Control of phosphoric acid induced volume change in clays using fly ash

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Abstract. Volume changes of soils induced by inorganic acids cause severe foundation and superstructure failures in industrial buildings. This study aimed to assess the potential of fly ash to control volume changes in soils under acidic environment. Two soils such as black cotton soil predominant with montmorillonite and kaolin clay predominant with kaolinite were used for the present investigation. Both soils exhibited an increase in swelling subjected to phosphoric acid contamination. Ion exchange reactions and mineralogical transformations lead to an increase in swelling and a decrease in compressibility in black cotton soil, whereas phosphate adsorption and mineral dissolution lead to an increase in swelling and compressibility in case of kaolin clay. Different percentages of Class F fly ash obtained from Ramagundam national thermal power station were used for soil treatment. Fly ash treatment leads to significant reduction in swelling and compressibility, which is attributed to the formation of aluminum phosphate cements in the presence of phosphoric acid.

Keywords: black cotton soil; contamination; fly ash; kaolin clay; phosphoric acid; volume change

1. Introduction

Fly ash is commonly considered to be a waste material resulting from combustion of powdered coal in thermal power stations (Zhou et al. 2002). Fly ash is extracted from the flue gases by electrical precipitation. Fly ash may be used as a long-term exploitable resource to mitigate the problem of environmental concerns over safe disposal (Pei et al. 2015). Currently, worldwide production of fly ash is estimated to exceed 750 million tons per year in which less than 50% of the fly ash produced being used (Izquierdo and Querol 2012, Ram and Masto 2014). The quantity of fly ash production is expected to reach around 400 million tons in India by the year 2017 (Haque 2013, Saride and Dutta 2016). In India, fly ash is utilized as a filler material, a raw material in cement, an admixture in cement concrete, a raw material for brick and geo-polymer production and a stabilizer for soil and pavement construction.

Several research studies have confirmed the potential of fly ash in the stabilization of soils (Sivapullaiah *et al.* 1996, Phani Kumar and Sharma 2004, Misra*et al.* 2005, Kumar *et al.* 2007, Rekha *et al.* 2016, Mohanty *et al.* 2017). Fly ash enhances the strength of soil by pozzolanic reaction (Phanikumar 2009) and reduces swelling, shrinkage and plasticity by flocculation and agglomeration of clay particles (Nicholson and Kashyap 1993). However, the pozzolanic reaction takes place slowly, which lead to slow gain in strength, whereas flocculation is an immediate reaction which results in quick reduction in swell-shrink behavior (Indraratna *et al.* 1991, Zha *et al.* 2008). The field performance of expansive sub-grade soil also gets improved considerably with fly ash treatment (Zia and Fox 2000, Arora and Aydilek 2005, Parsons and Kneebone 2005, Bin-Shafique *et al.* 2010). The micro level analysis confirmed that, pozzolanic reaction dominates over the cation exchange capacity in the stabilization process (Sharma *et al.* 2012). Fly ash has also been used to mitigate the adverse effect of alkali on soil swelling (Sivapullaiah *et al.* 2008, Sivapullaiah and Reddy 2009, Reddy and Sivapullaiah 2011, Vindula *et al.* 2016). However, its utilization to control acid induced heaving in soils is scanty.

The growing rate of acid contamination of soils and consequent effects of failures of foundations and super structures (Vronskii et al. 1978, Sridharan et al. 1981, Stephenson et al. 1989, Izbash et al. 1989, Joshi et al. 1994, Isaev et al. 1995, Shekhtman et al. 1995, Rao and Reddy 1997, Assa'ad 1998, Al-Omari et al. 2007, Sivapullaiah et al. 2009), emphasizes the need to identify proper mitigation measures to control acid induced heave problems. Lukas and Gnaedinger (1972) were probably the first to report failure of foundations of industrial buildings as a result of contamination. They found failures of two buildings were due to acid spillage and one building was due to alkali spillage. The chemical contamination resulted in dissolution of either limestone in glacial till or high silica sand which was found to be the cause of large settlements in each case. Coming to the Indian scenario, Sridharan et al. (1981) presented the distress to the floors, beams and upheaval of foundations in a fertilizer plant due to the heaving of subsoil. Laboratory investigations revealed that phosphoric acid contamination of subsoil resulted in formation of insoluble phosphate salts which lead to subsoil heaving. More recently, Al-Omari et al. (2007) investigated the cyclic heave and settlement of isolated footings in

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Property	Black	Kaolin	Fly	Standards		
	cotton soll	clay	asn			
Specific gravity	2.63	2.65	2.17	IS 2720 Part 3		
Liquid Limit (%)	60	52	-	IS 2720 Dout 5		
Plastic Limit (%)	18	27	-	15 2720 Part 5		
Plasticity Index (%)	42	25	-	-		
Clay, <2 µm (%)	26	21	3			
Silt, 75 to 2 μ m (%)	41	79	83	IS 2720 Part 4		
Fine sand, 425 to 75 μm (%)	33	-	14	15 2,20 T uit 1		
Soil classification	CH	CL	-	IS 1498		
Cation exchange capacity (meq/100g)	37.5	5.62	-	Birand and Cocka(1993)		
Maximum dry density (kN/m ³)	16.5	14.8	12.4	IS 2720 Dowt 7		
Optimum moisture content (%)	16.6	27.3	25.1	15 2720 Part 7		

Table 1 Physical properties of materials used

phosphate fertilizer complex in the western Iraqi city of AI-Kaim. Laboratory deformation tests suggested that the leakage of phosphoric and sulfuric acids into the limestone bedrock with subsequent interaction between calcium carbonate in the limestone and the acids generated volume instability to the bedrock. In addition to these cases, during 2015-2016, more than 30 cases of accidental spillages of acids took place on highways and railways during transportation (SAW 2017). These cases further enhance the need to arrest the complex nature of acid induced heave problem.

Recently, Mahyar and Erdogan (2015) recognized the potential of phosphoric acid activated fly ash to produce acid-base cements. Thus, an attempt has been made in the present study to control the phosphoric acid induced volume changes in soils with class F fly ash obtained from National Thermal Power Corporation (NTPC), Ramagundam (India).

2. Materials and methods

2.1 Soils and pore fluids used

Two soil samples such as black cotton soil (BCS) from NIT Warangal campus and commercially available kaolin clay (KT) were selected for the study. The cation exchange capacity of natural soil and kaolin clay is 37.5 and 5.6 meq/100g respectively. The physical properties and chemical composition of soils are presented in Table 1 and Table 2. The chemical composition of soils was evaluated with the help of x-ray fluorescence (XRF) spectroscopy technique. The presence of *montmorillonite*, *quartz*, *calcite* and *microcline* minerals in black cotton soil, similarly kaolinite, calcite and ankerite minerals in kaolin clay were confirmed by X-ray diffraction analysis. From the previous studies conducted by the authors, it was found that 4N phosphoric acid has a very severe effect on the volume change behavior of soils (Chavali and Ponnapureddy 2017). Thus, distilled water and 4N phosphoric acid (Assay 88%) solutions were used as pore fluids to simulate those conditions.

Table 2 Chemical composition of materials used

			1								
Soil	SiO_2	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P_2O_5	MnO	SO_3
Black											
cotton	43.6	22.8	14.7	7.3	7.5	0.255	0.63	1.71	0.28	0.47	0.14
soil											
Kaolin	34.5	49.0	6.5	2.3	5.3	0.034	0.17	1.22	0.15	0.17	0.11
clay				~ -							
Fly ash	21.9	50.3	17.2	8.7	0.5	0.8	1.03	2.60	-	0.65	-

2.2 Fly ash

The fly ash (FA) used in this study is obtained from National Thermal Power Corporation (NTPC), Ramagundam (India). The physical properties and chemical composition of fly ash are presented in Table 1 and Table 2. Based on its chemical composition, it can be classified as Class F fly ash. Loss on ignition of fly ash is 2.12 %. The X-ray diffraction pattern confirms the presence of *quartz, mullite* and *calcite* minerals.

2.3 One-dimensional consolidation test

The one-dimensional consolidation tests were performed according to IS 2720-15 (1985) to examine the consequences of phosphoric acid contamination on the volume change behavior of fly ash treated soils. Special Teflon consolidation rings were made, which were entirely non-reactive to acid. Fly ash treated soils was mixed thoroughly with distilled water at optimum moisture content, which were sealed in a plastic bag, and kept in desiccator for 24 hours to attain uniform moisture content. Samples were then compacted in the consolidation cell (6 cm in diameter and 2 cm in height) to their maximum dry density to a height of 1.4 cm. Care was taken to minimize the errors due to side friction and air entrapment. The prepared fly ash treated soil specimens were inundated with phosphoric acid solutions as pore fluid and allowed to swell at a seating load of 5 kPa to reach equilibrium. The volume of the pore fluid used for all specimens was about 500 ml and testing was carried out at room temperature of 27°C. The loads were subsequently increased to 10, 20, 40, 80, 160, 320 and 640 kPa (with a standard load increment ratio of unity from 5 kPa onwards) and later in a similar manner unloading was carried out. The obtained results were compared with natural soil inundated with distilled water and phosphoric acid. In a significant number of cases, the experiments were repeated to check the reproducibility of results. The changes in thickness of the specimen were recorded against time to obtain the swelling at the seating load, and the compression at the end of each load stage.

2.4 X-ray diffraction test

Mineralogical composition of samples was determined using PANanalytical X-ray diffractometer. At the end of each consolidation test representative soil samples were collected. The representative samples collected at the end of consolidation tests were oven dried for 24 hours. The dried samples were scanned between two theta values of 6° to 70° with a step size of 0.02° . The X-Ray Tube was operated at



Fig. 1 Swelling behavior of fly ash treated black cotton soil contaminated with phosphoric acid

60 kV and 55 mA using an X'Celerator ultra fast detector. Qualitative identification of minerals was conducted using X'pert high score plus software based on database provided by Joint Committee of Powder Diffraction Data Service (PCPDFWIN 1999).

2.5 Scanning electron microscopy

Scanning electron microscopy was conducted to evaluate the microstructural changes occurring in the samples due to acid contamination. TESCAN VEGA 3LMU microscope with conventional tungsten heated cathode having live stereoscopic imaging using 3D beam technology was used for SEM analysis. The soil specimens were mounted onto the tape glued to the flat surface of SEM stub and sputter coated with gold prior to scanning. Numerous SEM images of treated and untreated soil were taken at different magnifications and the micrographs shown are those with 2 μ m magnification that best reveal the unique microstructure of the clay due to contamination.

3. Results and discussions

The volume change behavior of soils was discussed in three phases (1) natural soils inundated with distilled water (2) natural soils subjected to phosphoric acid contamination and (3) soil treated with fly ash subjected to phosphoric acid contamination.

3.1 Consolidation test results of black cotton soil

The swelling and compressibility results of black cotton soil contaminated with phosphoric acid solution were shown in Figs. 1 and 2. Black cotton soil has shown an equilibrium swelling of about 7% and 14% with water and phosphoric acid respectively. It can be observed that the soil inundated with both water and phosphoric acid exhibited swelling whereas soil with phosphoric acid shown more swelling than with water. The black cotton soil with montmorillonite mineral generally shows high swelling and compressibility at nominal load. However, in the present study, the amount of swelling exhibited by black cotton soil



Fig. 2 Compressibility behavior of fly ash treated black cotton soil contaminated with phosphoric acid

is comparatively less, which may be due to the presence of calcite (calcium carbonate) and microcline (potassium aluminium silicate) minerals. During interaction of black cotton soil with phosphoric acid, hydrogen ions in the pore fluid would replace the exchangeable cations from the double layer. These exchange reactions enhances the clay fabric to more flocculated one. The released cation interacts with the phosphate anions in the solution leads to acid phosphate mineral formations. An increase in swelling and a decrease in compressibility with phosphoric acid are attributed to these exchange reactions and mineral transformations.

In order to reduce the swelling exhibited by soil with phosphoric acid three different percentages (20, 30 and 40) of fly ash were used. It is well known that the amount of fly ash required to improve the soil properties must be either greater than 80% or less than 50% (Sivapullaiah and Reddy 2009). Thus, 20, 30 and 40 percentage of fly ash was chosen. It can be observed that the fly ash treated soil contaminated with phosphoric acid exhibited a decrease in swelling and compressibility than without treatment. An equilibrium swelling of about 6%, 4% and 3% was observed in black cotton soil with 20%, 30% and 40% fly ash, respectively. It can be seen that the higher the amount of fly ash, the higher the reduction in swelling. The swelling and compressibility reduction may not be fully due to the decrease in the clay content alone with the addition of high amounts of fly ash. The reduction in swelling and compressibility is partially due to arresting the formation of expansive mineral (Brushite) and mainly attributed to synthesis of cementing phosphates (Berlinite) as confirmed from XRD analysis.

3.2 X-ray diffraction test results of black cotton soil

X-ray diffraction patterns of fly ash treated black cotton soil contaminated with phosphoric acid were shown in Fig. 3. Black cotton soil contains montmorillonite, quartz and microcline as major minerals along with calcite. Phosphoric acid contaminated soil exhibited new peaks pertaining to brushite mineral with dissolution of calcite. It is well known



Fig. 3 Mineralogical changes in fly ash treated black cotton soil contaminated with phosphoric acid

that calcite quickly dissolves in inorganic acids with the liberation of carbon-di-oxide.

$$CaCO_3 + H_3PO_4 + H_2O \rightarrow CaHPO_4 \bullet 2H_2O + CO_2 \uparrow (1)$$

The above mentioned reaction produces a theoretical volume increase of about 120% (Joshi *et al.* 1994). Thus, it is reasonable to establish that, along with cation exchange reactions, formation of brushite mineral lead to an increase in swelling.

On the other hand, black cotton soil treated with 20% and 30% fly ash showed new peaks pertaining to mullite and cristobalite. Similarly, new peaks pertaining to mullite and berlinite were seen in soil with 40% fly ash. The peaks of cristobalite and berlinite were confirmed by d(001)values. Mullite is a predominant mineral in fly ash. Mullite is the only stable intermediate phase in the alumina-silica system at atmospheric pressure, which has no charge balancing cations (Duval et al. 2008). Berlinite is an anhydrous form of aluminium phosphate cements, isostructural with silica and has framework structures similar to zeolites. Berlinite rapidly converts into cristobalite form at low temperatures (Morris et al. 1977). The temperature-stability relationships of the aluminium phosphate polymorphs studied by Morris et al. (1977) using differential thermal analysis revealed the following scheme of transformations:

	$AlPO_4$					
<i>berlinite</i> \leftarrow^{108}	$\xrightarrow{8 \pm 4 \text{ K}} tridymite - form \xleftarrow{1298 \pm 5 \text{ K}}$	cristobalite – form				
$\beta \xleftarrow{446 \text{ K}} \alpha$	$\beta \xleftarrow{366 2 \times K}{\alpha 1} \xleftarrow{403 K}{\alpha 2}$	$\beta \xleftarrow{4832 \times K} \alpha$				

These mineral transformations and formation of aluminium phosphate cements supporting the reduction in swelling and compressibility of fly ash treated soils during phosphoric acid contamination.

3.3 Scanning electron microscopy test results of black cotton soil

The scanning electron microscopy tests were carried out to assess the role of microstructure on the volume change behavior of fly ash treated black cotton soil subjected to



Fig. 4 Morphological changes in fly ash treated black cotton soil contaminated with phosphoric acid



Fig. 5 Swelling behavior of fly ash treated kaolin clay contaminated with phosphoric acid

phosphoric acid contamination. Micrographs of black cotton soil contaminated with phosphoric acid solution are shown in Fig. 4. It can be seen that soil showed dispersed undulating filmy particle microstructure (Fig. 4(a)), which confirms the presence of montmorillonite mineral (Latifi et al. 2016). Black cotton soil subjected to acid contamination exhibited more void spaces along with flower-like brushite structures (Brundavanam et al. 2014). These morphological changes further supporting the increase in swelling with acid (Fig. 4(b)). In contrast, fly ash treated soil exhibited more dense clay particles (Figs. 4(c)-4(e)) along with smooth spherical cenospheres (Prasad and Reddy 2016). Cenosphere is an extreme stable material and does not absorb water and resistant to most of the acids (Sear 2001). The cementitious mineral formations were validated by these morphological changes.

3.4 Consolidation test results of kaolin clay

The variations in the swelling and compressibility of kaolin clay subjected to phosphoric acid solution were shown in Figs. 5 and 6. Kaolin clay exhibited almost noswelling with water. The non-swelling nature of kaolin clay



Fig. 6 Compressibility behavior of fly ash treated kaolin clay contaminated with phosphoric acid

is well known as the hydrogen bonding between two adjacent layers do not allow cations and water to enter between the structural layers. An increase in equilibrium swelling of about 13% was observed in kaolin clay with phosphoric acid. The compressibility of kaolin clay also increased upon contamination with phosphoric acid. The increase in percentage of swelling exhibited by kaolin clay with phosphoric acid is attributed to fast ligand replacement of hydroxyl ion by phosphate ion and subsequent formation of flocculated fabric. The size of the phosphate ion being larger than the hydroxyl ion, each particle becomes bigger on replacement, which leads to increase in volume. Dissolution of calcite leads to an increase in compressibility behavior of kaolin clay with phosphoric acid contamination. Adsorption of phosphate anions and dissolution of calcite can be evidenced from mineralogical and morphological transformations.

Reduction in swelling and compressibility can be observed in fly ash treated kaolin clay contaminated with phosphoric acid than untreated soil. An equilibrium swelling of about 11%, 9% and 8% was observed in kaolin clay with 20%, 30% and 40% fly ash, respectively. The non-swelling nature of fly ash subjected to phosphoric acid contamination was reported by Prasad and Reddy (2016). The reduction in swelling and compressibility is similar to the trend shown by black cotton soil. The addition of higher amount of fly ash resulted in higher reduction in swelling. The reduction in swelling and compressibility is partially attributed to arresting the formation of metal phosphate mineral (Sarcopside) and mainly due to synthesis of cementing phosphates (Berlinite) which can be evident from XRD analysis.

3.5 X-ray diffraction test results of kaolin clay

Fig. 7 shows the X-ray diffraction patterns of fly ash treated kaolin clay contaminated with phosphoric acid. Kaolin clay predominantly consists of kaolinite along with a peak pertaining to each calcite and ankerite. It is well known that tropical soils were preponderant with aluminum along with iron. Phosphoric acid interaction with aluminium



Fig. 7 Mineralogical changes in fly ash treated kaolin clay contaminated with phosphoric acid



Fig. 8 Morphological changes in fly ash treated kaolin clay contaminated with phosphoric acid

and iron available in the soil leads to formation of highly insoluble and hard aluminum phosphate or iron phosphate minerals. Kaolin clay subjected to phosphoric acid showed peaks pertaining to sacropside mineral with dissolution of ankerite and calcite minerals. Sarcopside is an iron phosphate mineral. The reaction between iron oxide in the clay and phosphoric acid may lead to the formation of sarcopside mineral. Fast ligand replacement of hydroxyl ion by phosphate ion, further ensures the formation of sarcopside mineral.

On the other hand, kaolin clay treated with 20%, 30% and 40% fly ash showed new peaks pertaining to mullite and berlinite. The peaks of mullite and berlinite were confirmed by d(001) values. The berlinite mineral formation and its cementitious prowess are already stated earlier. These mineral transformations and formation of aluminum phosphate cements lead to a reduction in swelling and deformation behavior of fly ash treated kaolin clay subjected to phosphoric acid contamination.

3.6 Scanning electron microscopy test results of kaolin clay

Scanning electron micrographs of fly ash treated kaolin

clay subjected to phosphoric acid contamination were shown in Fig. 8. It can be seen that kaolin clay showed *layered crystalline structures* (Fig. 8(a)) that appeared much like pages in a book with *hexagonal crystal* (Brady and Well 1984). Kaolin clay contaminated with phosphoric acid revealed flocculated *face-to-edge structures/flower like structures* (Fig. 8(b)). These changes confirm the effect of acid contamination on mineral structure and formation of new minerals. Fly ash treated kaolin clay showed more aggregated dense particles along with an increased amount of spherical particles (Figs. 8(c)-8(e)). The spherical particles increased as the amount of fly ash in kaolin clay increased. These morphological changes support the volume changes in kaolin clay subjected to phosphoric acid contamination.

5. Conclusions

The potential of fly ash to stabilize the soils subjected to phosphoric acid has been evaluated in the study and the conclusions are as follows:

- Acid induced volume changes are manifested by both montmorillonitic and kaolinitic soils.
- Formation of calcium phosphate and aluminum phosphate minerals due to cation exchange reactions and mineral dissolution lead to volume changes in soils.
- Severe morphological changes are observed in black cotton soil contaminated with acids followed by kaolin clay. The transformation of clay original microstructure to flower like microstructures is typified by new mineral formations.
- Reduction in volume changes in phosphoric acid contaminated soils are attributed to the presence of cenospheres and formation of cementitious aluminum phosphates.

• Though the obtained results showed fly ash could be used as a stabilizing material under acid condition, further studies need to be carried out to completely understand the potential of fly ash to control acid induced volume changes in soils.

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