# Sulfuric Acid Induced Volume Changes in Fly Ash Amended Soil

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#### Abstract

Sulfuric acid induced volume changes and consequent distress to the industrial structures has been reported in a number of cases in the past few decades. The exponential growth of costs required for repair of such structures necessitates a better understanding of soil behavior subjected to acid contamination to plan proper control measures. Thus, an attempt is made in the present study to evaluate the influence of sulfuric acid contamination on the volume change behavior of black cotton soil and to assess the potential of fly ash to control the induced volume changes. Formation of sulfate based minerals leads to an increase in swelling and a decrease in compressibility of soil during sulfuric acid contamination. Results of fly ash amended soils subjected to sulfuric acid contamination indicate a marginal reduction in swelling and compressibility than the original soil. The mineralogical and morphological studies indicated that the formation of sulphate minerals and consequent microstructural changes were similar in original soil and fly ash amended soil during sulfuric acid contamination. Based on the study, it can be concluded that the addition of fly ash did not show a supplementary advantage in soil prone to sulfuric acid contamination.

Keywords: Black cotton soil, Compressibility, Mineralogy, Morphology, Sulfuric acid, Swelling.

# **1. INTRODUCTION**

The impact of rapid industrialization around the globe and unsafe waste management practices has become increasingly significant within the last few decades. These anthropogenic activities mainly manifest as contamination of ground and surface water resources, and of foundation clays. Soil contamination due to accidental spills or leakages of industrial effluents, petroleum hydrocarbons, heavy metals and pesticides from industrial wastes are being increasingly recognized in the literature (Assa'ad 1998; Al-Omari et al. 2007; Jiang et al. 2014; Chavali et al. 2017). Chemical contamination of foundation soils, more specifically acid contamination and consequent distress to the industrial structures, have been reported in a number of cases in the recent past and is a problem likely to be encountered in areas of imminent industrial growth (Vronskii et al. 1978; Izbash et al. 1989; Isaev et al. 1995). Prediction of volume changes in such soils becomes more complicated as it involves complex chemical reactions. The exponential growth of costs required for repair of such structures necessitates identification of proper control measures. Thus, in this paper an attempt is made with fly ash to control sulfuric acid induced volume changes in black cotton soil.

# 2. MATERIALS AND METHODS

#### 2.1. Soil used

Natural black cotton soil from NIT Warangal campus was selected for this laboratory investigation. The black cotton soil can be classified as CH clay or fat clay with a liquid limit of 60% and a plastic limit of 18%. The fines content (< 75  $\mu$ m) was found to be 67% and the clay content (< 2  $\mu$ m) was

26%. X-ray diffraction analysis revealed the presence of montmorillonite, microcline, quartz and calcite minerals.

#### 2.2. Fly ash used

The fly ash used in this study is Class F fly ash obtained from National Thermal Power Corporation (NTPC), Ramagundam (India). The fly ash has a composition consisting of silica content= 63.99%, (silica +alumina +iron oxide) content = 92.7%, Calcium oxide = 1.71%, Magnesium oxide = 1.0%, Sulfuric anhydride = 0.73%, water and soluble salts = 0.04%, pH =10 and loss on ignition = 2.12%. Presence of quartz and mullite minerals was found in fly ash from X-ray diffraction analysis.

#### 2.3. Solutions used

The contaminants used in the study are 1N and 4N concentrations of sulfuric acid (98% assay).

### 2.4. Oedometer test

One-dimensional consolidation tests were conducted according to IS 2720-15 (1985) to investigate the effects of acid contamination on the swelling and compressibility of fly ash. Specimens of fly ash were then compacted in the oedometer cell (6 cm in diameter and 2 cm in height) to their maximum dry density to a height of 1.4 cm. The fly ash was allowed to swell at a seating load of 5 kPa before subsequent loading and unloading. All the samples were loaded to 640 kPa, with a standard load increment ratio of unity, and unloaded to 5 kPa in the same order as loading. The changes in thickness of the specimen were recorded against time to obtain the compression at the end of each load stage. As per conventional one-dimensional consolidation tests, the compressibility of the soil samples was expressed with reference to their compacted state. However, in the present study, the compressibility was assessed after reaching equilibrium swelling. Thus, modified compression index was used to evaluate compressibility. The modified compression index ( $C_{cm}$ ) is defined as the slope of the linear portion of the consolidation curve plotted on a logarithmic scale of pressure. Representative samples from the oedometer tests were evaluated with XRD and SEM.

### **2.5. X-Ray Diffraction studies**

PANanalytical X-ray diffractometer was used to assess the mineralogical variations in soil and fly ash. The samples were scanned between two theta values of  $6^{\circ}$  to  $70^{\circ}$  with a step size of  $0.02^{\circ}$ .

#### 2.6. Scanning electron microscopy studies

Morphological studies on soil and fly ash samples were carried out using TESCAN VEGA 3LMU microscope with conventional tungsten heated cathode having live stereoscopic imaging using 3D beam technology. The samples were coated with gold using a sputter coater prior to scanning.

### 3. RESULTS AND DISCUSSIONS

Figure 1 shows the effect of fly ash on the swelling and compressibility of black cotton soil subjected to sulfuric acid solutions. An increase in swelling of about 12% and 25% was observed in black cotton soil with 1N H<sub>2</sub>SO<sub>4</sub> and 4N H<sub>2</sub>SO<sub>4</sub>, respectively, than that of swelling in the black cotton soil with water (7%). An experimental swelling of about 11% and 23% was observed in fly ash (20%) treated black cotton soil with 1N H<sub>2</sub>SO<sub>4</sub> and 4N H<sub>2</sub>SO<sub>4</sub>, respectively. Addition of fly ash to black cotton soil resulted in marginal reduction in swelling with sulfuric acid solutions. A marginal reduction in compressibility was observed in fly ash treated black cotton soil with H<sub>2</sub>SO<sub>4</sub> solutions compared to untreated black cotton soil. The variations in the modified compression index of the fly ash treated black cotton soil with acid solutions are given in Table 1. The inadequacy of fly ash to control induced volume changes with sulfuric acid may be attributed to the susceptibility of fly ash itself to sulfuric acid contamination (Chavali and Reddy 2016).



Figure 1. Effect of fly ash on swelling and compressibility behavior of black cotton soil inundated with H<sub>2</sub>SO<sub>4</sub> solutions

Pore fluid	Modified compression index, C <sub>cm</sub>
Water	0.15
1N H <sub>2</sub> SO <sub>4</sub>	0.09
4N H <sub>2</sub> SO <sub>4</sub>	0.11
20% FA : 1N H <sub>2</sub> SO <sub>4</sub>	0.07
20% FA : 4N H <sub>2</sub> SO <sub>4</sub>	0.08

Table 1. Modified compression index of fly ash treated black cotton soil

XRD patterns of fly ash treated black cotton soil inundated with different pore fluids are shown in Figure 2. The black cotton soil with 1N H<sub>2</sub>SO<sub>4</sub> revealed peaks pertaining to new minerals *gypsum* and *bassanite* along with dissolution of calcite whereas black cotton soil with 4N H<sub>2</sub>SO<sub>4</sub> showed *alunogen* and *bassanite* minerals along with dissolution of calcite. The peak pertaining to *montmorillonite* transformed into *alunogen* with 4N H<sub>2</sub>SO<sub>4</sub>. The fly ash treated black cotton soil showed peaks pertaining to *anhydrite* and *bassanite* minerals with 1N and 4N H<sub>2</sub>SO<sub>4</sub>, respectively, along with *mullite*. The formation of *alunogen* mineral was restricted upon addition of fly ash. The mineral changes in fly ash treated soils were almost similar to untreated soils, which indicate fly ash ineffectiveness during sulfuric acid contamination.



Figure 2. Effect of fly ash on XRD patterns of black cotton soil inundated with H<sub>2</sub>SO<sub>4</sub> solutions



Figure 3. SEM micrographs of black cotton soil interacted with (a) water (b) 1N H<sub>2</sub>SO<sub>4</sub> (c) 4N H<sub>2</sub>SO<sub>4</sub> (d) 20% FA: 1N H<sub>2</sub>SO<sub>4</sub> and (e) 20% FA: 4N H<sub>2</sub>SO<sub>4</sub>

Micrographs of fly ash treated black cotton soil contaminated with different pore fluids were shown in Figure 3. Black cotton soil transformed from undulating film like structure to *lath like microstructure* and *needle like microstructure* upon contamination with sulfuric acid. The micrographs of fly ash treated black cotton soil showed microstructures similar to that of untreated soil with sulfuric acid except the presence of *spherical microstructures*. This clearly supporting the mineralogical variations observed in fly ash treated soil with sulfuric acid solutions.

# 4. CONCLUSIONS

- 1. Fly ash treated soils contaminated with sulfuric acid exhibited marginal reduction in swelling and compressibility.
- 2. Fly ash treated soil exhibited formation of new minerals such as *bassanite, anhydrite* and *mullite* minerals which were found even in untreated soils upon sulfuric acid contamination.
- 3. The morphological changes observed in fly ash treated soils were almost similar to that of untreated soils except the additional *spherical microstructure*.

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