



Assessment of the Swelling Behavior of NaOH-Contaminated Red Earth in the Visakhapatnam Region of India Using X-ray Diffraction Analysis

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ABSTRACT

Research on the impact of alkali contamination on the swelling behavior of red earth in the Visakhapatnam region has been notably limited. Therefore, this study aims to investigate the effects of alkali (NaOH) contamination on the swelling characteristics of the region's red earth. The red earth of this region was found to be a well-graded sandy soil with 81% sand and 18% fines. X-ray diffraction studies showed that this region's red earth mainly consists of quartz, kaolinite, and hematite. The soil is inherently non-swelling. However, the free swell tests showed considerable swell under contamination of NaOH solutions of various normalities (0.05, 0.1, 1, 2, and 4N). One-dimensional consolidation tests have shown that the swell increased with the concentration of the NaOH solution and with the duration of the interaction. Red earth exhibited 'an equilibrium swelling' of 5.6, 10, 15, 17, and 20% when contaminated with 0.05, 0.1, 1, 2, and 4N NaOH solutions, respectively. XRD studies revealed that the red earth sample contaminated with even 0.05N NaOH solution and cured for 56 days exhibited the formation of zeolites analcime and natrolite. Silicate minerals like paragonite and ussingite were also formed along with the zeolites. N-A-S-H compounds, hydrosodalites, and zeolites like super hydrated natrolite, zeolite SSZ16, and zeolite ZK-14 were formed at higher normalities of NaOH after a curing period of 56 days, which caused increased swell. The research demonstrated that the formation of zeolites resulting from the alkali contamination led to swelling in the red earth.

INTRODUCTION

Caustic soda (NaOH) is an inorganic pollutant with a highly deleterious impact on the soil (Reddy et al. 2017, Mulyukov 2008, Jozefaciuk 2002, Mal'tsev 1998). Soils can be contaminated by NaOH from the effluents of dyes, paper, pulp, aluminum, ceramic industries, etc., during the operation and due to the effluents (Imran et al. 2015, Sivapullaiah & Manju 2005, Mal'tsev 1998). Non-expansive soils, which were stable, became unstable due to alkali contamination (Chavali et al. 2020, Sruthi et al. 2019, Vindula et al. 2017, Chavali et al. 2017, Vindula et al. 2016). Alkali-related contamination led to foundational and structural deformation, rendering industrial facilities unusable within one to one and a half decades. In the alumina plants, alkali aluminate solutions penetrate the backfill soils causing swelling and damage to the structures, requiring a vast expenditure of material and labor for repairs (Kabanov et al. 1977). Both clayey and sandy soils exhibit swelling under contamination by alkali aluminate solutions used in alumina production. In a three-year case study at Bogoslovskii Aluminum Plant,

the floor in the alumina shop rose from 29 to 133 mm, with cracks exceeding 50 mm. A similar issue was observed at the Acha Alumina Complex, where floors were uplifted more than 80 mm over two years (Kabanov et al. 1977).

One major factor affecting NaOH-contaminated soil's behavior is its dispersion characteristics. The duration of the exposure significantly influenced the behavior of the alkali-contaminated red earth. The more kaolinite in the soil, the more susceptibility to physical, chemical, and mineralogical changes (Sruthi et al. 2019). Chemical contamination changes the pore-fluid properties of the soil and, ultimately, its settlement characteristics (Meegoda & Ratnaweera 1994). The swelling capacity of the alkali-contaminated soil depends majorly on the soil's gradation, mineralogical composition, and clay content (Chunikhin et al. 1988). "Chemical swelling" (Sorochan 1974) or "Chemical heaving" (Sokolovich 1976) increases with increasing content of the mineral alumina (Al_2O_3) and the number of clayey particles ($d < 0.005$ mm) in the soils (Mal'tsev 1998). The process of decomposition of the minerals of the soil

under chemical contamination and subsequent formation of new minerals is termed “artificial lithification” (Voronkevich 1992). RE exhibited abnormal swelling on contamination with 4N NaOH solution (Vindula et al. 2016, Sivapullaiah & Manju 2007). Notably, the heave pressure was 2-3 times the swell pressure (Sokolovich 1995).

Considerable work was done on the swelling nature of alkali-contaminated red earth in regions other than Visakhapatnam (Ma et al. 2022, Chavali et al. 2020, Wang et al. 2020, Liu et al. 2019, Sruthi et al. 2019, Ashfaq et al. 2019, Vidula et al. 2017, Chavali et al. 2017). However, such studies in the Visakhapatnam region are very limited. The RE in the Visakhapatnam region was well-graded sandy soil with a sand content of 81% and fines of 18%. The soil predominantly consists of quartz, kaolinite, and hematite (Fig. 2).

Many industries, like HPCL, Hindustan Zinc Ltd., Coromandel Fertilisers Ltd., Vizag Steel Plant, etc., were established in Visakhapatnam due to the proximity to the natural harbor and seaport, among other reasons. Hence, there is a high probability of alkali contamination in this region (Satyanarayana et al. 2021). Also, as Visakhapatnam is the fastest-growing city in Andhra Pradesh, more and more industrial development is on the cards in the coming decade. Therefore, studying the impacts of alkali contamination on the soil in this region is significant.

The objectives of the research are to analyze the swell properties and mineralogical changes of the red earth in the Visakhapatnam region, contaminated with various concentrations of NaOH solution (0.05, 0.1, 1, 2, and 4N).

MATERIALS AND METHODS

Materials

Red earth (RE), which is found abundantly in the Visakhapatnam region, was collected by open excavation from a depth of 1m from the ground level from the open land opposite Visvesvaraya Bhavan of (Lat. 17.78171705, Long. 83.37515625) of the Visakhapatnam campus of GITAM Deemed to be University. The soil composite was prepared by coning and quartering. Around 50 kg of sample was collected. Approximately 50 kg of samples were gathered, air-dried, sieved through a 425-micron IS sieve, and then stored in an airtight container.

Based on the research works of Sruthi et al. (2019), Reddy et al. (2017), Sruthi & Reddy (2017a), Sruthi & Reddy (2017b), Vindula et al. (2017), Chavali et al. (2017), Vindula et al. (2016), Reddy & Sivapullaiah (2010a), Reddy & Sivapullaiah (2010b), Sivapullaiah & Manju (2007), and Sivapullaiah & Manju (2005), 0.1, 1, 2, 4 normalities of Sodium Hydroxide (NaOH) or caustic soda were selected

as the alkali contaminants. As no research was done on the impacts of 0.05N NaOH, this normality was also included in the study. Sodium hydroxide solutions were prepared using certified ACS-grade sodium hydroxide pellets (CAS: 1310-73-2) with a minimum percent purity of 97.0%, purchased from Fisher Chemical™. These pellets have a density of 2.1 g.cm³, a molar mass of 39.9971 g.mol⁻¹, and a water solubility of 111 g.100 mL⁻¹ at 20°C.

Methods

Free swell tests: Free swell tests were carried out to determine the swell potential of the contaminated samples according to IS 2720: Part 40: 1977 (Reaffirmed Year: 2021) - Methods of test for soils: Part 40 Determination of free swell index of soils. The free swell index was calculated as the percentage increase in the original volume. These tests indicate the possible damage to structures constructed atop due to swelling.

Consolidation tests: One-dimensional consolidation (standard oedometer) tests were conducted under seating load according to IS 2720: Part 15: 1965 (Reaffirmed Year: 2021) - Methods of Test for Soils - Part XV: Determination of Consolidation Properties. These tests provide insights into settlement and swelling characteristics, which help design the ground improvement and remediation methods.

X-ray diffraction (XRD) studies: A PANalytical X-ray diffractometer was used to analyze the mineralogical changes in the NaOH-contaminated RE samples under various curing periods. The samples were grounded in a porcelain crucible, and the material passing through a 75-micron sieve was sent for XRD analysis. The X-ray tube was energized at a voltage of 60 kilovolts (kV) and a current of 55 milliamperes (mA) in conjunction with an X'Celerator ultra-fast detector. Acetone was employed to meticulously eliminate any remnants of prior materials adhered to the mortar and pestle. Subsequently, with thorough attention to detail, the powdered sample was delicately deposited and gently compacted within a rectangular stainless-steel holder. The samples were scanned from a 2θ value of 6° to 70° with a step size of 0.02° using copper k-alpha radiation. Crystal Impact Match! Software based on Crystallography Open Database (COD) was used for identifying mineralogical changes.

RESULTS AND DISCUSSION

Free Swell Tests

The free swell test results presented in Table 1 show that RE exhibited swell even with 0.05N NaOH, and the swell increased with the concentration of the NaOH solutions. This observation is in confirmation of the findings of Sruthi et al. (2019). Thus, it was

preliminarily found that the inherently non-swelling RE exhibited swelling on NaOH contamination. Further, consolidation tests were carried out to study the swelling behavior.

Consolidation Tests

Percentage swell was calculated using the final swell displacements and the original heights of the specimen (Chavali et al. 2017). The Red earth exhibited 'an equilibrium' swelling of 5.6, 10, 15, 17, and 20% (Fig. 1) when contaminated with 0.05, 0.1, 1, 2, and 4N NaOH solutions, respectively. The swell with time was impacted by the concentration of NaOH solution (Reddy & Sivapullaiah 2010b). The alkali-silica reaction, which formed an alkali-silicate gel, was the primary cause of the swell (Sibley & Vadgama 1986).

The higher the concentration of the alkali solution, the faster the reaction between the clay fraction of the soil and the alkali solution. Hence, relative stabilization occurs in a relatively lower period (Kabanov et al. 1977). The higher percentage of swell exhibited by RE at higher concentrations of NaOH can be attributed to the dissolution of the authigenic mineral structure (Vindula et al. 2016) and the formation of the zeolites (Chavali et al. 2017, Sivapullaiah & Manju 2007) which resulted in more significant heave. The formation of zeolites was discussed in the XRD Analysis section. The swelling can also be attributed to the changes in pore-fluid properties (Meegoda & Ratnaweera 1994). The interaction of clayey minerals with alkalis causes mineralogical

decomposition and highly hydrated formations, resulting in the volumetric increase (Sokolovich & Troitskii 1976).

It is significant to note that compaction does not resist the swelling tendency of the foundation soil on interaction with NaOH solution. Prolonged chemical interaction between the spilled caustic soda solution and the kaolinitic red soil from Bangalore (now Bengaluru) district converted the inherently non-swelling soil to soil with high swelling potential (Rao & Rao 1994).

X-Ray Diffraction (XRD) Analysis

XRD tests were conducted, and the resulting graphs were analyzed to identify the new mineral formations influencing the RE's swelling behavior. Microstudies helped understand the interaction of the alkali contaminant (NaOH) with the soil (RE), which further gives an understanding of geotechnical problems like uneven settlements and progressive failures (Sivapullaiah & Manju 2007). Fig. 2 depicts the XRD analysis of RE and the NaOH-contaminated RE samples. The reaction of alkaline contaminants with RE was prolonged. Hence, it is difficult to find the damage in the early stages (Sruthi & Reddy 2017b). At 25°C, kaolinite continues to react with hydroxide ions even after 50 days, and the reaction is irreversible. The reaction of kaolinite and high concentrations of NaOH solution proceeds incongruently, leading to the formation of new minerals, mostly zeolites (Mohnot et al. 1987). Hence, the XRD results were analyzed after a 56-day curing period.

Table 1: Free swell index of NaOH-contaminated RE Samples.

Property	RE + Water	RE + 0.05N NaOH	RE + 0.1N NaOH	RE + 1N NaOH	RE + 2N NaOH	RE + 4N NaOH
Free Swell Index(%)	-	~10	~12	~16	~22	~28

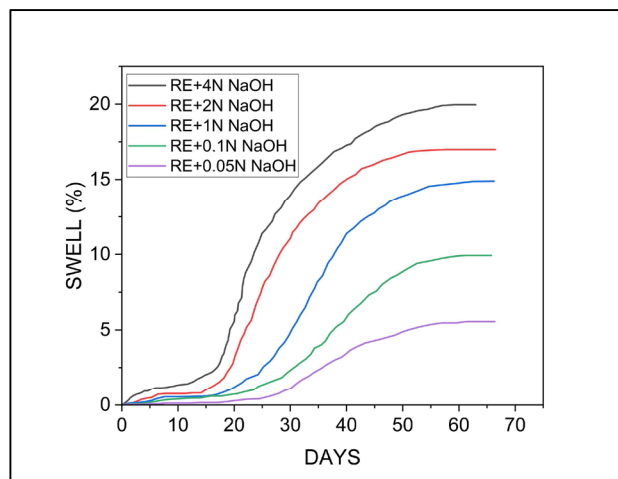


Fig. 1: Variation of swell percentage with duration for red earth contaminated with 0.05, 0.1, 1, 2, and 4N NaOH solutions.

Table 2: X-ray diffraction analysis codes for naoh-contaminated re samples cured for 56 days.

A56	RE + 0.05N NaOH
B56	RE + 0.1N NaOH
C56	RE + 1N NaOH
D56	RE + 2N NaOH
E56	RE + 4N NaOH

XRD pattern (fig. 3) of RE showed the significant peaks of quartz (at 4.21, 3.32, and 1.81 [Å]), kaolinite (at 7.07, 3.54, and 2.27 [Å]), and hematite (at 2.55, 1.67, and 1.45 [Å]). XRD pattern of RE contaminated with even the low concentration of 0.05N NaOH (A56) showed the peaks of zeolites like analcime (at 2.34 [Å]) and natrolite (at 3.57 [Å]). Peaks of ussingite (at 3.83 and 2.99 [Å]) and paragonite (at 2.56, 2.45, and 2.13 [Å]) were also observed. There was an apparent reduction in the quartz and kaolinite peaks, indicating the reaction of NaOH with silica and alumina, which led to the formation of zeolites analcime and natrolite. As the RE in the study area is sandy soil, the formation of silicate minerals like ussingite and paragonite was observed. It is significant to note that even at lower concentrations, zeolites were formed for a curing period of 56 days, indicating the mineralogical changes.

RE contaminated with 1N NaOH (C56) exhibited the peaks of analcime (prominent peak at 2.34 [Å]), natrolite (major peaks at 3.58 and 2.28 [Å]), sodium aluminosilicate hydrate (N-A-S-H) compound (at 2.57 [Å]), ussingite (prominent peak at 3.04 [Å]), paragonite (at 9.94, 4.26, 3.24, and 1.49 [Å]). No kaolinite peaks were observed, indicating the complete reaction of NaOH with the alumina and silica of kaolinite. Peaks of quartz were also reduced. On reacting with 2N NaOH, RE (D56) had shown the peaks of zeolite SSZ16 (at 10.06 [Å]), natrolite (at 4.46, 3.57, and 2.28 [Å]), hydrosodalite (at 2.56, and 2.13 [Å]), N-A-S-H compounds (at 2.88, and 2.45 [Å]), paragonite (at 4.25, 2.52, 1.93, and 1.49 [Å]) and ussingite (at 3.03 [Å]). At 4N NaOH, RE (E56) exhibited peaks of zeolites ZK-14 (at 5.02, 2.13, and 1.82 [Å]), analcime (at 5.57, and 2.80 [Å]), natrolite (at 4.46, 3.58, and 2.28 [Å]), super-hydrated natrolite (at 2.89, and 2.34 [Å]). Peaks of N-A-S-H compounds (at 7.17, 2.56, and 2.46 [Å]), ussingite (at 3.77, and 3.00 [Å]), and paragonite (at 4.25, 3.19, 2.51, and 1.49 [Å]) were also exhibited. Peaks of silica were further reduced, giving rise to new zeolites.

The reaction of Kaolinite and NaOH resulted in the formation of sodium aluminosilicate hydrate (N-A-S-H) compounds. The amount of N-A-S-H formed depends on the clay mineral in the soil, concentration of alkali solution, and interaction time. As the normality increased, the quantity of N-A-S-H compounds also increased. For RE

with less kaolinite, like that in the Visakhapatnam region, the changes in the properties were predominantly due to the formation of N-A-S-H compounds and zeolites (Sivapullaiah & Manju 2005). Changes in the surface charge of alkali-exposed minerals resulted in the formation of new minerals (Jozefaciuk 2002). When the alkali attacked the nucleus of the soil particle, the behavior of the soil was governed by the zeolites (Sruthi et al. 2019).

More aluminous zeolites like analcime are more likely to form in alkaline environments (Ming & Boettinger 2001). Under high pH, kaolinite was converted to water-sensitive analcime. The formation of analcime increased with alkalinity (Zhuang et al. 2018). The reaction of NaOH solution with kaolinite formed Analcime. Analcime is usually formed at lower alkalinity levels (Zhang et al. 2022). Analcime, a microporous zeolite, has a high specific surface area, cation exchange, catalytic capacity, and high adsorption per surface area (Jin et al. 2022, Ma et al. 2015, Atta et al. 2012), and hence influences the swelling behavior and the stability of the soil. Natrolite is a fibrous small-pore zeolite with a total pore volume of 22%. It consists of helical channels of an elliptical section allowing water diffusion (Tsai et al. 2021, Sabylnskii et al. 2016, Paczwa et al. 2016, Demontis et al. 2005, Ibrahim 2004, Line & Kearley 1998, Engelhardt & Michel 1987), which resulted in swelling. Swell caused due to the formation of sodium aluminosilicate hydrate (N-A-S-H) compounds is well documented (Sivapullaiah & Manju 2007), (Sivapullaiah & Manju 2005). N-A-S-H gel is a precursor reaction product to zeolites (Zhou et al. 2022).

Ussingite is a tectosilicate mineral (Williams & Weller 2012) and one of the most apatitic minerals of aluminosilicates. In natural processes, ussingite replaces sodalite (Kotel'nikov et al. 2010). Paragonite is a basic sodium aluminum silicate that belongs to the mica group. It is a rare sodium analog of mica or sodic mica (Gupta & Fareeduddin 2013, Chatterjee 1970). Ussingite and paragonite might not have contributed to the swell. However, their formation, along with the other zeolites, might have increased the swell.

In sample D56 (Table 2), N-A-S-H compounds and natrolite zeolite formation increased. Zeolite SSZ16, a small-pore high-silica zeolite of ABC type, has good adsorption capacity (Lobo 1996). Hydrosodalite is another small-pore zeolite with decent adsorption and high ion exchange capacity (Arasi 2020, Aprea 2014). The formation of these zeolites caused the swell in D56.

In sample E56 (Table 2), along with N-A-S-H compounds, zeolites analcime, natrolite, super hydrated natrolite, and zeolite ZK-14 were also formed. Zeolite ZK-14 is a synthetic chabazite with an open pore structure and stacking faults

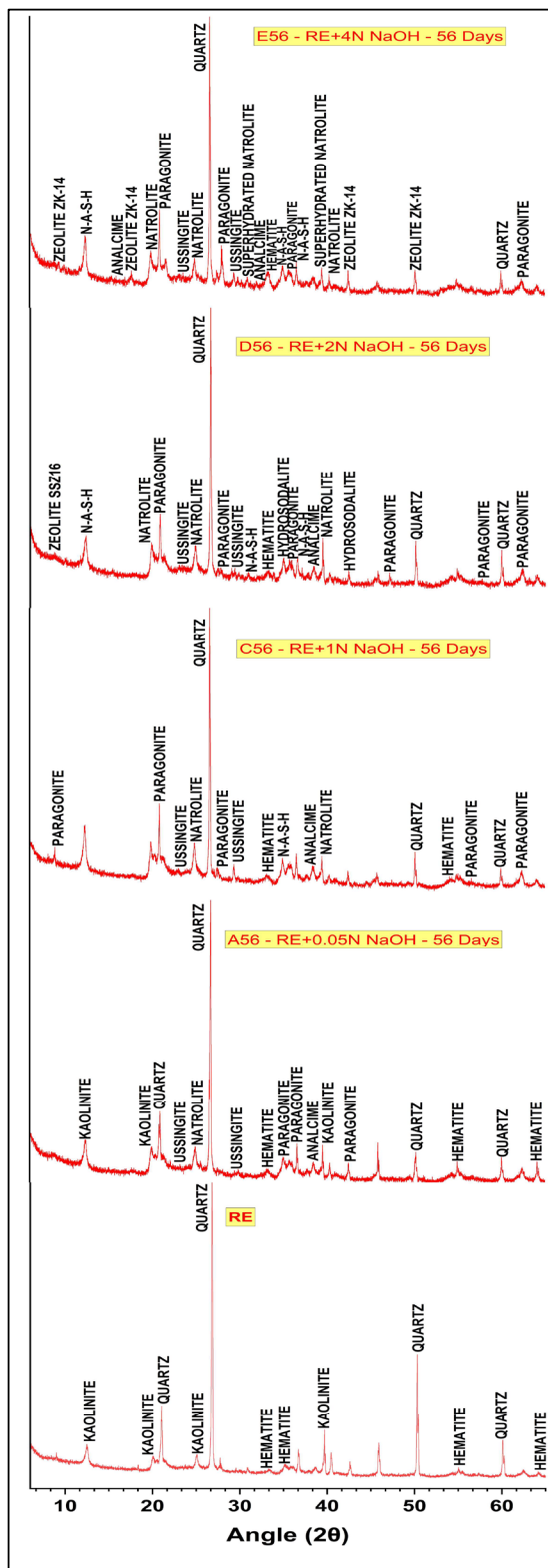


Fig. 2: XRD analysis for RE and RE contaminated with 0.05, 1, 2, and 4N NaOH solutions for a curing period of 56 days.

(Silva et al. 2016, Cartlidge et al. 1983) contributing to the swell. Super hydrated natrolite formed from natrolite by phase transition at 4N NaOH interaction caused anomalous anisotropic swelling in RE due to the selective sorption of NaOH. Superhydrated natrolite contains twice as much water content as that of natrolite. Also, the compressibility of the superhydrated natrolite is more than that of natrolite (IZA n.d.) (Lee et al. 2002, 2001, Belitsky et al. 1992). Hence superhydrated natrolite causes more swell and severe heave than those caused by natrolite.

CONCLUSIONS

- The inherently non-swelling red earth in the Visakhapatnam region underwent swelling when contaminated with NaOH solution, even at 0.05N.
- The swell percentage increased with the increase in the concentration of NaOH solution and the duration of the interaction.
- Red earth exhibited 'an equilibrium' swelling of 5.6, 10, 15, 17, and 20% when contaminated with 0.05, 0.1, 1, 2, and 4N NaOH solutions, respectively.
- RE+0.05N sample exhibited the formation of zeolites analcime and natrolite, along with silicate minerals ussingite and paragonite.
- The RE+1N sample showed the formation of zeolites analcime and natrolite and N-A-S-H compounds, which are precursor reaction products to zeolites.
- In the RE+2N sample, the formation of N-A-S-H compounds and natrolite zeolite increased. Zeolite SSZ16 was formed.
- In the RE+4N sample, super hydrated natrolite and zeolite ZK-14 were formed.
- The formation of the zeolites under the action of various normalities of NaOH solution caused a swell in the naturally non-swelling red earth in the study area.

The study has yielded pivotal findings, offering actionable insights that can be effectively leveraged to mitigate soil contamination issues prevalent in industrial regions where detrimental alkali discharges and effluents are prominent.

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