THE CHANGE OF ADHESION FORCES ON THE SURFACE OF THE MONTMORILLONITE CLAY PARTICLES DURING HIGH PRESSURE PROCESSING

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IZMENENIE SIL ADGREZII NA POVYRSHTOCHNOSTI CHASTIC PRI OBRABOTKE MONTMORILLONITOVYX GLINNYX VYSOKIM DAVLENIIEM

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For the formation of the technological properties of clays, various methods for their processing have been developed: thermal, chemical, ultraviolet, mechanical and others. However, the issues of changing the properties of clays and clay particles, previously compacted with high pressures, are not well understood. Therefore, the aim of the work is to study the patterns of change in adhesion forces on the surface of particles of montmorillonite clay treated with stress pressure and shear. The adhesion force on the surface of clay particles and in the space between them was measured by atomic force microscopy on previously prepared samples of montmorillonite clay, compacted under different pressure (from 25 to 800 MPa).

Experimental results showed that with increasing pressure on montmorillonite, adhesion forces change. So, with an increase in compaction pressure from 25 to 200 MPa, adhesion forces increase from 0.32 to 0.70 nN. When exposed to pressures above 200 MPa, adhesion forces decrease (to 0.40 nN at P = 800 MPa).

To explain the dependence of the strength of adhesion of montmorillonite on the compaction pressure, the roughness of the samples and the amount of adsorbed water on their surface were studied. It is revealed that the roughness and thickness of the water film on the surface of the clay particles increases with increasing pressure. An increase in the thickness of the water film is probably due to the defects on the particle surface caused by pressure and shear. The presence of additional defects causes an increase in the charge density on the surface of the particles, increasing the number of adsorbed water molecules and the thickness of the water film, respectively.

Keywords: montmorillonite, clay, pressure, adhesion force, clay particle, colloid, particle surface roughness, atomic force microscopy, adsorption water, water film thickness, surface energy activity, surface charge, silicon cantilever, microrelief, correlation matrix.

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**Introduction**

It is known that on the surface of clay particles there is uncompensated negative charge, the formation of which is most affected by cations isomorphic substitution in tetrahedral or octahedral sheets of clay minerals [1–7].

The change in adhesion strength depends on the surface potential and surface charge density. The average surface potential of montmorillonite, according to [8], is $-62.8 \pm 10.6$ mV. Using an atomic force microscope (AFM), it was found that with increasing charge density of clay minerals, the adhesion force increases.

In [9, 10], it was shown that the adhesion force is greatly influenced by the clay particle surface morphology (roughness), and with decreasing roughness, the adhesion force increases. In [11] authors indicate that with a decrease in the roughness of the silicon surface from 13.7 to 0.67 nm, the adhesion force increases 12 times (from 90 to 1080 nN) at a humidity close to zero. Leite et al. [12], studying the substrates of mica and silicon oxide, found that the average adhesion force between the silicon tip and the smooth surface of mica is $26.6 \pm 0.4$ nN, and the rough surface of silicon oxide is $19.0 \pm 1.7$ nN. It is assumed that the dispersion is higher in a rough substrate.

Studies [13–15] showed that the type and properties of the film covering the surface of the particles have a great influence on the adhesion force changes. Thus, with an increase in the water film thickness (at a moisture content of up to 70 %) on a smooth silicon surface, the adhesion force increases by 40 %, and for a rough surface by 400 % [11]. When the silicon oxide surface was contaminated with hydrocarbons, the adhesion force decreased as compared with the cleaned surface [12]. The mechanisms of the surface water films formation and their properties are given in the works of V.I. Osipova, V.N. Sokolova and V.T. Trofimov [16–18].

For the formation of desired clays properties (sorption capacity, plasticity, stickiness, adhesion), various methods have been developed. Thus, during the clays heat treatment high-energy centers on the surface of the mineral are releasing [19, 20]. Grigoriev et al. [21] found that mechanical treatment of titanium carbide powder in a ball mill leads to a decrease in the size of the coherent scattering field. V.V. Boldyrev, in a review paper, noted that pressures affect changes in the structure of a solid particle, primarily the concentration of various types of defects [22]. In the works of J.A. Saponov [23] and S.P. Nichiporenko et al. [24] ultraviolet and ultrasonic activation of clays are carried out, respectively. All of the above methods cause an increase in the particles surface charge density, due to which an increase in adhesion forces occurs.

A limited number of studies are devoted to the study of the treated with pressure clay soils physicochemical properties. LaIglesia [25] found that when kaolinite samples are subjected to pressure in the range of 100–2000 MPa, the coherent dispersion field size (D100) does not change significantly, and at pressures greater than 4000 MPa, these changes occur. Studies by Galán et al. [7] showed that when processing kaolinite with pressures up to 13,200 kg/cm$^2$ (bar), structural changes-defects are formed in it. Isostatic pressure effects on the defects formation 1.4 times higher than that of dry uniaxial pressure. Investigating kaolinite at pressures of 150–2300 bar, E.A. Goylo [26, 27] found that stress pressure is the main cause of disorder in the kaolinite structure. V.A. Frank-Kamenetsky et al. [28, 29], V.V. Boldyrev [22] et al. came to similar conclusions.

When pressure is applied to kaolinite, its physicomechanical properties change [30]; for example it is transformed into diktit, potassium-feldspar, etc. [27, 31–33].

Thus, despite the available research, the issues of the surface adhesion forces changing of the compacted clay particles by high pressure, are not fully understood.

Therefore, the aim of the work is to study the patterns of the particles surface adhesion forces.
changes during the machining of montmorillonite clay with high pressure and shear.

**Research methodology**

**Sample preparation**

The object of the study was montmorillonite clay of the Lobanovsky district. A fraction of less than 0.01 mm was extracted from the natural clay by the sedimentation method. The mineral composition of this fraction was determined by X-ray analysis. According to its results, in the initial clay samples the dominance of montmorillonite (75.0 %) and quartz (11.4 %) was revealed. In addition to montmorillonite, the presence of albite (6.7 %), kaolinite (3.6 %) and calcite (3.3 %) is also observed. The density of clay particles is 2.73 g/cm³, hygroscopic humidity – 3.59 %.

To determine adhesion forces preparation of montmorillonite clay samples was carried out as follows: dried at room temperature natural clay was placed in a mortar and ground with to powder. Then a powder weighing about 0.2 g was placed in the working area (position 3, Fig. 1, b) of the device. The working surfaces of the device 3 are made of carbide material, the area was S = 0.78 cm². As a load device was used press brand PLG-20. After that, the press 6 supplied the vertical pressure on the ground according to the following scheme: the first stage – \( P = 25 \) MPa, on the second and subsequent stages the vertical pressure increased by \( P = 25–50 \) MPa. The maximum pressure was 800 MPa. The pressure on the powder was maintained for 1 minute, after which the upper holder 2 was moved relative to the lower holder 1 by 90° by the handles 4 turning.

As a result, compacted “tablet” samples of montmorillonite clay weighing about 0.2 g and about 1 cm in diameter were obtained (see Fig. 1, c).

**Measurements of roughness and surface adhesion forces of the particles**

Measurements of adhesion and roughness were carried out on an AFM NT-MDT NTEGRA Prima.

At the first stage, the samples roughness was studied (Fig. 2, a). For this purpose, kaolinite clay samples were scanned in semicontact mode using a GOLDEN Silicon Probes NS G10 silicon cantilever with a constant hardness of 3.1–37.6 N/m and a tip radius of about 25 nm. The microrelief of the sample surface was studied in a field of view of 10×10 and 2×2 \( \mu \)m. Scanning the samples surface allowed us to calculate their arithmetic average roughness according to the formula ISO 4287/1-1997 [34]:

![Fig. 1. A device for clay samples compressing and shifting (a) device scheme (b) finished clay samples subjected to compression and shear (c): 1 – lower holder; 2 – upper case; 3 – ground shear area of 0.78 cm²; 4 – handles for the upper case turning (soil shear); 5 – centering ball; 6 – upper plate load device (press); 7 – devices for fixing the transmitted load on the ground](image-url)
where: \( R_a = \frac{1}{N} \sum_{j=1}^{N} |r_j| \) - the sum of the sample surface roughness measurements; \( N \) - number of measurement points.

At the second stage, the adhesion forces were studied (see Fig. 2, a) on the surface of the particles (\( F_n \)) and in the space between the particles (\( F_m \)). The values of adhesion forces were taken from the graphs of the dependence of the force of interaction between the probe and the sample surface on the distance between them (force-distance curves) [35]. According to the force-distance curve, the adhesion force can be calculated using the following formula:

\[
F = k \cdot \Delta \text{Height},
\]

where: \( k \) - cantilever stiffness, in calculations \( k = 3.1 \) N/m; \( \Delta \text{Height} \) - the difference between the cantilever positions (bending of the console) with a jump to the surface and detachment from it, nm [12, 35].

The adhesion forces measurement was carried out at 10 points both on the surface of the particle and in the space between the particles (see Fig. 2, a). A total of 54 samples were examined.

**Calculation of the bound water film thickness on the sample surface**

After montmorillonite clay technogenic processing (compression and shear), samples were obtained. Samples were scanned on AFM in air at a relative humidity of about 30 % (RH), therefore, active adsorption of water from air took place on the surface of clay particles, as a result of which a thin film of bound water was formed.

To calculate the thickness of this film, the following information was used: the loss of mass of compacted montmorillonite clay samples during drying [36] and the change in the active surface area of montmorillonite subjected to shear compression [37]. According to this work, the film of adsorbed water on the surface of montmorillonite completely evaporates at \( t = 150–200 \) C. Knowing the initial mass of the sample and its mass after drying, the mass of bound water was determined. The ratio of the bound water mass to the active surface area indicates the amount of bound water per unit area. Knowing the mass of one water molecule \((2.99 \cdot 10^{-23} \text{ g})\) and its effective diameter \((0.27 \text{ nm})\), we can calculate the film thickness. These calculations were carried out for each value of pressure on the sample. A detailed description of the calculation method is presented in [38].
Research results and discussion

The effect of pressure on the adhesion strength

The results of experimental studies of changes in adhesion forces on the surface of montmorillonite samples subjected to compression with a shift are shown in Fig. 3.

Figure 3 shows that with an increase in $P$ from 25 to 200 MPa, adhesion forces on the surface of particles and between them increase. A subsequent increase in pressure ($P > 20$ MPa) leads to a decrease in adhesion forces. Thus, visually this samples can be divided into two classes: in the first class, at pressures of $P = 25–200$ MPa, an increase in adhesion forces occurs; in the second ($P = 200–800$), the adhesion forces decrease.

To explain this pattern, we should study the factors that influence the magnitude of adhesion forces. These factors, according to [9, 10, 13–15], include:

- the presence of defects, chips of clay minerals and structural disorders of the crystal lattice, which directly affect the magnitude of the surface energy potential;
- surface roughness of clay particles;
- the presence of adsorbed water layers on the surface, which increase the adhesion force due to capillary forces.

After studying the change in these indicators from compaction pressure, it will be possible to judge their role in the adhesion force formation.

The effect of pressure on the particles surface roughness formation

The concentration of various kinds of defects and chipping of the mineral, structural damage to the crystal lattice, changes in the interatomic distance and bond angles can be used as criteria for assessing the clay particle surface energy potential [22].

At the same time, different types of crystal planes, growth stages, crystal edges and corners, irregularities in the crystallographic structure of the surface, such as cracks and defects, as well as functional groups and impurities are the main cause of the clay minerals surface heterogeneity [1]. Consequently, the clay particle surface energy potential depends on its heterogeneity (roughness).
It is seen that the clay particle surface roughness increases with increasing pressure up to \( P = 200 \) MPa. With a further increase in pressure to \( P = 800 \) MPa, a significant variability of \( R_c \) is observed. Therefore, at pressures up to 200 MPa, the clay minerals surface energy activity increases. In this pressure range (from 25 to 200 MPa), the shift significantly changes the structure of minerals, additional energy centers are activated, which increase the surface charge density. At a pressure of 200 MPa and above, changes in the minerals structure are insignificant, since the disturbance of their surface occurs at lower pressures.

Thus, the clay minerals energy potential increases at pressures up to 200 MPa, activating additional energy centers in defects and cracks. The subsequent increase in pressure does not significantly change the energetics of the clay particles surface.

This conclusion contradicts the results of the adhesion strength measuring. If at loads up to 200 MPa, the adhesion force increases with increasing energy potential, then at pressures above 200 MPa, the adhesion force decreases, unlike the surface charge, which remains the same. Therefore, other factors that impede the interaction of the surface of the particles and the AFM cantilever should be considered.
consider the direct influence of the surface roughness and the bound water film thickness on the adhesion forces.

**Analysis of the effect of roughness and the bound water film thickness on the adhesion force of montmorillonite clay**

All the dependencies discussed above have a similar structure: the sampling data can be visually divided into two classes: 1st class with \( P \leq 200 \text{ MPa} \); 2nd class – at \( P > 200 \text{ MPa} \) (Fig. 6). A pressure of 200 MPa should be taken as the critical pressure at which the adhesion forces formation conditions change.

Figure 6 shows how compaction pressure, surface roughness and bound water film thickness affect the adhesion force. Of course, there is no reason to talk about the linear dependence of the adhesion strength and roughness with such a spread of research points. However, this is done to compare the change in adhesion from roughness in classes. It can be seen that in the first class with increasing roughness and thickness of the bound water film adhesion forces increase. Thus, the conclusion is confirmed that the clay minerals energy potential, estimated through the roughness parameter, increases at pressures up to 200 MPa due to the activation of additional energy centers in defects and cracks of minerals. The subsequent increase in pressure does not significantly alter the energetics of the clay particles surface, since pressure of about 200 MPa is sufficient for the destruction of minerals.

Figure 7 shows the pattern of change in the adhesion force \( F \) depending on the adsorbed water film thickness \( H_{ob} \). It is seen that in the first class, with an increase in \( H_{ob} \), the adhesion forces \( F_n \) and \( F_m \) increase. In the second class, adhesion forces decrease with a further increase in the film thickness. Perhaps this is due to an overabundance of moisture on the particles surface. All surface energy is spent on the adsorption of water, thereby increasing the thickness of the bound water film. The AFM probe “gets stuck” in the layer of this film and with increasing of the water film thickness is farther and farther from the surface, thereby fixing a lower attractive force.

To study the relationship between these indicators correlation analysis was used. Evaluation was made by linkages correlation coefficient. This method is described in [39, 40]. The results of correlation coefficients calculations between the paired indicators are presented in the table.
It should be noted that all the obtained calculated values of the correlation coefficients ($r_p$) are greater than the critical value of the correlation coefficient ($r_t = 0.24$), calculated for $n = 54$, $\alpha = 0.05$. Therefore, we can conclude that pressure affects the formation of adhesion forces on the surface of clay particles and in the space between them. The roughness and thickness of the bound water film affect the adhesion forces formation in the first class. In the second class, the roughness does not affect the adhesion forces, unlike the water film thickness.

To assess the effect of pressure ($P$) on the adhesion forces formation, a regression analysis was used, the essence of which is that the higher the value of the angular coefficient at the independent variable ($P$), the greater the influence exerted on the formation of adhesion forces [39].

Calculated mathematical models have the form:

- for class 1: $F_n = 0.2001 + 0.0022P$, $F_m = 0.1856 + 0.0021P$;
- for class 2: $F_n = 0.5571 - 0.0002P$, $F_m = 0.5815 - 0.0003P$.

Analysis of the obtained equations shows that in class 1, the coefficients at $P$ are 7–10 times higher than in class 2. Therefore, it can be assumed that $P \leq 200$ MPa shows a greater influence on the adhesion forces formation than $P > 200$ MPa.

Thus, in the first class, as the pressure increases, the adhesion force on the particles surface and in the space between them increases due to an increase in the surface energy potential, estimated through the roughness parameter. In the second class, adhesion forces decrease due to an increase in the thickness of the bound water film, which prevents the AFM probe from penetrating to the surface.

**Conclusion**

The results of experimental studies have shown that during the mechanical processing of kaolinite clay by pressure and shear, the surface roughness of the clay particles increases. The growth of roughness is caused by the growth of defects on the particle surface. The presence of
additional defects causes an increase in the charge density on the particles surface. An increase in the charge density leads to an increase in the thickness of the water film around the particle. Collectively, an increased charge density and an increased water film thickness on the particle surface increase the adhesion forces. This pattern occurs when $P \leq 200$ MPa. At pressures above 200 MPa, adhesion forces decrease due to an increase in the thickness of the bound water film, which prevents the AFM probe from penetrating to the surface.

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