Engineering Geology 162 (2013) 14–21

Contents lists available at SciVerse ScienceDirect

Engineering Geology

journal homepage: www.elsevier.com/locate/enggeo

Technical Note

Application of nanoindentation to establish influence of heat on soils

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ARTICLE INFO

Article history:
Received 26 December 2012
Received in revised form 20 April 2013
Accepted 2 May 2013
Available online 19 May 2013

Keywords:
Nanoindentation
Hardness
Residual modulus
Sands
Fine-grained soils
Thermal energy

ABSTRACT

In the scenario of rapid industrialization and infrastructural development, many situations are encountered where in soil like bentonite is subjected to elevated temperatures (about 200 °C). This may lead to alteration in the mechanical and engineering characteristics (such as hardness and residual modulus) of the soil. In this context, these characteristics can be quantified by measuring deformation of individual soil grains exposed to different elevated temperatures, by employing nanoindentation. With this in view, an attempt was made to explore the potential of this technique, normally used for material characterization by material scientists to study heat induced alteration in mechanical and engineering characteristics of the metals, to study the behavior of soils when they get exposed to elevated temperatures. As such, soils of entirely different characteristics were exposed up to 200 °C (in steps of 50 °C), nanoindentation studies were conducted on the residues and the results are reported in this technical note. It has been observed that there is a significant change in the hardness, residual modulus and resistance to indentation of the soils due to their exposure to elevated temperatures. It has also been demonstrated that the effect of elevated temperature is more pronounced on fine-grained soils as compared to coarse-grained soils.

1. Introduction

The present day geotechnical engineering related projects deal with laying of buried power supply cables and air condition ducts (Gangadhara and Singh, 1999), ground modification or stabilization techniques using chemicals and thermal treatment (Ma and Hueckel, 1992; Alcocer and Chowdhury, 1993; Akinmusuru, 1994; Joshi et al., 1994; Yang and Farouk, 1995; Krishnaiah and Singh, 2006), disposal of high level radioactive (Varlakov et al., 1997) and industrial toxic wastes (Farag, 1993), designing foundations for the furnaces (Li et al., 2011), boiler units, forging units, brick kilns, rocket launching pads, volcanic eruptions, underground explosions, etc. In such situations the soil gets exposed to elevated temperatures (about 200 °C), which might result in particle breakage and alteration of surface characteristics such as physical (changes in the specific gravity, Yilmaz, 2011; specific surface area and particle size, Utkaeva, 2007), chemical (variation in CEC, pH, EC; Parlak, 2011) and mineralogical (Ghuman and Lal, 1989; Certini, 2005; Hatten et al., 2005). Furthermore, changes in these properties would also influence their engineering behavior (such as hardness and residual modulus). Hence, it becomes essential to quantify and analyze, accurately, the influence of elevated temperatures on the soil, which is a conglomerate of various grains or particles (Lado and Ben-Hur, 2004). However, due to lack of understanding related to such exposure conditions of the soil, proper instrumentation and analytical techniques, little efforts have been made in the past to understand and measure variations in the behavior of the soil when it is exposed to elevated temperatures. Incidentally, nanoindentation technique appears to be a panacea for establishing changes in mechanical properties of soils under these circumstances and it has been observed that the potential of nanoindentation has been exploited in the various fields of material characterization by material scientists [viz., determination of residual modulus, hardness (Doerner and Nix, 1986; Oliver and Pharr, 1992; Field and Swain, 1993, 1995; Swain, 1998); cracking, phase transformations, creep and energy absorption (Fischer-Cripps, 2004); hardness and modulus of ultrathin films and coatings (Hakiri et al., 2009; Han and Joost, 2009); hardness and modulus of minerals (Dutta and Penumadu, 2007; Bathija, 2009; Wei, 2009; Zhang et al., 2009); quantifying fracture toughness and interlayer adhesion of semiconductors (Volinsky et al., 2003); mechanical properties and creep behavior of lyocell fibers and concrete (Lee et al., 2007; Vandamme and Ulm, 2009); mechanical properties of shale (Ulm and Abousleiman, 2006; Ulm et al., 2007; Bobko and Ulm, 2008; Ortega et al., 2010; Deirieh et al., 2012); mechanical properties of concrete (Dejong and Ulm, 2007; Ulm et al., 2007; Miller et al., 2008; Vandamme and Ulm, 2009)]. These studies also indicate that nanoindentation technique has been widely used in the field of metallurgical engineering to evaluate material performance.

Though, in this context earlier researchers (Dutta and Penumadu, 2007; Bathija et al., 2009; Penumadu et al., 2009; Wei, 2009; Zhang et al., 2009) have demonstrated application of nanoindentation in geomechanics successfully, this technique has not yet been employed to establish changes undergone by the soil when it gets exposed to

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0013-7952/$ – see front matter
http://dx.doi.org/10.1016/j.enggeo.2013.05.004

elevated temperatures. With this in view, attempts were made to explore the utility of the nanoindentation for establishing changes in the mechanical properties (viz., hardness and residual modulus) of the fine- and coarse-grained soils when they get exposed to elevated temperatures. Details of the methodology adopted for this purpose are presented in this technical note and based on a critical synthesis of test results; the utility of nanoindentation in understanding the behavior of soils exposed to elevated temperature has been demonstrated.

2. Experimental investigations

2.1. Materials and their characterization

Two commercially available soils; bentonite and white clay designated as BT and WC, respectively, two naturally occurring soils, sampled from western region of India, designated as S1 and S2, and one standard sand sample, designated as SS, were used in this study. These five soils were characterized for establishing their physical, chemical and mineralogical characteristics by conducting a series of investigations. For the sake of completeness, and clarity, details of these investigations are presented in the following.

2.2. Physical characterization

2.2.1. Specific gravity

The specific gravity, *G*, of the soil sample was determined with the help of an ULTRA-PYCNOMETER (Quanta-chrome, USA), which employs helium gas as a displacing fluid as per the guidelines provided by ASTM D 5550-06. For the sake of accuracy, the average specific gravity obtained from the results of three tests is reported in Table 1.

2.2.2. Gradational and consistency characteristics

The particle-size distribution characteristics and consistency limits (i.e., the Atterberg limits) of the soil sample were determined as per the guidelines provided by ASTM D 422-63; ASTM D 4318-93 and ASTM D 427, respectively. Consequently, soil samples have been classified based on the Unified Soil Classification System, USCS, ASTM D 2487-10. The test results are presented in Table 1 from which it can be noticed that the soils considered for this study are of entirely different characteristics.

2.2.3. Specific-Surface Area

The Specific Surface Area, *SSA*, of the soil samples was determined by employing ethylene glycol monoethyl ether, EGME, method which has been shown to be the most efficient method for determining the *SSA* (Carter et al., 1986; Cerato and Lutenegger, 2002; Arnepalli et al., 2008). The amount of EGME, W_a, that gets absorbed on per gram of the soil, W_s, was computed by subtracting the dry weight of the sample from the weight of the EGME mixed sample. Subsequently, by employing Eq. (1), the *SSA* of the sample was determined and the results are presented in Table 1.

$$SSA = W_a \cdot (0.000286 \cdot W_s)^{-1}. \quad (1)$$

Table 1
Physical characteristics of different soils.

Soil	*G*	*SSA* (m^2/g)	Fraction (%)			Atterberg limits (%)				*USCS*
			Clay	Silt	Sand	*LL*	*PL*	*PI*	*SL*	
BT	2.73	629	82	18	–	305	140	165	30	CH
WC	2.63	35	54	46	–	54	27	27	17	CH
S1	2.63	214	39	61	–	47	21	26	9	CH
S2	2.69	91	27	73	–	45	23	22	8	CL
SS	2.77	3	–	–	100					SP

Note: CH: clay of high plasticity; CL: clay of low plasticity; SP: poorly graded sand.

Table 2
Mineralogical composition of different soils.

Soil	Minerals
BT	Montmorillonite
WC	Kaolinite
S1	Quartz, calcite
S2	Quartz, calcite, muscovite
SS	Quartz

2.3. Mineralogical characterization

The mineralogical composition of the soil sample was determined with the help of an X-ray diffraction spectrometer (Phillips, Eindhoven, the Netherlands), which is fitted with a graphite monochromatic and employs Cu-Kα as the source. Minerals present in the sample were identified with the help of the Joint Committee on Powder Diffraction Standards, (JCPDS, 1994) search files, from the diffractograms and are listed in Table 2. It can be observed from the table that the soil samples consist of a wide range of minerals and hence their mineralogy is entirely different.

2.4. Chemical characterization

2.4.1. Chemical composition

Chemical composition of the soil sample, in the form of major oxides, was determined using an X-ray Fluorescence setup, XRF (Phillips 1410, Holland). Details of the sample preparation are presented in the following. 4 g finely powdered sample, 1 g microcrystalline cellulose and a few drops of isopropyl alcohol were mixed thoroughly and the mixture was kept below an infrared lamp for slow drying. A small aluminum dish (with inner diameter of 33 mm and height of 12 mm) was taken and two thirds of this dish was filled with mixture of 70% methyl-cellulose and followed by filling up the container by the dried sample. For making a pellet, the dish was compressed with the help of a hydraulic jack by applying a load of 15 ton. The chemical composition of the sample has been determined by mounting the pellet in the sample holder of the XRF setup and the results are presented in Table 3.

2.4.2. Cation-exchange capacity

The cation-exchange capacity, *CEC*, of the soil signifies its capacity to retain cations up to its highest limit; or it can also be defined as the power of the soil to combine with cations in such a manner that they cannot be easily removed by leaching with water, but can be exchanged by an equivalent amount of other cations. Capacity of a soil to hold cations mainly depends on pH and ionic strength of the soil–fluid system, and the presence of salts. The guideline presented by IS, 2720 Part XXIV-1976 were followed for determination of the *CEC* of the samples used in this study. *CEC* of the sample can be obtained by employing Eq. (2). The chemical properties of the soils used in this study are listed in Table 4, for the sake of completeness.

$$CEC = \frac{Concentration\ of\ Na\left(\frac{\mu g}{ml}\right) \times 100 \times Vol.\,of\ extract\,(ml)}{Equivalent\ wt.\,of\ the\ cation \times 1000 \times wt.\,of\ sample\,(g)}. \quad (2)$$

Table 3
Chemical compositions of different soils.

Soil	% by weight								
	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	Na_2O	TiO_2	MgO	P_2O_5
BT	42.06	18.90	31.17	1.11	0.35	3.55	1.36	0.96	0.11
WC	37.94	52.84	2.52	1.59	1.84	0.19	2.69	0.20	0.03
S1	37.98	30.70	14.96	8.92	0.91	1.65	2.26	2.16	0.11
S2	39.92	27.81	8.55	11.39	3.51	5.36	0.76	2.27	0.23
SS	93.24	3.86	1.64	0.21	0.03	0.93	0	0	0.03