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Effects of Sulphuric Acid on Black Cotton Soil

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Abstract: Sub surface soil pollution by various processes with high concentration of contaminants can significantly alter geotechnical properties of soils causing unexpected failures of structures founded on them. The changes can occur due to alteration in soil water interaction processes and by intense chemical interactions leading to mineralogical and micro structural changes. Unexpected behavior of soil upon contamination with acid solution is one of the major challenges faced by the geotechnical engineers. This paper is an attempt to understand the mineralogical and micro-structural changes occurring in black cotton soil by immediate interaction with sulphuric acid.

Keywords: *Acid, black cotton soil, mineralogy, morphology.*

1. Introduction

Increasing industrialization has led to release of variety of pollutants into the environment contaminating the soil. Ground pollution arises from the impact of past and current industrial activity and due to improper disposal of waste generated by society. One of the major sources of soil pollution is discharge of industrial waste into soil through leakages from waste containment facilities, accidental spills and industrial operations. Soil-pollutant interaction can cause changes in soil behavior and can also lead to various geotechnical problems. Attempts to understand the soil response to various pollutants and methods to control the same are to be made.

One of the major industrial soil pollutants are alkalis and acids. Chemical heaving of clayey soils occurs when they are wetted by aqueous solutions of alkalis and acids. Foundation problems are being increasingly recognized mainly due to spillage of acids into soil. Intense heaving due to the infiltration of acids into the bed soils can cause dangerous deformations of the foundations and super structures. Studies has indicated that physico-chemical reaction taking place between clayey minerals and acid actively decompose clay minerals resulting in increased volume and decrease in strength. The nature of soil pollutant interactions depends on the mineralogy of soil and type and concentration of the pollutant [1]. The sensitivity of soil to environment depends not only on the local environment but also influenced by mineral structure, such as particle size, bonding characteristics between particles, ion exchange capacity, etc. [2].

Acids are one of the major waste effluents from industries. Acid solutions are released into the soil environment from various industries such as copper smelting industries, production of fertilizers, refining of petroleum, oil-burning industries, and lead-acid storage battery etc. In 2004, 180 million tons sulphuric acid was produced in the world out of which Asia constitutes 35%. Sulphuric acids are widely used in industries such as petrochemicals, paper, Iron & Steel. Phosphoric Acid is another acid which is used extensively in fertilizer and leather industry and its effects on soils holds importance in context to India as it is an agrarian country.

2. Effects of Contamination

Prolonged interaction of soil with the contaminants can lead to accumulation of contaminants in top soils. It may affect the buildings, dams, highways which cause landslides, settlements, soil erosion and underground structural instability. Results of some studies indicate that the detrimental effect of seepage of acids into subsoils can cause severe foundation failures [3]. The contaminated soil, depending on nature of pore fluid, shows significant changes in swell potential values [4]. Investigations carried out on Khvalynian clays showed that even an insignificant content of sulfuric acid in water leads to a substantial increase of swelling [5].

Based on literature reviewed it is clear that there is a significant effect of H_2SO_4 on the foundation soil leading to abnormal behaviour. Though the mechanism for such behaviour was explained, the mineral transformation and microstructural variations have not been focused. A geotechnical engineer, based on conventional geotechnical principles, could not completely explain the behaviour of acid contaminated soil without considering the significant mineralogical and microstructural changes that may occur. Hence in this paper, an attempt was made to show the effect of different concentrations of sulphuric acid on the mineralogical and microstructural changes that may influence the engineering properties of soil.

3. Materials and Methods

Black cotton soil used in this study was collected from NIT Warangal campus of Telangana state. Table 1 shows the geotechnical properties of black cotton soil. Soil is having a liquid limit of 61% and is classified as clay of high compressibility (CH) as per IS: 2720 (part 5) classification.

Sulphuric acid was used as a pollutant in different concentrations of 1N, 4N and 8N. Experiments were conducted on black cotton soil to determine the mineralogical and microstructural changes along with elemental composition using X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Energy Dispersive Analysis of X Rays (EDAX) analyses respectively.

Table 1: *Geotechnical properties of black cotton soil*

Properties	Value
Gravel (%)	10
Sand (%)	28
Silt (%)	42
Clay (%)	20
Liquid limit (%)	61
Plastic limit (%)	27
Plasticity index	34
Specific gravity	2.68
Optimum moisture content (%)	19.62
Maximum dry unit weight (g/cm^3)	1.68
Free swell index (%)	77
Unconfined compressive strength (kg/cm^2)	2.80
Soil classification	CH

4. Preparation of Acid Contaminated Soil

The soil is thoroughly mixed with 1N, 4N and 8N sulphuric acid at a ratio of 1:2 and kept for 1 and 14 days curing period. The sample is taken out and dried after their respective curing period. The dried samples were analyzed to observe mineralogical and microstructural changes.

5. Results and Discussions

5.1. Microstructural Studies for 1-Day Curing Period

Microstructural changes for 1day interaction of soil-pollutant are obtained using SEM Analysis. Fig. 1-4 show the SEM images of uncontaminated soil and soil contaminated with 1N, 4N and 8N sulphuric acid respectively.

The untreated black cotton soil shown in Fig. 1 exhibits a fairly flocculated type of microstructure. This may be due to the presence of iron oxides, which might have added the flocculent nature to the black cotton soil [6]. The SEM images of soil contaminated with 1N and 4N sulphuric acid shown in Fig. 2 and Fig. 3 shows partial disintegration by exhibiting a fairly open type of microstructure compared with the uncontaminated soil sample. The SEM image of soil contaminated with 8N sulphuric acid shown in Fig. 4 indicates complete disintegration of particles with clearly defined boundaries and shapes. The dispersed nature may be due to cation and mineralogical changes.

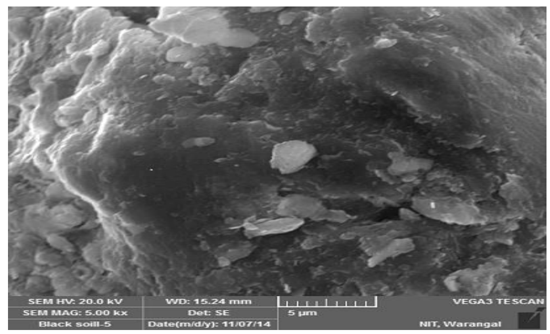

Figure 1 *SEM micrograph of uncontaminated black cotton soil*

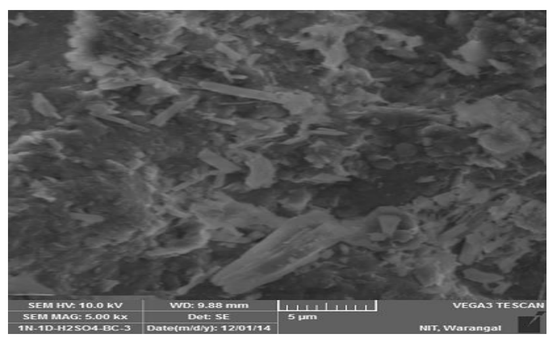

Figure 2 *SEM micrograph of black cotton soil contaminated with 1N H_2SO_4*

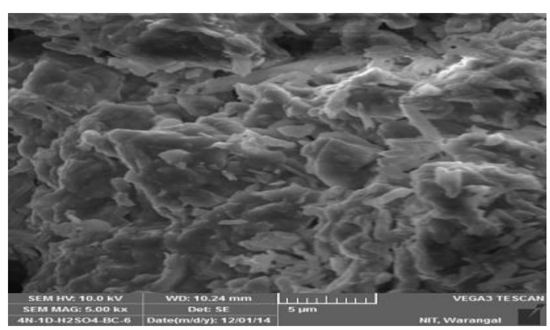

Figure 3 *SEM micrograph of black cotton soil contaminated with 4N H_2SO_4*

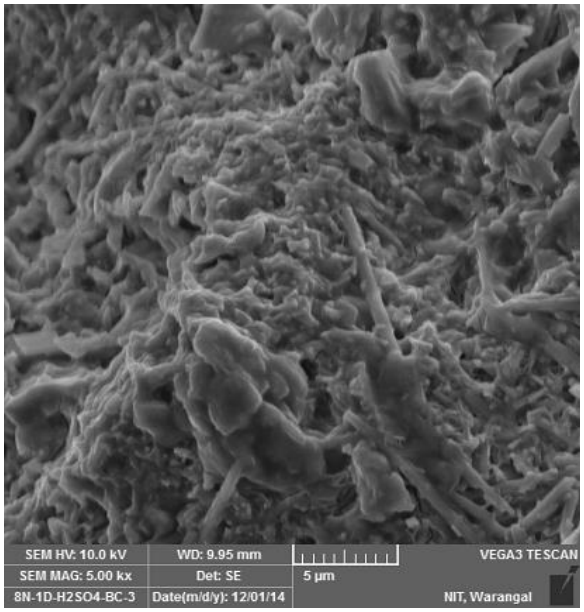

Figure 4 *SEM micrograph of black cotton soil contaminated with 8N H_2SO_4*

From SEM analysis it is clear that acids can show immediate effect on soil microstructure and the effect increases with increase in concentration of acid. To support the acid disintegration of original mineral leading to alteration in the microstructure, soil samples were subjected to EDAX analysis

5.2. EDAX Studies for 1-Day Curing Period

The elemental composition by EDAX analysis shows that soil mainly contains Si^{4+} and Al^{3+} followed by Fe^{2+} Mg^{2+}, K^+, Ca^{2+} and Na^+ (Table 2). Based on weight percent given in the table it can be stated that Al^{3+} with some Fe^{2+} and Mg^{2+} as octahedral cations and Ca^{2+}, K^+ and Na^+ as exchangeable interlayer cations. From EDAX analysis of soil exposed to 1N H_2SO_4 after 1 day curing showed that alumina has escaped from the lattice (wt. % of alumina decreased), which indicates that 1N H_2SO_4 attacked octahedral sheet with no effect on tetrahedral sheet. But EDAX analysis of soil when exposed to 4N H_2SO_4 showed a significant decrease in silica percent and a marginal decrease in alumina percent when compared to untreated soil. This indicates that soil when exposed to higher concentration of acid will attack even tetrahedral sheet and leaching out silica from the lattice [7]. The increase in alumina percent compared to 1N H_2SO_4 may be due to formation of new compounds with the leached alumina from the lattice. Further, EDAX analysis on soil exposed to 8N H_2SO_4 for 1 day showed that more than 50% {i.e (25.26-11.44)*100/25.26} of silica compared to untreated soil had leached out from the lattice indicating that tetrahedral sheet gets affected at higher acid concentration. Variation in alumina percent is due to escape of alumina from lattice and subsequent combination with anions forming new compounds.

Table 2: *EDAX analysis (wt. %) of untreated and treated Black Cotton Soil after 1 day*

Element	BC soil	1N H_2SO_4	4N H_2SO_4	8N H_2SO_4
O	55.65	58.3	65.4	65.64
Na	0.6	0.16	0.22	0.26
Mg	2.11	0.28	1.29	1.06
Al	9.59	1.52	6.02	4.98
Si	25.26	30.48	16.28	11.44
K	1.11	0.23	0.67	0.46
Ca	1.05	1.72	0.47	1.25
Fe	5.07	0.68	2.59	2.66

From EDAX analysis it is clear that soil when exposed to higher concentration of acid will attack both tetrahedral and octahedral sheets of the mineral leaching silica and alumina from the lattice, which subsequently may combine with other ions to form new compounds.

In order to verify the disintegration of the original minerals and formation of new compounds the samples were further subjected to XRD analysis

5.3. Mineralogical Studies for 1-Day Curing Period

The X-Ray diffraction analysis was performed to understand the mineralogical changes occurring in the soil samples. The fine-grained, oven-dried, treated and untreated soil samples were used for the analysis. Samples were scanned from 6^0 2θ to 70^0 2θ. The data was recorded and analyzed to identify mineral types by comparing the diffraction pattern of each sample with standard patterns given by X-pert High Score Plus Service.

The XRD patterns of untreated black cotton soil showed peaks with d-spacing values of 4.49, 2.56 and 1.50 Å, which are diagnostic peaks of volkonskoite mineral. However, other minerals like microline (peaks at 3.70, 3.24 and 2.15 Å), albite (peaks at 3.48, 1.97 and 1.81 Å), and quartz (peaks at 4.25, 3.34 and 1.81 Å) are also found. Generally, soils contain quartz since it is the most weather-resistant material [6].

The XRD patterns of black cotton soil contaminated with 1N sulphuric acid for a duration of 1day showed new peaks at 3.00, 2.80 and 1.84 A^0, which are characteristic peaks of bassanite. The black cotton soil contaminated with 4N sulphuric acid shows albite mineral (peaks at 3.19, 2.99 and 1.49 A^0) and soil contaminated with 8N sulphuric acid shows anhydrite (peaks at 3.49, 2.32 and 1.74 A^0). The comparison of patterns is shown in Fig. 5.

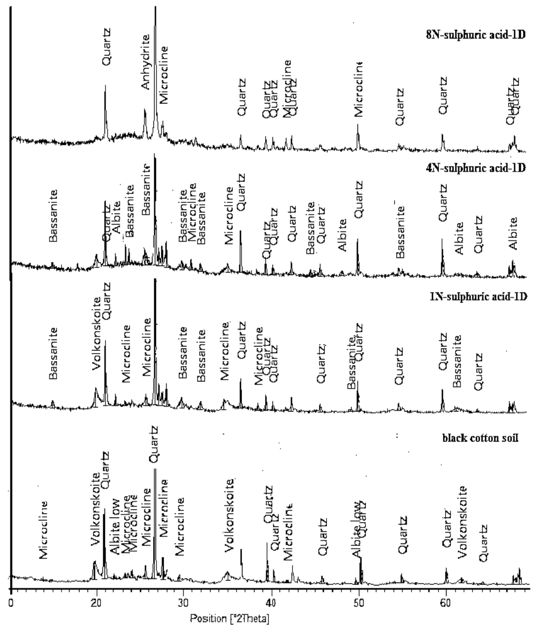

Figure 5 *X-ray diffraction patterns of black cotton soil before and after reaction with sulphuric acid of different concentrations for curing period of 1 day*

Based on above discussion, showing the acid effect for 1-day curing period, it can be concluded that acids shows immediate effect on the disintegration of original mineral and subsequently leading to formation of new compounds.

In order to understand the effect of curing period on mineralogical transformations, soil was exposed with different concentration for 14-days curing.

5.4. Mineralogical Studies for 14-Days Curing Period

XRD patterns of contaminated soil kept for 14 days curing period are shown in Fig. 6. XRD pattern of black cotton soil contaminated with 1N sulphuric acid show new peaks at 2.59, 1.84 and 1.45 A^0, which are indicative peaks of muscovite mineral. The black cotton soil contaminated with 4N sulphuric acid show qilianshanite mineral (peaks at 3.46, 2.28 and 1.98 A^0) and potassium-feldspar (peaks at 3.24, 2.23 and 1.67 A^0). Soil contaminated with 8N sulphuric acid shows 3 new peaks namely natromontebrasite (peaks at 3.14, 2.07 and 1.47 A^0); palygorskite (peaks at 2.23, 2.07 and 1.75 A^0) and taranakite mineral (peaks at 2.95, 2.12 and 1.98 A^0).

Thus by comparing the XRD pattern of original soil with XRD patterns of soil contaminated with different concentration for different curing periods as shown in Fig. 5 and Fig. 6, it can be observed that more number of new peaks were formed with increase in curing period. These mineralogical transformations due to the acid contamination may lead to unexpected behavior of soils [8, 9].

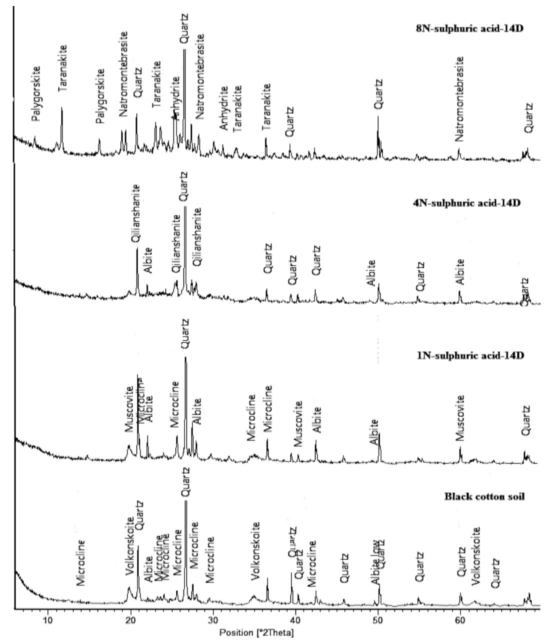

Figure 6 *X-ray diffraction patterns of black cotton soil before and after reaction with sulphuric acid of different concentrations for curing period of 14 days*

6. Conclusions

Based on the laboratory studies conducted on the black cotton soil using sulphuric acid as a contaminating fluid, the following conclusions are drawn.

1. Acid shows immediate effect on the soil leading to mineralogical and microstructural alterations. The effect increases with increase in concentration.
2. Soil when exposed to higher concentration of acid will leach both silica and alumina from the crystal lattice of tetrahedral and octahedral sheets respectively, which subsequently leads to formation of new minerals.
3. The formation of new minerals increases with curing period. These mineralogical transformations may lead to unexpected changes in geotechnical properties of soil.

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