WE-CEC at the two MN roadsides. CGR and depth can be seen to cause significant differences, as reflected in their *p*-values greater than 0.05. With respect to interactions, Site 1 and Site 2 provide different conclusions because their soil compositions, CGR sources, and environmental conditions are different. CGR does not negatively affect the ability of soil matrix to hold nutrient cations.



Figure 5. The results of concentration of (a) Ca, (b)Mg, (c)K and (d) Na at the Minnesota roadsides.

Exchangeable Sodium Percentage: Figure 7 shows that the control area exhibits the highest ESP value at Site 1, but at Site 2 the non-CGR area exhibits a higher ESP than the other areas. Soil depth also influences ESP, with the deepest layer exhibiting the highest ESP. Table 1 presents the results from ANOVA model for ESP at the MN sites. Depth is a significant factor, and the deeper layer exhibits the highest ESP values. At Site 1, CGR significantly influences ESP, contrary to the CGR area and the control area at Site 2, with different soil composition, CGR source, and environmental condition responsible for these differences. It is possible that an increase in soil ESP can lead to reductions in soil water infiltration (Subbarao et al., 2003). The lowest ESP value in the CGR area indicates that an application of CGR has no negative influence

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on soil sodicity.

 Table 2. The ANOVA results of concentrations of leached metals at the (a) Minnesota Site 1 and (b) Site 2.

(a)												
Companison	Egotong	<i>p-value</i>										
Comparison	Faciors	Ca	K	Mg	Na	Al	Ba	Cr	Си	Fe	Mn	Zn
CGR area and non-CGR area	CGR	< .01	0.02	0.02	<.01	0.21	0.43	0.61	0.15	0.08	0.78	0.25
	Depth	< .01	< .01	< .01	< .01	0.10	0.17	0.16	0.22	0.20	0.08	<.01
	Interaction	0.24	0.63	0.09	<. 01	0.15	0.23	0.51	0.20	0.66	0.75	0.10
CGR area and control	CGR	< .01	0.03	<.01	<.01	0.39	0.29	0.45	0.23	0.29	0.68	0.65
	Depth	< .01	<.01	<.01	0.06	0.10	0.28	0.69	0.76	0.81	0.06	0.09
area	Interaction	<.01	0.15	0.81	0.34	0.78	0.43	0.45	0.17	0.79	0.70	0.58
(b)												
Comparison	Egotong	p-value										
	FUCIOIS	Ca	K	Mg	Na	Al	Ba	Cr	Си	Fe	Mn	Zn
CGR area and non-CGR area	CGR	<.01	<.01	0.32	0.04	0.55	0.51	0.06	0.89	0.42	0.86	0.14
	Depth	<.01	<.01	<.01	0.19	0.57	0.59	0.43	0.29	0.36	0.06	0.55
	Interaction	0.12	0.04	0.54	0.88	0.40	0.88	0.99	0.47	0.47	0.12	0.87
CGR area and control	CGR	< .01	<.01	0.62	0.71	0.87	0.49	0.36	0.61	0.31	0.722	0.08
	Depth	<.01	<.01	<.01	0.02	0.59	0.18	0.40	0.04	0.46	0.135	0.94
area	Interaction	0.44	<.01	0.19	0.89	0.94	0.38	0.13	0.14	0.34	0.715	0.40



Figure 6. The results of WE-CEC at the Minnesota roadsides.

Percent Base Saturation: PBS results with Std from the MN sites are presented in Figure 8. At MN Site 1, the CGR area exhibits higher PBS than both the control area and the non-CGR area, while at MN Site 2, the non-CGR area exhibits a lower PBS than the others. The surface layer PBS is also higher than for other layers, indicating that the CGR area has relatively more Ca, K, and Mg than the non-CGR and control areas. Table 1 displays the statistical results of PBS at the MN sites, showing that CGR and depth significantly affect PBS at Site 1, while at Site 2, only depth significantly influences PBS. Since PBS is determined by the concentration of metals (Ca, Mg and K) and WE-CEC, differences in statistical results can be a result of the

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different soil compositions, CGR sources, and environmental activities.

Figure 7. The results of ESP at the Minnesota roadsides.



Figure 8. The results of PBS at the Minnesota roadsides.

CONCLUSIONS

Based on tests conducted at two sites where CGR was previously offloaded along I-90 near Austin, MN, to evaluate the effects of concrete grinding residue on chemical properties of soil, key findings can be summarized as follows:

- The pH of the area receiving CGR slurry is about 0.78 lower than the areas not receiving CGR.
- The CGR area exhibits the highest EC of 1.65 dS m⁻¹, and EC decreases to 0.69 dS m⁻¹ with soil depth.
- The surface layer of the control area exhibits the highest alkalinity of 98 mg L⁻¹ as CaCO₃.
- The Ca concentration in the CGR area is about 97 mg kg⁻¹ higher than the areas without CGR, but for other metals (K, Mg and Na), Sites 1 and 2 exhibit different trends.
- The levels of toxic metals such as Ba, Cr, Cu, Fe, Mn, and Zn are not significantly influenced by CGR at either site because the p-values are greater than 0.05.
- CGR areas exhibit higher WE-CEC values, and the WE-CEC of 2.89 meq 100g⁻¹ at topsoil is larger than for other layers.

- The CGR areas at the Minnesota Site 1 and 2 exhibit the lowest ESP values of 6.5% and 7.9%, respectively, and the deeper soil layers in general have larger ESP than the top-surface soil.
- The CGR areas exhibit larger PBS values of 93.5% and 92.1% than the other areas at the Minnesota Site 1 and 2, and the deeper soil layers exhibit lower PBS values of 78.9% and 75.8%, respectively.

Two Minnesota roadsides are studied to investigate the long-term effects of CGR on roadside soil chemical properties. The results for Site 1 show that CGR significantly influences alkalinity, WE-CEC, ESP, and PBS. For Site 2, the results differ from those of Site 1, indicating that the CGR significantly influences soil alkalinity and WE-CEC. Multiple factors contribute to the differing results for Sites 1 and 2, including the de-icing agents used on road surface, vehicle emissions, CGR composition, CGR discharge rate, and time after spreading. The non-CGR area exhibits results different from the control area, indicating that slope is another important factor. Because CGR applications do not cause major changes in soil properties or reductions in soil quality, CGR is not found to be of long-term concern with respect to roadside environments.

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Effect of Acid and Alkali Contamination on Swelling Behavior of Kaolin Clay

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ABSTRACT

This paper aims at studying the influence of acid and alkali solutions on the swell behavior of a non-expansive soil. Sulphuric acid (acidic medium); sodium hydroxide (alkaline medium) of 1 N and 4 N concentrations were used as pore fluids to understand the influence of acid and alkali pore fluids on the swell behavior of kaolin clay relying on one-dimensional free swell tests. Results showed that kaolin clay exhibits maximum equilibrium swelling of about 41% in acidic medium and 22% when interacted with alkaline medium. It is further observed that the magnitude of induced swell in clays is dependent on dispersion of clay particles leading to the formation of new minerals under both acidic and alkaline media. In order to better understand the mechanism responsible, the contaminated soil was investigated for changes in mineralogy and microstructure relying on x-ray diffraction (XRD) analysis, scanning electron microscopy (SEM) at the end of interaction. The formation of zeolite minerals viz. sodalite in alkaline media and aluminum sulphate hydroxide in acidic media were noted from XRD results. Furthermore, SEM studies revealed rosette-type morphological structure(s) in case of 4N NaOH and needle-like structures in H₂SO₄.

Keywords: Acid, Alkali, Free Swell, Kaolin clay, XRD, SEM

INTRODUCTION

One of the major causes for foundation and superstructure deformations at industrial locations associated with the production and utilization of inorganic chemicals is soil contamination by leakage or spillage of inorganic pollutants. It is well established that soils exhibit a capacity to swell under the action of various inorganic pollutants such as salts, alkalis, and acids (Sokolovich 1976; Sokolovich 1995; Sivapullaiah et al. 2009; Reddy et al. 2017; Chavali and Reddy 2018). Both crystalline swelling and formation of neogenic minerals during interaction of contaminant with the soil are responsible in dictating overall swelling of soil (Mal'tsev 1998). In the past few years, a number of reported structural failures have been documented due to very large amounts of heave in the subsoil (Vronskii et al. 1978, Sridharan et al. 1981, Shekhtman et al. 1995, Assa'ad 1998, Al-Omari et al. 2007).

Sivapullaiah et al. (2009) studies found that the mineralogical changes in particular formation of acid sulphate minerals lead to increase in swelling. Chavali et al. (2017) reported the swell

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trigger in non-expanding kaolinitic clays such as ball clay, china clay, and red earth due to alkali contamination. Later on, Reddy et al. (2017) investigated the influence of acidic and alkaline solution on the swelling behavior of black cotton soil and summarised that mineralogical changes due to acidic and alkaline contamination lead to an increase in swelling. In line with the above studies, the current study also focused to investigate the influence of acid and alkali on swelling behavior of kaolin clay.

MATERIALS AND METHODOLOGY

Kaolin clay: Kaolin clay, white in color, was procured from Godavari Mines and Minerals, Visakhapatnam, India. Cation exchange capacity of kaolin clay was 5.6 meq/100 g. The kaolin clay had a liquid limit of 52, and a plasticity index of 25. From the X-ray diffraction analysis, it is found that the clay consists of kaolinite, calcite, and ankerite as predominant minerals.

Solutions used: In this study, distilled water, sulphuric acid (H_2SO_4) solution, and sodium hydroxide (NaOH) solution were used as pore fluids.

One-Dimensional free swell test: The one-dimensional free swell test (as per ASTM D4546-14e1) measures the amount of swelling in the vertical direction of a confined specimen (Puppala et al. 2005). Chemically non-reactive Teflon cells (6 cm in diameter and 2 cm in height) were used to perform free swell tests. Oven-dried clay (at 110° C) was cooled and spread over an impermeable sheet and mixed with water to achieve maximum dry density (MDD) at a given optimum moisture content (OMC). The mixed specimens were kept in a desiccator in sealed plastic bags for 24 hours so as to achieve uniform moisture content.

In order to reduce friction during compaction process, silicon grease was applied to the inner surface of the oedometer ring. Compaction was done in three layers to get MDD of 14.8 kN/m³ at given OMC of 27.3% in a rigid Teflon cell to a height of 1.4 cm. To allow the movement of pore fluid, porous stones were placed at both top and bottom of the soil specimen. The specimens were saturated with solutions of distilled water, NaOH and H_2SO_4 as pore fluids for respective scenarios and allowed to swell under free loading conditions. The final swell (at which no significant changes were observed in dial gauges readings) and the original height of the clay specimen were used to determine % swell in the vertical direction. Representative clay samples were collected at the end of each test and were analyzed for morphological and mineralogical changes using Scanning electron microscope (SEM) and X-ray diffraction (XRD) studies.

Scanning electron microscope analysis: Morphology (physical examination of microstructure) of the soil was studied using scanning electron microscope (VEGA 3 series) with a focused beam of electrons using a tungsten filament.

X-ray diffraction analysis: The mineral composition of selected soil was determined using PANanalytical X-pert powder diffractometer relying on a conventional test procedure to identify new mineral formations. The scanning angle was maintained at 6° to 70° with a step size of 0.02°. Kaolinite, calcite, and ankerite are the dominant minerals observed in the natural soil.

RESULTS AND DISCUSSIONS

Swelling behavior of kaolin clay exposed to given pore fluid: Unlike with water, considerable swelling was observed when clay sample interacted with acid and alkali solutions individually (Fig. 1). An equilibrium swelling of 3% was exhibited by clay when saturated with water. In addition, clay exhibited about 23% and 12% equilibrium swelling with 1N H₂SO₄ and 1N NaOH solutions, respectively. Consequently, an equilibrium swelling of about 41% and 22% was revealed by kaolin clay exposed to 4N H₂SO₄ and 4N NaOH solutions, respectively.



Figure 1. Swelling (%) of kaolin clay exposed to given pore fluids

It can be evidenced from Fig. 1 that an increase in concentration of an acid and alkali solutions leads to an increase in equilibrium swelling. Alterations in the aluminosilicate nucleus of clay minerals and neogenic mineral formations lead to an increase in equilibrium swelling during soil contaminant interaction (Sokolovich, 1995, Chavali et al. 2017, Chavali and Reddy 2018).



Figure 2. Acid interaction with kaolin clay

Mechanism involved in swell behavior of china clay: In acidic environments, the charges on the edges of kaolin clay particles will become increasingly positive due to adsorption of hydrogen ions (Fig. 2). As the edge sites become positive, the face-to-edge Coulomb attraction prevails. This process increases the volume of kaolin clay, which results in high swelling.

In alkaline environments, the swelling in kaolin clay is mainly governed by three possible

mechanisms: (i) adsorption of OH⁻ ions on the edges of the particles, which are responsible for dispersion of particles initiating swelling (Fig. 3); (ii) formation of new minerals as shown in equation 1, and the same is observed from Fig. 4; (iii) leaching of calcite mineral coatings (Fig.4).

$$Al_2Si_4O_{10}(OH)_2 + NaOH \rightarrow NaAlSi_2O_6 \cdot H_2O \rightarrow$$
(1)

Mineralogical changes in kaolin clay exposed to given pore fluid: XRD analysis revealed neogenic formations, which triggered an increase in swelling of kaolin clay upon interaction with acidic and alkaline pore fluids. When kaolin clay is exposed to H₂SO₄ solution of given normality, the contaminant breaks the carbonate bonds, resulting in the formation of "loose" structure with voids, which eventually manifests as increased swelling (Imai et al., 2006). Formation of new minerals viz., aluminum sulphate hydroxide hydrate, and calcium sulphate hydrate (gypsum) were observed in clay followed by acidic interaction (Fig. 4).



Figure 3. Alkali interaction with kaolin clay

XRD results revealed decreased or disappearance of kaolinite peaks when kaolin clay was exposed to 1N NaOH and 4N NaOH (Fig. 4). The disappearance of kaolinite peaks led to the formation of new minerals viz., sodium aluminium silicate hydroxide hydrate (sodalite) and sodium aluminium silicate calcium carbonate hydrate (cancrinite). Further, from Fig. 4, it is observed that sodalite, a mineral belonging to zeolite group, was formed due to precipitation of dissolved silica and alumina with NaOH. Also, cancrinite formed due to the precipitation of dissolved alumina, calcium and silica with NaOH.

Morphological changes in kaolin clay exposed to given pore fluid: Micrographs from SEM analysis of selected clay interacted with given pore fluid is shown in Fig. 5. The hexagonal layered crystalline structure is observed for the micrograph of kaolin clay saturated with water. The kaolin clay microstructure altered significantly after being exposed to acid and alkali solutions, supporting the mineral structure transformation(s) in respective cases. The layered crystalline structures modified into needle-like microstructures upon interaction with 1N and 4N concentrations of H₂SO₄. In general, aluminum sulphate and calcium sulphate minerals are characterized by the needle-like microstructure (Fig. 5). Furthermore, rosette and pellet microstructures, characteristic of sodalite minerals, were observed when kaolin clay interacted with 1N and 4N concentrations of NaOH.



Figure 4. Mineralogical transformations of contaminated kaolin clay exposed to given pore fluids



Figure 5. Morphological transformations of kaolin clay exposed to different pore fluids