Impact of Exposure to Pressure of 50 MPa on the Specific Surface Area of Clay

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Abstract. The paper presents results of laboratory tests conducted to determine the impact of pressure of 50 MPa on specific surface area of clay. These tests were carried out in an original, high-pressure test stand. The specific surface area of clay extracted directly from an open pit mine was compared with the specific surface area of the same clay subjected to the pressure of 50 MPa in a high-pressure chamber. The study found that the specific surface area of the clay subjected to the pressure of 50 MPa increased distinctly by over 35 %. The increase in specific surface can be a result of changes in the microstructure of clay particles and microstructural alteration in the soil skeleton, caused by the pressure.

1. Introduction

The impact of high pressures is widely described in the literature in the aspect of phenomena occurring in rocks at large depths, in tectonic, seismic and metamorphic zones [1, 2]. It constitutes an important branch of science in many fields of knowledge, such as geology, geophysics, geochemistry, seismology, mining and geotechnics. The influence of high pressures is described mainly in the aspect of identifying changes in the strength and mechanical properties of igneous, metamorphic and some sedimentary rocks [3, 4]. Studies of the effects of high pressures on clayey soils concentrate most of all on description of the impact of preconsolidation phenomenon [5]. Interesting studies were conducted for clay minerals behaviour under high pressures [6, 7]. It has been shown that structural changes in smectite occur at high pressure of 4.0 GPa [6]. In the literature there are also works on the phenomenon of compression mechanisms of unsaturated clay under high stresses [8]. According to the authors, the results from this study provide insight into how constitutive models for unsaturated soils can be extended to high stress conditions for drained and undrained conditions [8].

The influence of pressures exceeding 50 MPa (reaching several hundred MPa) on clay soils has not been sufficiently identified and described so far. This paper presents results of laboratory tests conducted to check the impact of high pressures of 50 MPa on the specific surface area of clay. These studies constitute one of the stages in the research program carried out by the author, concerning identification of the impact of high pressures (from 50 MPa to 400 MPa) on the physicochemical and mechanical properties of fine-grained soils.

Samples for the tests were selected from neogenous clay from Kransk (Lower Silesia, SW Poland), which contains over 60% of clay fractions. A dominant mineral in those clays is kaolinite. Also minor quantities of illite and smectite occur [9].

Previous studies of those soils, carried out by the author, showed distinct changes occurring in clay under high pressure [10-12]. It was found, inter alia, that high pressure caused sorption activation of clay minerals and a distinct change in the content of metal ions in aqueous clay suspension [10]. Microstructural studies were performed in a scanning electron microscope (SEM) and found structural changes occurring in the clay particles under high pressure [12]. After applying pressure of 200 MPa a distinct increase was noticed in specific surface area of clays subjected to such pressure [11].

In the next stage of the study it was decided to examine how the pressure of 50 MPa affects the specific surface area of clays. Laboratory tests were performed on identical soil samples as in tests at the pressure of 200 MPa [11]. Test results are presented in this paper.

2. The purpose and methodology of tests

The purpose of the study was to investigate whether and to what extent the high pressure of 50 MPa changes the values of specific surface area of clay.

The high-pressure tests were carried out in a prototype testing device, designed and built at Wroclaw University of Science and Technology [13]. The specific surface area was determined by the methylene blue adsorption method. The specific surface area of non-compressed clay was compared with results obtained for the soil subjected to the pressure of 50 MPa.

2.1. High-pressure tests

The most important components of the high-pressure test stand are: a high-pressure reactor, a pressure pump and a pressure recording system. The reactor chamber is filled with a working fluid (a mixture of glycol and glycerine).

One test cycle included preparation of 6 glass cylinders filled with aqueous suspensions of clay (2 g of dry soil/20 cm^3 of distilled water) and closed by watertight pistons equipped with a vent valve. Tests were carried out on 6 identical samples of clay suspensions (K50/1, K50/2, K50/3, K50/4, K50/5, K50/6), while maintaining the same conditions and procedure. Cylinders with samples were placed in turn in the high pressure reactor chamber. The pressure was transmitted onto the sample through a piston moving in the cylinder under pressure of working fluid in the reactor. The pressure was raised slowly up to 50 MPa and then maintained for 5 minutes. After this time, it was slowly lowered to 0 MPa. The temperature during tests was constant at 21 ºC. The pressure acting on the soil was isotropic and subject to Pascal's law. After completion of pressure tests the clay samples were transferred to further studies to determine the specific surface area.

2.2. Tests of specific surface area

Determination of the specific surface area was performed by the methylene blue adsorption method and recommendations provided in the literature [14].

The specific surface area was determined for 12 samples of soil suspension (2 g of soil/100 cm^3 of distilled water), prepared from the same clay:

- 6 samples K0/1, K0/2, K0/3, K0/4, K0/5, K0/6) not subjected to the pressure in the high pressure reactor;
- 6 samples (K50/1, K50/2, K50/3, K50/4, K50/5, K50/6) previously subjected to the pressure of 50 MPa.

3. Results and discussions

In order to achieve the intended purpose of the paper - a comparison was made between the specific surface area of clay samples not subjected to high pressure (table 1) and samples subjected to the pressure of 50 MPa (table 2).

Table 1. The results of specific surface area of clay samples not subjected to high pressure (K0).

No. of sample	Soil sorption capacity	Specific surface area	Average of specific surface area
	MBC [g/100g]	**S_t** [m^2/g]	**S_{ta}** [m^2/g]
K0/1	5.95	124.59	
K0/2	5.16	108.05	
K0/3	4.63	96.95	
K0/4	5.69	119.15	**111.71**
K0/5	5.42	113.49	
K0/6	5.16	108.05	

Table 2. The results of specific surface area of clay samples subjected to the pressure of 50 MPa (K50).

No. of sample	Soil sorption capacity	Specific surface area	Average of specific surface area
	MBC [g/100g]	**S_t** [m^2/g]	**S_{ta}** [m^2/g]
K50/1	7.54	157.89	
K50/2	7.28	152.44	
K50/3	7.28	152.44	
K50/4	7.01	146.79	**151.46**
K50/5	7.54	157.89	
K50/6	6.75	141.35	

The specific surface area of clay not subjected to pressure was within the range from 96.95 to 124.59 m^2/g. The average value from three measurements amounted to 111.71 m^2/g. In case of clay samples subjected to the pressure the specific surface area ranged from 141.35 to 157.89 m^2/g, and the average value amounted to 151.46 m^2/g. An increase in specific surface area by 39.75 m^2/g was recorded for soils subjected to pressure. Thus the specific surface area rose by 35.58 %.

The increase in the specific surface area of clay subjected to the pressure of 50 MPa shows indirectly, that in the soil skeleton of clay there must have occurred changes in the microstructure of clay particles. The increase in specific surface area due to the action of high pressures had been observed also previously in studies on the impact of the pressure 200 MPa on samples of the same clay [11]. Then the increase in specific surface area by 53.68% was noted after the action of pressure on the soil [11]. The rise in specific surface area of clay under pressure which is four times less, i.e. 50 MPa, is lower by about 18 % than the rise in this parameter for soil compressed by 200 MPa.

In other, separate tests performed in the scanning microscope, under high pressures irreversible mechanical changes were noted in the microstructure of clay particles [12]. Results of these studies confirm that as a result of high pressures in the soil the clay particles undergo structural alteration, which may have a direct impact on the change in specific surface area.

4. Summary and conclusions

1) The study examined the impact of pressure 50 MPa on changes in the specific surface area of clay. To this end a comparison was made between specific surface areas obtained for samples not subjected to pressure with those obtained for samples compressed in a high pressure chamber.

2) The examination found, that specific surface area amounted to:

- for samples not subjected to pressure: from 96.95 to 124.59 m^2/g, giving the average value of 111.71 m^2/g
- for samples subjected to the pressure of 50 MPa: from 141.35 to 157.89 m^2/g, giving the average value of 151.46 m^2/g.

3) An increase in specific surface area by 35.58 % was noted for samples subjected to the pressure of 50 MPa in relation to clay not compressed in a high pressure reactor.
4) The increase in specific surface area of clay subjected to the pressure of 50 MPa can be an indicator of structural alteration of clay particles and microstructural changes in the soil skeleton of clay, caused by the pressure.

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