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# THE EFFECT OF 200 MPa PRESSURE ON SPECIFIC SURFACE AREA OF CLAY

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**Abstract:** The paper presents the results of laboratory studies of the 200 MPa pressure effect on specific surface area of clay. The original high-pressure investigation stand was used for the pressure tests. Determination of the specific surface area was performed by the methylene blue adsorption method. The results of the specific surface area test were compared for non-pressurized clays and for clays pressured in a high-pressure chamber. It was found that the specific surface area of pressurized soil clearly increased. This shows that some microstructural changes take place in the soil skeleton of clays.

Key words: *clay, high pressure, specific surface area, microstructural changes*

## 1. INTRODUCTION

The knowledge of the effects of pressure values exceeding several dozen MPa, reaching even as much as several hundred MPa, on the fine-grained soils is of significant importance for the needs of solving specific problems connected with underground and harbour engineering, in particular, in the earthquake zones, with the mining exploration, primarily of water and petroleum or natural gas deposits. The problems are of the limits of the well-known soil mechanics that considers the effects of pressure on soils, from the viewpoint of the soil skeleton and pore water mechanics, within the pressure range of 0–10 MPa [8], [9], [13]. In Poland as well as in the world, the high-pressure investigations were done on solid rock samples, especially of igneous and metamorphic rocks, as well as some sedimentary rocks. The investigations showed that the pressure values exceeding 10 MPa may be the reason for irreversible changes in the properties of such rocks [3], [5], [6]. The influence of high pressure values on fine-grained soils has not been recognized sufficiently yet. The petrographic structure of fine-grained soils determines, at the same time, a higher sensitiveness of the structural components (also at the micro and nano scales) to the action of the pressure set from the outside. An example may be the changes of the specific surface area of clay.

In the study presented, the results of laboratory investigations of the impacts exerted by high pressure values on the level of 200 MPa onto the specific surface area of clay. The examinations constitute one stage of a research program concerning recognition of the high pressure impact onto the physical and chemical properties of fine-grained soils.

As the specific soil surface area, the mineral skeleton surface area (of the solid phase) related to the skeleton mass, expressed in $m^2/g$, is considered [4]. The specific surface area is a feature connected strictly with the clay fraction content, clayey mineral type and with the organic part content in the soil [2], [16]. Many soil properties significant for the engineering practice, such as cohesion, consistency limits, swelling, shrinkage, as well as the ion exchange, depend on it [2], [10], [12], [16]. The minerals of the smectite group (swelling minerals) deserve special attention, since they have, in addition to external particle surfaces, the surfaces in the interlayer spaces, available to water and exchangeable ions [4], [8]. The total specific surface area of these minerals amounts to 700–800 $m^2/g$ [4]. The not-swelling minerals, such as kaolinites, possess the external particle surface and their specific surface area is included within the interval from 10 up to 30 $m^2/g$ [4].

For the investigations, samples of neogenic clay from Krańsk (Lower Silesia, SW Poland), containing more than 60% of the clay fraction, were chosen. The clays are described as a poly-mineral deposit.

The mineral dominating in these clays is kaolinite and it is accompanied by illite and smectite [1]. Soil-water suspensions prepared from this clay were exposed to 200 MPa pressure. Such high pressure values were obtained in an original test stand. Determination of the specific surface area of the samples was conducted by the methylene blue adsorption method. It is a reliable and simple method which is frequently used for gaining information on the presence of clay minerals and for some of their properties [15].

## 2. WORK OBJECTIVE AND RESEARCH METHODOLOGY

The objective of this work was to determine if high pressure acting upon clay exerts influence on the size of the specific surface area of the soil involved. This was realized by comparing the specific surface area of clay not exposed to any action of pressure in the high pressure chamber (K0) with the results obtained for clay exposed to the action of pressure of 200 MPa (K200).

The most important components of the research methodology were the following:

– preparation of water clay suspension samples,
– tests on the high pressure stand,
– testing the specific surface area of clay by the methylene blue adsorption method.

In the research work, the following assumptions were undertaken:

(a) Pre-prepared soil samples are homogenous;
(b) The method of determination of the specific surface area with methylene blue is accurate enough for the comparative evaluation of the specific surface area of soil prior and post exposing the latter to the action of high pressure;
(c) In the pre-prepared soil sample (liquid phase), water has free access to the soil particle surface;
(d) Temperature during tests is constant and equal to 21 °C;
(e) Pressure acting upon the soil is isotropic, subject to the Pascal law;
(f) The pressure value of 200 MPa is obtained through increasing the pressure value in the high-pressure reactor, from 0 MPa to 200 MPa, continuously (not rapidly), the return to 0 MPa happens in a similar way;
(g) 200 MPa pressure action time for the clay sample – 5 minutes.

### 2.1. CHARACTERIZATION OF THE TEST STAND FOR HIGH-PRESSURE INVESTIGATIONS

The test stand serving for pre-setting high hydrostatic pressure values, presented in Fig. 1, was designed and manufactured in the Institute of Fluid Mechanics of the Wrocław University of Technology [14]. The stand makes it possible to carry out tests under hydrostatic pressure up to 800 MPa, in practically unlimited time.

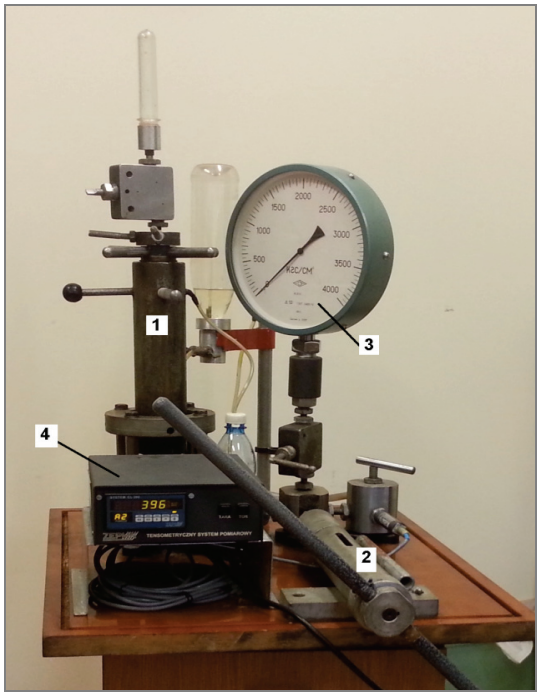


Fig. 1. The high-pressure test stand

The most important components of this device (Fig. 1) are: high-pressure reactor (high-pressure chamber) (1), pressure pump of infinitely-variable pressure control (2), double pressure recording systems: hydraulic (3) and electronic (4).

### 2.2. HIGH PRESSURE INVESTIGATIONS

A glass measuring cylinder filled with aqueous suspension of clay was put into the pressure chamber of the high-pressure reactor. The reactor was filled with working fluid composed of a mixture of glycol and glycerine. The chamber was closed with a header. The system was vented and, using a hand pump, the required pressure value of 200 MPa was produced. The pressure value was gained by the working fluid in the pressure chamber and transferred this pressure onto the sample of soil suspension. The pressure value

of 200 MPa was maintained for the period of 5 minutes. Next, it was systematically decreased until the value of 0 was obtained. The tests were taken for 3 samples of the same aqueous suspension of clay (2 g of clay in the air-dry state/20 $cm^3$ distilled water) in three separate cylinders (K200/1, K200/2, K200/3), maintaining the same procedure and conditions. After completion of the pressure tests, the suspensions were handed over to further investigations in order to determine the specific surface area.

### 2.3. SPECIFIC SURFACE AREA TESTS

Determination of the specific surface area was done by the methylene blue adsorption method, in accordance with the standard PN-88/B-04481 [17] and directions of Myślińska [11].

The specific surface area of clay was calculated from the formula

$$S_t = K_1 MBC$$

where

$S_t$ – specific soil surface area ($m^2/g$),

$K_1$ – coefficient equal to 20.94 $m^2/g$, defining the sum of the projections of the surface of 1 g of the methylene blue particles,

*MBC* – soil sorption capacity denoting the number of grams of methylene blue, adsorbed by 100 g of dry soil.

The sorption capacity of the soil samples with respect to the methylene blue (MBC) was calculated from the formula

$$MBC = \frac{100m}{m_s} \times \frac{V_i + V_{i-1}}{2}$$

where

*MBC* – the soil sorption capacity denoting the number of grams of methylene blue adsorbed by 100 g of dry soil,

*m* – the mass of methylene blue, in grams, contained in 1 $cm^3$ of the solution, recalculated for the 3-aqueous substance ($g/cm^3$),

$m_s$ – the mass of soil used for the test, recalculated for a sample dried at a temperature of 105–110 °C (g),

$V_i$ – the methylene blue solution volume at which the sorption capacity was exceeded ($cm^3$),

$V_{i-1}$ – the methylene blue solution volume, at the last but one portion, of the solution prior to exceeding the soil sorption capacity ($cm^3$).

The specific surface area was determined for 6 samples of aqueous suspension of clay, which were prepared from the same soil (2 g clay in the air-dry state/20 $cm^3$ distilled water):

- 3 samples nor exposed to action of pressure in the high-pressure reactor, subject only to influence of atmospheric pressure (K0/1, K0/2, K0/3) – "state 0",
- 3 samples exposed to action of pressure 200 MPa (K200/1, K200/2, K200/3) – "state post".

## 3. TEST RESULTS AND DISCUSSION

To obtain the intended objective of this work, the specific surface area of clay not exposed to the action

Table 1. The results of the specific surface area of clay not exposed to the action of pressure in the high-pressure reactor (K0)

| No. of sample | $V_i$ [$cm^3$] | $V_{i-1}$ [$cm^3$] | $V_{is} = \frac{V_i + V_{i-1}}{2}$ [$cm^3$] | Soil sorption capacity MBC [g/100g] | Specific surface area $S_t$ [$m^2/g$] |
|---|---|---|---|---|---|
| K0/1 | 11.5 | 11.0 | 11.25 | 5.952 | 124.63 |
| K0/2 | 9.0 | 8.5 | 8.75 | 4.629 | 96.93 |
| K0/3 | 10.0 | 9.5 | 9.75 | 5.158 | 108.01 |
| Average of specific surface area: $S_{ts}$ = 109.85 $m^2/g$ | | | | | |

Table 2. The results of the specific surface area of clay exposed to the action of pressure 200 MPa (K200)

| No. of sample | $V_i$ [$cm^3$] | $V_{i-1}$ [$cm^3$] | $V_{is} = \frac{V_i + V_{i-1}}{2}$ [$cm^3$] | Soil sorption capacity *MBC* [g/100g] | Specific surface area $S_t$ [$m^2/g$] |
|---|---|---|---|---|---|
| K200/1 | 16.0 | 15.5 | 15.75 | 8.333 | 174.49 |
| K200/2 | 15.5 | 15.0 | 15.25 | 8.068 | 168.94 |
| K200/3 | 15.0 | 14.5 | 14.75 | 7.80 | 163.33 |
| Average of specific surface area: $S_{ts}$ = 168.82 $m^2/g$ | | | | | |

of pressure values (K0) was compared with the results obtained for the samples compressed in the reactor (K200). The results of the laboratory tests are shown in Tables 1 and 2.

The specific surface area of the clay in the case of samples not exposed to the action of high pressure amounted from 96.93 up to 124.63 $m^2/g$. As the result of the specific surface area for this soil, the mean value of three measurements was assumed, which amounted to 109.85 $m^2/g$. For the clay exposed to the action of 200 MPa pressure, the specific surface area was on the level from 163.33 up to 174.49 $m^2/g$ and the mean value amounted to 168.82 $m^2/g$. An increase in the specific surface area of 53.68% with respect to the samples not exposed to pressure was noted, while the difference in the specific surface area was equal to 58.97 $m^2/g$. This may be the evidence for the high pressure value of the order of 200 MPa resulting in microstructural changes in the soil skeleton of clay. This may follow from the structural reconstruction of clay minerals and mineral aggregates. In addition, the increase of the specific surface area may be connected also with the physical-chemical reactions occurring in the clay suspension on the phase borders, i.e., clay mineral–water. In this aspect, the results obtained in this work are convergent with the interpretation of the research results published in earlier work, where the influence of pressure of 400 MPa onto the chemistry of the water suspension of clay was considered [7]. It was then found that the consequence of action of such high pressure is the change in concentration of ions of 6 metals in water suspension of clay. It was recognized that this phenomenon is a result of the internal reconstruction of the plates of clay minerals [7].

## 4. RESUME AND CONCLUSIONS

1. In the study, the results of investigation of the impact of high pressure onto the specific surface area of clay have been presented. The specific surface area results obtained for the clay samples not exposed to the action of high pressure values have been compared with the results obtained for the samples exposed to pressure of 200 MPa in the reactor of the high-pressure test stand.
2. The specific surface area of clay not exposed to the action of high pressure values amounted, on average, to 109.85 $m^2/g$ while the specific surface area of clay, onto which 200 MPa pressure acted, increased by 53.68%, reaching the mean value of 168.82 $m^2/g$.
3. On the basis of the results obtained, it can be stated that the accepted method of determination of the specific surface area with methylene blue is accurate enough for the comparative determination of soil prior and post exposing it to the action of high pressure values.
4. The increase in the specific clay surface area may be explained by that, under the impact of high pressure, within the extent of clayey particles, there were created new additional surfaces accessible for the methylene blue used.
5. The results obtained indicate that the action of high pressure values of the 200 MPa order results in microstructural changes in the soil skeleton of clays.

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