	28.6	68.6	40	Silty clay, reddish brown	CH
SM06	17.1-18.5	4	6.05	2.620	18	16	67	10.3	20.8	10.5	Silty fine sand, pink and brown	SM

Table 2. Free swelling tests results

Sample	Soil Type	Volume change (%)		
		0.1N	1N	4N
SM05	3	19	21	25
SM07	3	15	2	15
SR02	4	6	7	21
SM06	4	20	7	18

The sample SM05 presented the highest volume at the end of all concentrations of NaOH solutions tests. The sample from borehole SM07 was also Type 3 soil, with greater clay content, but the initial pH was higher than typical natural values (pH = 8.28 > 6.5). Perhaps partial reactions had already occurred in the field caused by contamination. The Type 4 soil samples also presented volume increase, but at a lower magnitude, particularly at the intermediate 1N NaOH solution tests. Therefore, sample SM05 of Type 3 soil was selected for the oedometer swelling stress tests.

3.3. Soil-NaOH interaction mechanisms

The central mechanism of interaction is the chemical reaction alkali-(silica/silicates), that was first explained in Hansen (1944) for concrete and in an extensive literature since then. In the soil medium, this reaction is combined with other mechanisms that depend on the minerals present in the original soil composition and on local environment characteristics such as pH, water content, temperature and porewater chemistry.

For the alkali-silica reaction mechanism to occur, three factors are necessary: I) a source of silica (clay minerals); II) moisture and III) presence of alkali (NaOH for example).

In an alkaline medium, a chemical reaction occurs involving the decomposition of clay minerals such as kaolinite with the formation of a sodium aluminosilicate complex according to the equation (Sokolovich, 1995):

$$Al_2O_3.2SiO_2.2H_2O + 2NaOH \rightarrow Na_2O.Al_2O_3.2SiO_2nH_2O$$

This mechanism of interaction leads to an increase in soil volume, triggering expansive behavior in an alkaline medium. The success of the soil-NaOH interaction also depends on the specific surface available of clay minerals and the accessibility of alkaline solutions to these minerals (Sokolovich, 1995; Rao and Rao, 1994).

The increase in pH is also an important factor on the Diffuse Double Layer and, therefore, for the manifestation of the dispersion of negatively charged clay particles due to the absorption of OH^- ions, which acts on the arrangement of the particles, under the face-to-face association (Mitchell, 1993).

3.4. Oedometer swelling stress tests

Two specimens of SM05 sample were molded for the first set of tests. The specimens were statically compacted in the oedometer cells (7.15cm diameter and 3cm height), to achieve 20% water content and 1.96g/cm³ total density, based on values presented in a technical report for some undisturbed samples collected nearby.

Tables 3 and 4 present the physical parameters of the specimens tested in distilled water (cell 1) and in NaOH 4N solution (cell 2).

Table 3. Physical parameters (cell 1)

Dry unit weight (kN/m³)	15.69
Total unit weight (kN/m³)	19.20
Void ratio (e_o)	0.640
Porosity (n)	0.390
Gravimetric water content (%) (w_o)	22.38

Table 4. Physical parameters (cell 2)

Dry unit weight (kN/m³)	15.57
Total unit weight (kN/m³)	19.16
Void ratio (e_o)	0.652
Porosity (n)	0.395
Gravimetric water content (%) (w_o)	23.05

The results are presented in Fig. 3. The soil slightly expanded with water just after inundated, reaching 5kPa of vertical swelling stress, but soon returned to the initial condition.

In the case of the specimen inundated with 4N NaOH solution (cell 2), a maximum vertical swelling stress of 40kPa was reached in around 40 days after inundation, indicating stabilization of the reactions.

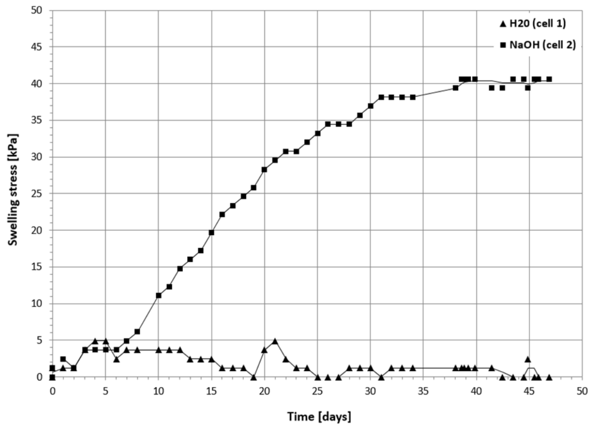

Figure 3. Evolution of vertical swelling stress with water and with 4N NaOH solution over time.

3.5. Soil mineralogic characteristics

The mineralogical analysis was carried out aiming at identifying new mineral formations in the samples, which could cause swelling behaviour of the soil. After the oedometer tests, the specimens were air-dried and sieved through ASTM#200 (0.075mm) sieve, with the following conditions: 1) natural soil; 2) soil inundated with distilled water and 3) soil inundated with NaOH (4N) solution.

The results of XRD tests on specimens at natural condition, treated with water and NaOH are presented in Fig. 4 to Fig. 6.

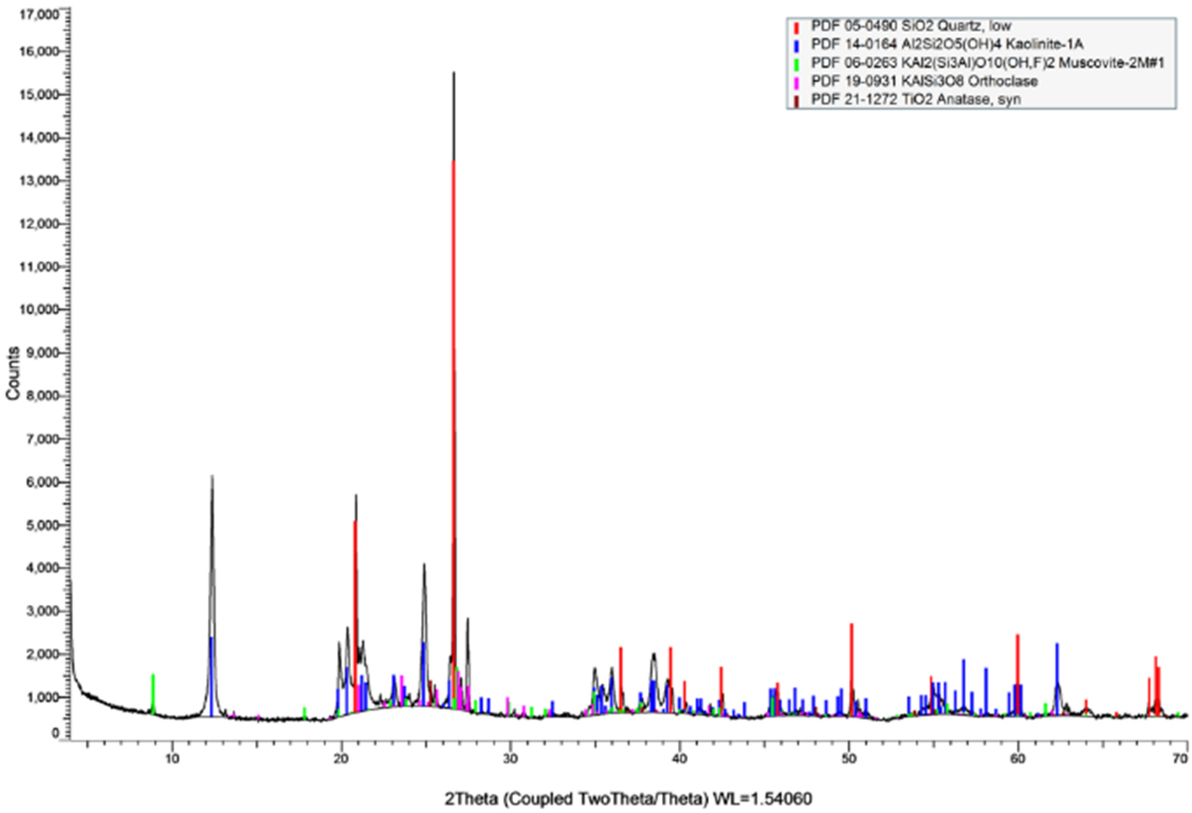

Figure 4. XRD result for the natural soil.

The X-ray diffraction test results for both natural and those inundated with distilled water (Fig. 4 and Fig. 5) show the presence of the same minerals: Quartz, Kaolinite, Muscovite, and the feldspars Orthoclase and Anatase. This indicates that there were no soil reactions in contact with water. Quartz and Kaolinite peaks are notable in both samples. The presence of Muscovite indicates a low degree of weathering process.

The specimen inundated with the 4N NaOH solution clearly demonstrates the chemical reaction mechanism between the alkaline solution and the clay minerals. It is possible to observe in the XRD diagram (Fig. 6) the degradation of kaolinite and the occurrence of new compounds, such as sodalite, a well-known compound with high expansive potential (Chavali et al, 2017). The presence of sodalite in this sample contributed to the expansion observed in the specimen tested in 4N NaOH (Fig. 3). It is interesting to observe that some Fe oxides appeared (Lepidocrocite, Hematite), and also some Vermiculite. Further mineralogical analyses searching for amorphous compounds in the natural soil are in progress to improve the analyses.

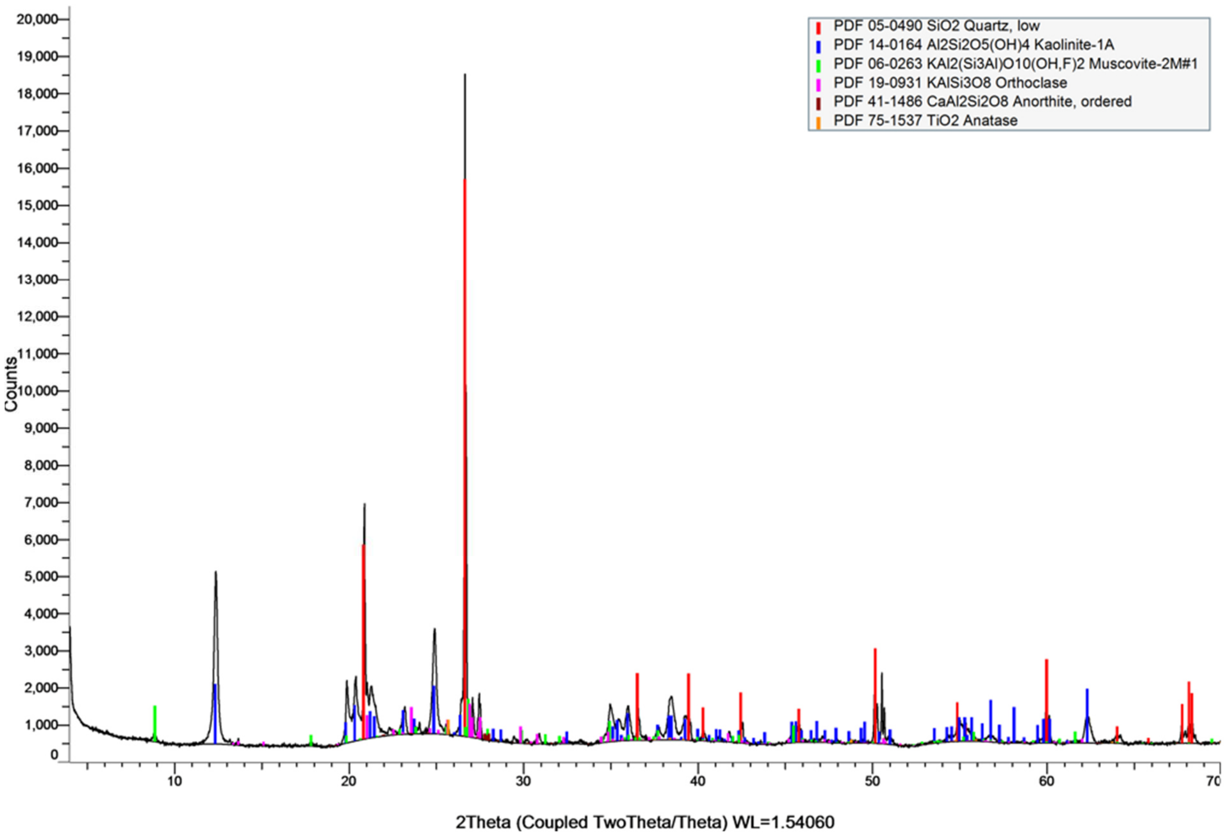

Figure 5. XRD result for soil inundated with H_2O for 45 days.

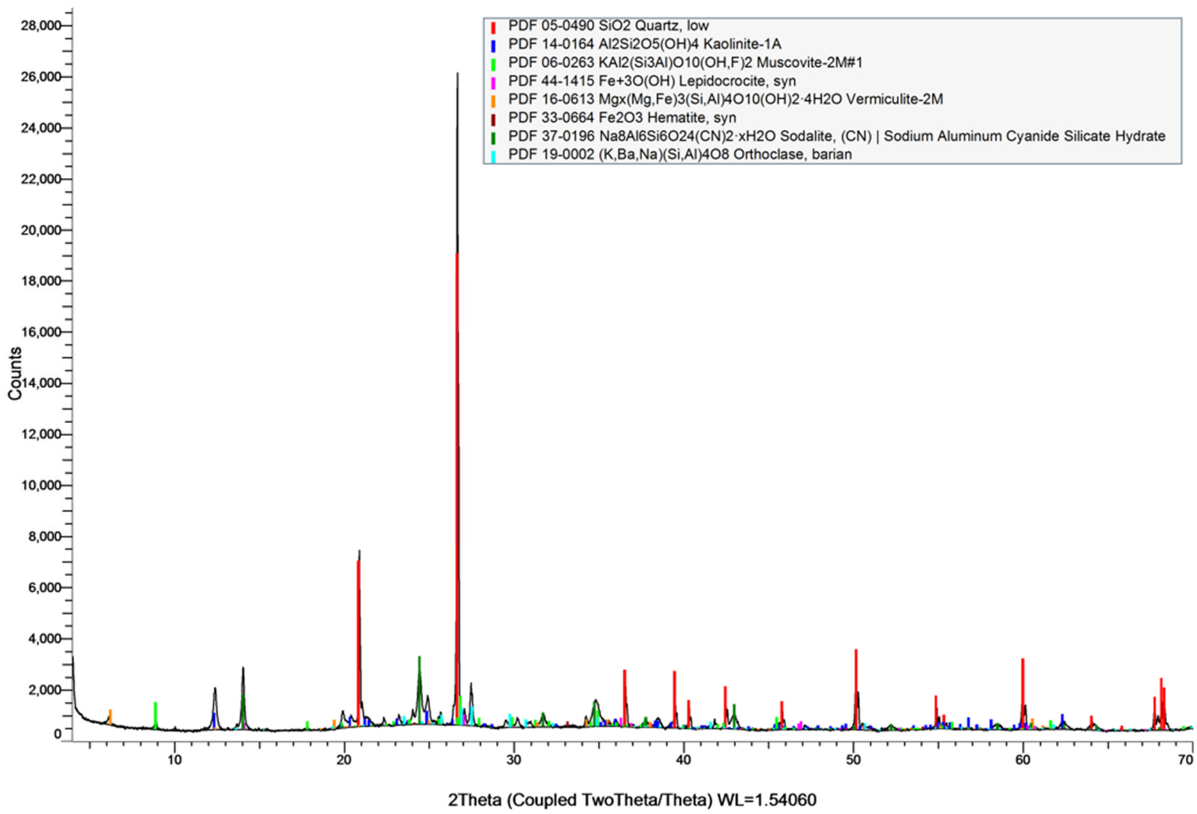

Figure 6. XRD result for soil inundated with NaOH 4N solution for 45 days.

3.6. Tests with ferric chloride solution

Two additional specimens from sample SM05 were molded under similar conditions (w_o 20% and ρ_t 1.96g/cm^3) for these tests. The initial physical parameters are presented in Tables 5 and 6. Both specimens were first inundated with the 4N NaOH solution in the oedometer, but after 10 days the alkaline liquid in cell 1 reservoir was drained out and replaced by 5% $FeCl_3$ solution and measurements continued.

The results are presented in Fig. 7, comparing the two tests: one specimen without any treatment in contact with the alkaline solution (cell 2), and the other specimen subjected to the 5% ferric chloride treatment solution (cell 1).

Table 5. Physical parameters (cell 1)

Dry unit weight (kN/m³)	15.95
Total unit weight (kN/m³)	19.19
Void ratio	0.612
Porosity	0.380
Gravimetric water content (%)	22.3

Table 6. Physical parameters (cell 2)

Dry unit weight (kN/m³)	15.57
Total unit weight (kN/m³)	19.16
Void ratio (e_o)	0.652
Porosity (n)	0.395
Gravimetric water content (%) (w_o)	23.05

At that time, the vertical swelling stress in the specimen from cell 1 had reached 15kPa. Five days after, the specimen was inundated with the treatment solution, the stress remained constant and even presented a small reduction.

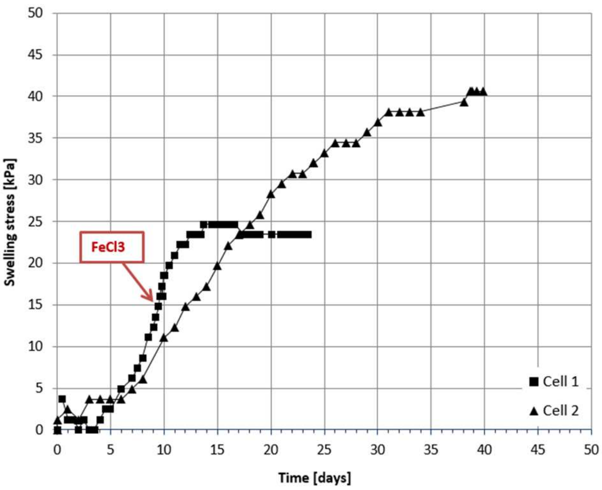

Figure 7. Ferric chloride action on swelling soil.

The addition of ferric chloride as a corrective measure can be explained by two reasons: (1) because it is an acidic compound and its reaction in an alkaline medium involves neutralization, as shown in the reaction: 3NaOH + $FeCl_3$ → $Fe(OH)_3$ + 3NaCl; (2) the precipitated iron oxide would act as a cementing agent, since some studies point to the loss of soil cementation as one of the mechanisms of soil expansion (Rao and Rao, 1994).

The Fe^{+3} ion reacts quickly with the hydroxyl (OH^-) ions, removing them from the reaction medium and preventing them from chemically attacking the minerals in the soil, especially kaolinite, that create expansive gels that can be transformed into sodalite through crystallization, as detected by XRD results.

However, it is important to note that this is not a definitive solution to the problem, since soil expansion may reoccur in case of exposure to alkaline environments (Sivapullaiah and Manju, 2005).

After 28 days, the ferric chloride solution was completely drained from the reservoir of cell 1, and the 4N NaOH solution was added again, submerging the entire specimen. It is possible to observe in Fig.8 that soil swelling restart in the specimen previously treated with 5% $FeCl_3$.

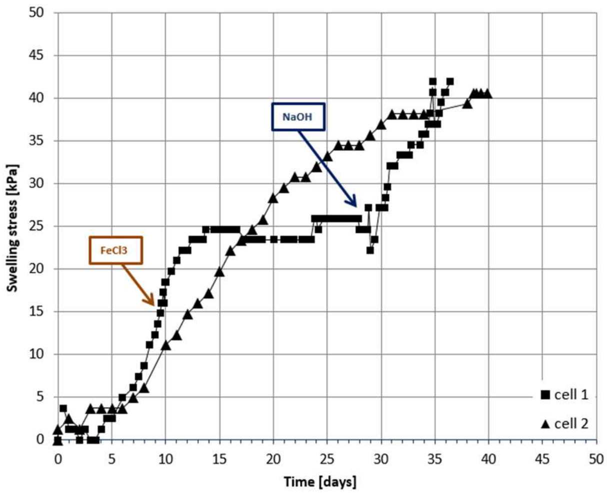

Figure 8. Swelling behaviour of treated soil specimen when subjected to new alkali contamination.

It is evident from the graph that the specimen rapidly expanded again for 7 days, resulting in a 68% increase in vertical swelling stress (to 42kPa) compared to the stabilization stress (25kPa) in $FeCl_3$ solution.

As the $FeCl_3$ solution previously added has already reacted neutralizing the NaOH, and no treatment solution was left in the cell, as the alkali contamination continues, the reactions between the remaining minerals present and the incoming NaOH occur again, resulting in the swelling behaviour observed in Fig. 8.

4. Conclusions

The results presented for a natural kaolinitic residual soil contaminated in the laboratory with alkaline solutions confirmed the reaction between the soil clay minerals and the NaOH that degrades the kaolinite and generates newly formed minerals and compounds that are expansive, such as Sodalite. This mechanism is responsible for the observed soil swelling behaviour. In the laboratory, the reactions only stabilized after 40 to 45 days, and the vertical swelling stress measured reached 40kPa for tests with a 4N NaOH solution.

A specially designed and manufactured experimental system involving an adapted Teflon® oedometer cell and reservoir was applied to measure continuously the corresponding vertical swelling stress over time until stabilization. The apparatus applies the quasi-constant volume method. The results obtained are representative of the soil swelling behaviour under confined condition, and can be useful in the foundation reinforcement design.

A chemical treatment using 5% $FeCl_3$ solution was tested. It proved to be a good corrective solution, mainly due to its neutralization action. But, as already pointed out by other authors, it does not work as a preventive method, since the treated soil can swell again if exposed to new alkali contamination.

Further research should explore other chemical treatments, looking not only into the efficacy of the expansion control but also to the risks involved when applied in the field.

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