BET surface area of phosphoric acid treated tropical soils

Amin Eisazadeh^a, Khairul Anuar Kassim^a, Hadi Nur^b

^a Geotechnic & Transportation Department, Faculty of Civil Engineering, Universiti Teknologi Malaysia

^b Ibnu Sina Institute for Fundamental Science Studies, Universiti Teknologi Malaysia, 81310 UTM Skudai, Malaysia

* Corresponding author. Tel.: +60177080315; fax: +6075566157. E-mail address: A.Eisazadeh@yahoo.com.

Abstract

The specific surface area is an important property in assessing the physical interaction of clay particles with additives. In this paper, the time-dependent changes induced in surface area of phosphoric acid treated tropical soils comprised mainly of kaolinite mineral were investigated. Comparison between the BET results indicated that the effects of acidic stabilizer on the surface area of soil particles were significant. Furthermore, it was found that the presence of free iron oxides in the form of micro-aggregates on the surface of soil particles contributed to achieving higher surface area values while limiting the attack of phosphoric acid on the clay particles.

Key words

Laterite clay, Kaolin soil, Acid stabilization.

Introduction

The specific surface area is an important characteristic in determining the chemical and physical interaction of the soil with its surroundings. This is due to the fact that most of the chemical reactions in soils take place at the surface of particles (Mitchell and Soga 2005). Furthermore, the surface area developed by soil minerals is related to many other soil properties such as ion exchange capacity, reactivity, and etc. Therefore, the measurement of this property has received much attention during the past decades (Aylmore 1974).

The tendency of all solid surfaces to attract surrounding gas molecules gives rise to a process called gas sorption. Physisorption is the most common type of adsorption. Physisorbed molecules are fairly free to move around the surface of the sample. As more gas molecules are introduced into the system, the adsorbate molecules tend to form a thin layer that covers the entire adsorbent surface. One can estimate the number of molecules (N_m) required to cover the adsorbent surface with a monolayer of adsorbed molecules. Multiplying N_m by the cross sectional area of an adsorbate molecule yields the sample's surface area. One of the most common methods for determining the surface area of finely divided materials is that of Brunauer, Emmett and Teller (Brunauer *et al.* 1938), known as the BET method. The method is based on collecting isotherm data for the physical adsorption of an inert gas and modeling the adsorption data using the BET isotherm equation.

Throughout these years considerable research has been carried out in studying the effect of traditional stabilizers such as lime on various properties of soil (Locat *et al.* 1990; Bell 1996; Narasimha Rao and Rajasekaran 1996). However, the use of phosphoric acid as a stabilizer has been limited.

Materials and experimental programme

Materials

Two soil types formed under extreme weathering conditions were chosen for this study. The first type was the slightly acidic White Kaolin (WK) sample. The soil was dominated by kaolinite and illite minerals along with minor constituents of quartz. Also, a reddish Laterite Clay (LC) rich in iron oxide excavated from a hillside located in Southern part of Malaysian Peninsular was used for this investigation. The physical properties and chemical composition of the untreated samples are presented in Table 1. It should be noted that the phosphoric acid used in this research was a Merck analysed, 85% H₃PO₄, of specific gravity 1.71.

Physical properties	values		Chemical	values (%)	values (%)	
	Laterite clay	White kaolin	(oxides)	Laterite clay	White kaolin	
pH (L/S = 2.5)	4.86	4.34	SiO ₂	21.55	48.18	
External surface area (m ² /g)	57.89	19.87	Al ₂ O ₃	24.31	31.10	
Liquid Limit, LL (%)	75.8	42.20	Fe ₂ O ₃	29.40	1.03	
Plastic Limit, PL (%)	39.60	21.30	MgO	-	0.86	
Plasticity Index, PI (%)	36.20	20.90	Na ₂ O	0.07	-	
IS Classification	MH	CL	CO ₂	3.65	1.34	
ICL (%)	5	2	P_2O_5	16.71	9.37	
Maximum dry density (Mg/m ³)	1.33	1.64	K ₂ O	0.11	4.01	
Optimum moisture content (%)	34.00	18.40	SO ₃	3.98	2.07	

Preparation of specimens

According to the previous studies conducted on phosphoric acid stabilization of clayey soils, different amounts of phosphoric acid were selected for each individual soil (Demirel *et al.* 1962; Medina and Guida 1995). Samples were then compacted into a thin wall PVC tubes (50 mm diameter \times 100 mm length) under constant compactive effort as specified in clause 4.1.5 of BS 1924: Part2. They were sealed to the atmosphere with rubber tight lids and stored in a room with constant temperature (27±2°C) until being tested at 1 month, 4 months, and 8 months curing period.

Testing programme

The surface area value was obtained by physical adsorption of nitrogen gas using Quantachrome Autosorb-1 surface area analyzer. Hence, approximately 0.15g of the cured sample was deposited into the sample holder. After degassing for 20hr at 300°C, nitrogen gas was injected and the surface area value was calculated using the multipoint BET method.

Results and discussion

As shown in Figure 1, the acid treated White Kaolin samples revealed a sharp reduction in the surface area at the early stages of curing. This was caused by the ion exchange reactions between H⁺ ions introduced by the stabilizer and the kaolinite mineral exchange sites which rendered a material with larger particles. Furthermore, the acid treated samples retained their low surface area values for all curing periods. The latter was consistent with the transformation of the natural soil to a completely new material. On the other hand, in Laterite Clay samples, based on the BET results, it was apparent that the presence of iron oxides as part of soil's secondary constituents, contributed to obtaining higher surface area values (Feller *et al.* 1992). Furthermore, the coating action of free iron oxides impeded the attack of acid on clay particles. As can be seen in Figure 2, the free oxides present in the soil environment have bonded the soil particles together, whereas, the neatly arranged book-like kaolinite particles were the predominant feature of the natural White Kaolin soil.



Figure 1 BET results for phosphoric acid treated White Kaolin (WK) and Laterite Clay (LC)



Figure 2 FESEM of untreated (a) White Kaolin and (b) Laterite Clay soil

Conclusions

This research was carried out in an attempt to further elucidate the effects of acidic stabilizers on soil's physical properties. In general, at the early stages of the curing, the substitution of exchangeable ions with H^+ introduced by the stabilizer lowered the surface area values for kaolinitic soils. However, in Laterite Clay samples due to the fact that clay particles were heavily coated and protected by iron oxides, these changes were rather limited. Also, it was found that the presence of free iron oxides in the form of micro-aggregates contributed to achieving higher surface area values.

References

Aylmore LAG (1974) Gas sorption in clay mineral systems. *Clays and Clay Minerals* **22**, 175-183. Bell FG (1996) Lime stabilization of clay minerals and soils. *Engineering Geology* **42**, 223-237. British Standards Institution (1990) Stabilized materials for civil engineering purposes: Part 2 Methods

- British Standards Institution (1990) Stabilized materials for civil engineering purposes: Part 2, Methods of test for cement-stabilized and lime-stabilized materials. London.
- Brunauer S, Emmett PH, Teller E (1938) Adsorption of gases in multimolecular layers. *Journal of American Chemical Society* **60**, 309-319.
- Demirel T, Benn CH, Davidson DT (1962) Use of Phosphoric Acid in Soil Stabilization. Highway Research Board Bulletin No. 282, 38-58.
- Feller C, Schouller E, Thomas F, Rouiller J, Herbillon AJ (1992) N₂-BET Specific Surface Areas of Some Low Activity Clay Soils and their Relationships with Secondary Constituents and Organic Matter Contents. Soil Science Journal 153, 293-299.
- Locat J, Berube MA, Choquette M (1990) Laboratory investigations on the lime stabilization of sensitive clays: shear strength development. *Canadian Geotechnical Journal* **27**, 294-304.
- Medina J, Guida HN (1995) Stabilization of Lateritic soils with phosphoric acid. *Journal of Geotechnical and Geological Engineering* **13**, 199-216.

Mitchell JK, Soga K (2005) 'Fundamentals of Soil Behavior.' (John Wiley and Sons: New York)

Narasimha Rao S, Rajasekaran G (1996) Reaction products formed in lime-stabilized marine clays. *Journal* of Geotechnial Engineering **122**, 329-336.

 $^{^{\}odot}$ 2010 19th World Congress of Soil Science, Soil Solutions for a Changing World 1 – 6 August 2010, Brisbane, Australia. Published on DVD.