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ALTERATION IN COMPOSITION OF CLAYS SUBJECTED TO MAN-MADE IMPACT

Karine A. Alvanyan, Aleksandr V. Rastegaev[1], Tatyana Yu. Khludeneva

Perm State National Research University (15 Bukireva st., Perm, 614068, Russian Federation)
[1]Perm National Research Polytechnic University (29 Komsomolskiy av., Perm, 614990, Russian Federation)

ИЗМЕНЕНИЕ СОСТАВА ГЛИН, ПОДВЕРЖЕННЫХ ТЕХНОГЕННОМУ ВОЗДЕЙСТВИЮ

К.А. Алванян, А.В. Растегаев[1], Т.Ю. Хлуденева

Пермский государственный национальный исследовательский университет (614068, Россия, г. Пермь, ул. Букирева, 15)
[1]Пермский национальный исследовательский политехнический университет (614990, Россия, г. Пермь, Комсомольский проспект, 29)

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Physical and chemical properties of clays depend on a group of factors determining energy potential on particle surface and on factors forming particle specific surface.
Particle specific surface formation is directly related to issues of microaggregates formation in soil. Studies of influence of pressure in aggregates formation in dispersive soils showed that at pressure up to 200 MPa slight alteration in aggregate composition is observed in wet soils. At pressure 300 MPa in powdery soil content of thin sandy fraction increased from 13 to 51 %, powdery fraction – from 5 to 23 %, and clay fraction – from 2,15 to 5,42 %. During test of covering loams with pressure P =2000 MPa and P =3 660 MPa similar results were received. From the stated above it follows that issue of pressure influence on formation of microaggregate composition and, as result, physical and chemical properties of clay are of substantial interest.
Regularities in alteration of composition of clays subjected to high pressure were studied. As result of experimental studies, it is established that along with pressure increase general tendency of reduction of clay fraction content and increase of powdery fraction content is observed. Together with this regularity, local alterations of clay fractional composition depending on pressure in each class are found. Along with pressure increase, particle specific surface value of kaolin and montmorillonite clays reduces. Alterations in granulometric composition are determined by particle aggregation and dispergation processes. During aggregation process caused by high pressure coagulation, transition and phase contacts between particles are formed. Breaking and wedging pressure of adhesive water film around particles are leading factors determining process of their dispergation.

Ключевые слова:
глина, монтмориллонит, каолинит, гранулометрический состав, микроагрегатный состав, агрегация, диспергация, давление, техногенная нагрузка, коагуляционные, переходные и фазовые контакты.

Физико-химические свойства глин зависят от группы факторов, определяющих энергетический потенциал на поверхности частиц, и от факторов, формирующих удельную поверхность частиц.
Формирование удельной поверхности частиц напрямую связано с вопросами образования микроагрегатов в грунтах. Исследования влияния давления на формирование агрегатов в дисперсных грунтах показали, что при давлениях до 200 МПа наблюдается незначительное изменение агрегатного состава сырых грунтов. При давлении 300 МПа пылеватого грунта содержание тонкой песчаной фракции увеличилось от 13 до 51 %, пылеватой – от 5 до 23 %, а глинистой – от 2,15 до 5,42 %. При испытании покровных суглинков давлением P =2000 МПа и P =3 660 МПа получены аналогичные результаты. Из приведенного выше следует, что вопросы влияния давления на образование микроагрегатного состава и, как следствие, физико-химических свойств глин представляют значительный интерес.
Исследованы закономерности изменения состава глин, подверженных высоким давлениям. В результате экспериментальных исследований установлено, что с увеличением давления наблюдается общая тенденция снижения содержания глинистой и увеличения пылеватой фракций. Наряду с этой закономерностью в каждом классе выявлены локальные изменения содержания фракционного состава глин в зависимости от давления. С увеличением давления площадь удельной поверхности частиц каолиновой и монтмориллонитовой глин уменьшается. Изменения гранулометрического состава обусловлены процессами агрегации и диспергации частиц. В процессе агрегации, вызванной высоким давлением, формируются коагуляционные, переходные и фазовые контакты между частицами. Дробление и расклинивающее давление пленки связанной воды вокруг частиц являются ведущими факторами, определяющими процесс их диспергации.

Karine A. Alvanyan – PhD student at the Department of Engineering Geology and Mineral Protection (tel.: +007 342 239 64 39, e-mail: karishuta@yandex.ru). The contact person for correspondence.
Aleksandr V. Rastegaev (Author ID in Scopus: 55531816900) – Doctor of Geology and Mineralogy, Professor of the Department of Geology of Oil and Gas (tel.: +007 342 219 83 14, e-mail: arastegaev@mail.ru).
Tatyana Yu. Khludeneva – PhD student at the Department of Engineering Geology and Mineral Protection (tel.: +007 342 239 64 39, e-mail: seredin@nedra.perm.ru).

Алванян Карине Антоновна – аспирант кафедры инженерной геологии и охраны недр (тел.: +007 342 239 64 39, e-mail: karishuta@yandex.ru). Контактное лицо для переписки.
Растегаев Александр Васильевич – доктор геолого-минералогических наук, профессор кафедры геологии нефти и газа (тел.: +007 219 80 00, e-mail: arastegaev@mail.ru).
Хлуденева Татьяна Юрьевна – аспирант кафедры инженерной геологии и охраны недр (тел.: +007 342 239 64 39, e-mail: seredin@nedra.perm.ru).

Introduction

Physical and chemical properties of clays depend on factors determining energy potential on particle surface [1–4] and on factors forming particle specific surface [5, 6].

To form energy potential in clays various methods for their processing (activation): thermal [7–10], chemical, using acids [11], using alkali [12], using salts with different exposure duration [13].

According to data of B.K. Kara-Sal et al. [8], when heating clays to temperatures 400–600 °C their sorption activity increases due to release of high energy centers at mineral surface. By opinion of Zh.A. Sapronova et al. [14], during ultraviolet clay activation metallic ions move away from octahedral positions due to weakening links in mineral matrix. This aids to increase clay activity by 1,3 times. Ultrasound clay processing leads to destruction of aggregates and matrix completion, which increases clay activity [15].

A limited number of works is devoted to study of alteration of physical and chemical properties of pressure-processed kaolin. In most details these issues were dealt with by A. G. Kosovskaya et al. [16], V.A. Eroschev-Schak et al. [17], E.A. Goilo et al. [18], K.J. Rangeetal. [19], V.A. Frank-Kamenetzky et al. [20], LaIglesia [21], Galan et al. [22]. LaIglesia [21] established that action of pressure (100–2000 MPa) on kaolin does not lead to substantial change of coherent scattering area d_{001} (energy potential), and at pressures over более 4 000 MPa these changes become manifested.

Particle specific surface forming is directly related to issues of microaggregate formation in soil [1, 23, 24]. Works [23, 25] provide information on formation of granulometric and microaggregate composition of wet clays in processes of their natural formation and transformation. Works [5, 6] publish information on clays subjected to man-made impact with $MgCl_2$, $CaCl_2$, KCl and NaCl solutions. The authors came to conclusion that aggregation process is related to concentration of salt solution and mineral composition of particles.

Issues of influence of a mechanical factor, for example, pressure, on aggregate formation in disperse wet soils were studied in works [25–27]. The studies performed [28] showed that at pressures up to 200 MPa insignificant alteration of aggregate composition of raw soils is observed. At pressure of 300 MPa on powdery soil this sandy fraction contents increased from 13 to 51 %, powdery fraction – from 5 to 23 %, and clay fraction – from 2,15 to 5,42 %. During tests of covering loams with pressure P = 2000 MPa and P = 3660 MPa similar results were achieved [28]. On basis of experimental studies, forecasting methods for granulometric composition were developed not only for terrestrial [29], but also for lunar soils [30].

During clay pressure processing together with aggregation process also dispergation breaking processes take place. For kaolinite [21, 31–33] breaking changes particle size and form, and pores distribution. Except that, breaking influences physical and chemical properties of clays [34–38]. Breaking kaolinite does not lead to gradual increase of defectiveness for all crystallites present in the sample [39–42].

From the stated above it can be seen that issues of influence of pressure on formation of microaggregate composition, and consequently, physical and chemical properties of clays are of substantial interest.

That is why purpose of the work is to study regularities of alteration of composition of clays subjected to high pressure.

Object of the study are quaternary kaolin clay from Nizhne-Uvelskoe field in Chelyabinsk Region and Lobanovskaya claystone-like montmorillonite clay from Upper Permian Sheshminsky horizon.

Methodology

In order to pass pressure to clay sample high pressure device was designed and built[43]. Its working surfaces are made from hard alloy material, their surface is S = 0,75 cm^2. Press PLG-20 was used as loading device.

Clay samples were prepared for granulometric analysis using method described in work [5]. Maximal pressure was P = 2200 MPa. In total were made 56 samples of montmorillonite clay and 65 samples of kaolin clay.

Granulometric composition of clays was determined using laser diffraction analyzer «Analyzette-22» according to method described in work [5].

Technical capabilities of the unit permit to measure particle size from 0,08 to 20000 μm. In work[25] it was noted that thin clay fraction less than 1 μm substantially determines physical and chemical properties of clays. That is why, basing on unit capabilities and substantial influence of finely dispersed particles on soil properties, we have studied the following fractions (μm): $F_{<0,1}$, $F_{0,1–0,2}$, $F_{0,2–0,5}$, $F_{0,5–1,0}$, $F_{1–2}$, $F_{2–5}$, $F_{5–50}$.

In total 319 determinations of granulometric composition of montmorillonite clay and 385 – kaolin clay were performed.

It is necessary to note that particle size determination on this device was performed in the following way: first dispergation takes place, then particles are measured, so in result we should obtain granulometric composition of soil. However, reduction of $F_{<0,1}$ fraction shows that not all particles during dispergation process are destroyed to primary size, so we actually obtain microaggregate composition of soil. So, in the context of the issue being discussed, further in the text we use terms «granulometric and microaggregate composition».

Findings

Studies were performed in stages. ***In the first stage*** influence of pressure and mineral composition of clay on alteration of its fractional composition was studied. Experimental data are given in fig. 1, from which it is seen that correlation fields have similar character of alteration for various fractions. However, fields of fraction $F_{<0,1}$ – $F_{2–5}$ of kaolin clay are above corresponding fields of montmorillonite clay, and for fraction $F_{5–50}$ it is vice versa. This is because mineral composition of clays substantially influences on alteration of clay fractions content in soil.

To confirm conclusion on influence of mineral composition of clays on formation of their granulometric composition statistical methods [44] were used, calculation results are shown in table 1. As it can be seen from table 1, calculated values of Student's coefficient (t_p) for each studied fraction are higher than t_m = 0,03, so we may consider that mineral composition exerts substantial influence on formation of fractional composition of pressurized soils. Student's criterion shows that sample population are statistically segregated.

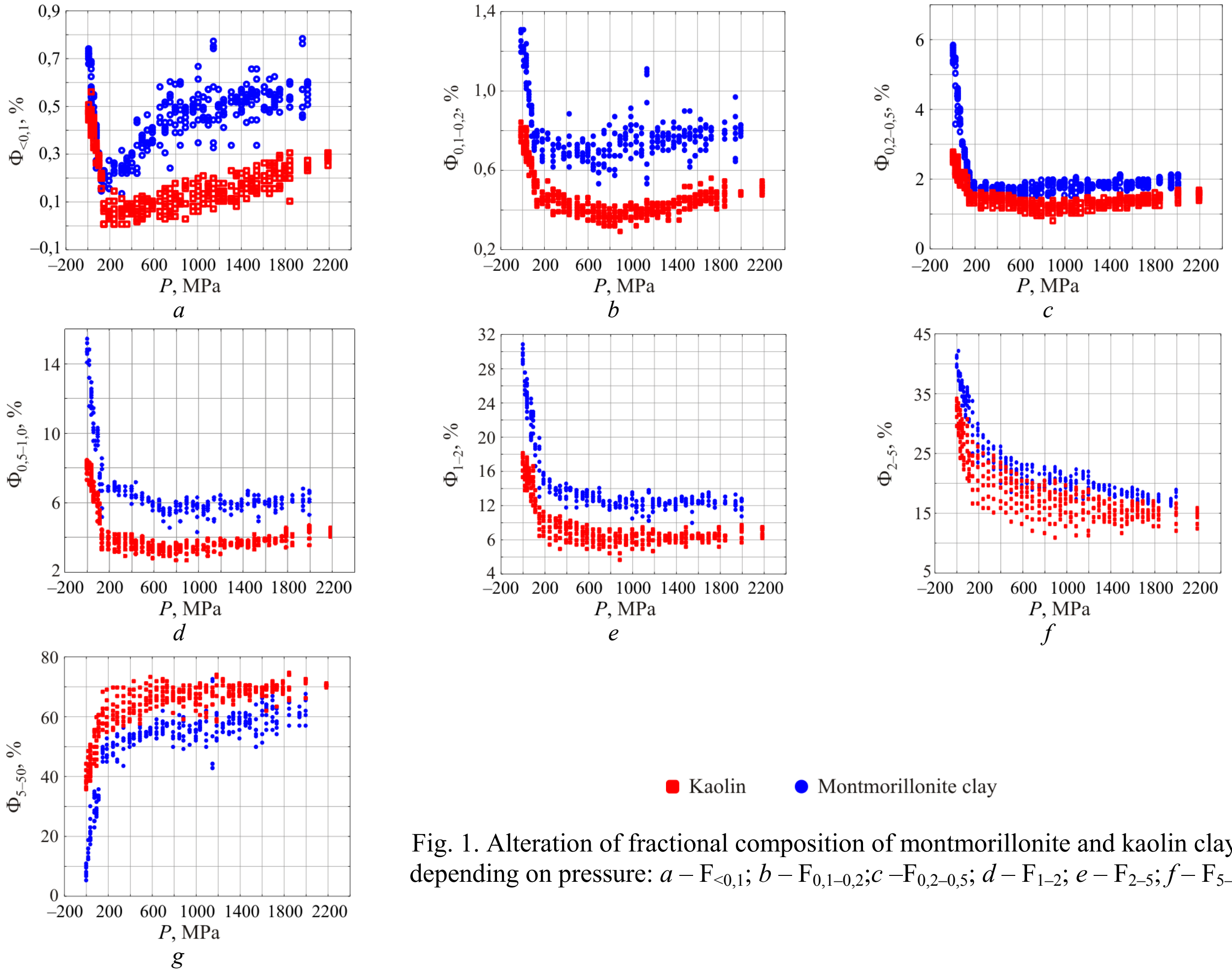

Fig. 1. Alteration of fractional composition of montmorillonite and kaolin clays depending on pressure: *a* – $F_{<0,1}$; *b* – $F_{0,1–0,2}$; *c* – $F_{0,2–0,5}$; *d* – $F_{1–2}$; *e* – $F_{2–5}$; *f* – $F_{5–50}$

Table 1

Statistical characteristics of clays

Fraction, %	Kaolin clay		Montmorillonite clay		Calculated value of Student's coefficient t_p	Recognition of sample populations, %		
	Average	Standard deviation	Average	Standard deviation		kaolin clay	montmorillonite clay	total
$F_{<0,1}$	0,449	0,139	0,180	0,120	25,911	80,0	85,9	83,0
$F_{0,1–0,2}$	0,787	0,148	0,471	0,122	29,197	93,4	85,0	89,1
$F_{0,2–0,5}$	2,184	0,938	1,508	0,397	11,801	42,3	83,4	63,3
$F_{0,5–1,0}$	6,961	2,291	4,262	1,420	17,771	83,6	83,4	83,5
$F_{1–2}$	14,637	4,538	9,673	2,798	16,526	67,5	82,1	75,0
$F_{2–5}$	23,203	6,637	18,812	5,329	9,134	44,6	74,6	59,9
$F_{5–50}$	49,961	13,960	62,695	9,197	–13,511	58,4	81,8	70,4
Z	–1,759	1,020	1,682	0,980	–42,967	97,0	93,4	95,2

Linear discriminant analysis was used for quantitative evaluation of segregation. Calculation results are given in table 1. It is shown that maximal difference between kaolin and montmorillonite is observed for fractions $F_{0,1–0,2}$ and $F_{<0,1}$, and minimal – for fraction $F_{2–5}$.

Other fractions take intermediate position. For them criterion t_p changes from 9,1 to 17,8, and general correctness of recognition – from 59,5 to 83,5 %. It is necessary to note that in the predominant majority of cases montmorillonite clay fractions are recognized better than those of kaolin clay.

In the second stage influence of pressure on general regularities of alteration of clay granulometric composition (separation of classes – pressure range) was studied.

Alteration of clay fractional composition at increase of pressure is given in fig. 1. It is seen that at pressure increase to P = 125 MPa significant reduction of clay fraction content ($F_{<5}$) and increase of powdery fraction content ($F_{5–50}$) is observed. At pressure increase to$P$ = 750 MPa content of clay fractions changes in different directions, and content of powdery fraction increases. At further pressure increase to 2200 MPa clay fractions content increases, and powdery fraction content changes chaotically.

To confirm assumption of presence of threshold pressures P = 125 MPa and P = 750 MPa linear discriminant analysis was used. Discriminant functions calculations performed showed that correct recognition of all samples is 100 %.

Therefore, it is proved that threshold pressure values P = 125 MPa and P = 750 MPa are validly selected. This shows that in each class intensity of aggregation and dispergation processes is different, and so conditions of forming clay fractional composition also have their individual specifics.

In the third stage influence of pressure on alteration of clay granulometric composition within segregated classes *(inside classes)* was studied. Correlation analysis was used for this purpose.

In class1 (P = 0–125 MPa) average content of clay fractions is less than in initial sample. This change is probably related to clay particles aggregation processes resulting in increase of powdery fraction $F_{5–50}$ content.

Calculations showed that statistical links are established between P and F, which is confirmed by valid pair correlation coefficients (r). Presence of negative r values between P and $F_{<5}$ confirms our conclusion that along with pressure increase content of clay fractions decreases. Positive values of r between pressure and powdery fraction content, on the opposite, mean that along with increase of P content of $F_{5–50}$ increases.

For evaluation of degree of pressure influence on alteration of studied fractions content parameter k – angle coefficient was used, being tangent of straight line slope in equation of relation between pressure and fraction. It may be interpreted as follows: the higher are values of k, the greater influence pressure exerts on alteration of content of the studied fraction [32]. Calculation results are given in fig. 2.

It is established that along with particle size decrease influence of pressure on alteration of these fractions content decreases, while the most pressure-sensitive is powdery fraction $F_{5–50}$, and least sensitive – clay fraction $F_{<0,1}$. It is necessary to note that from all clay fractions to the greater extent sensitive to pressure are fractions $F_{1–2}$ and $F_{2–5}$ in montmorillonite and kaolin clay respectively.

Speed of change of clay fractions content in wet kaolin clay is higher comparing to montmorillonite clay, which is proved by higher values of k (fig. 2).

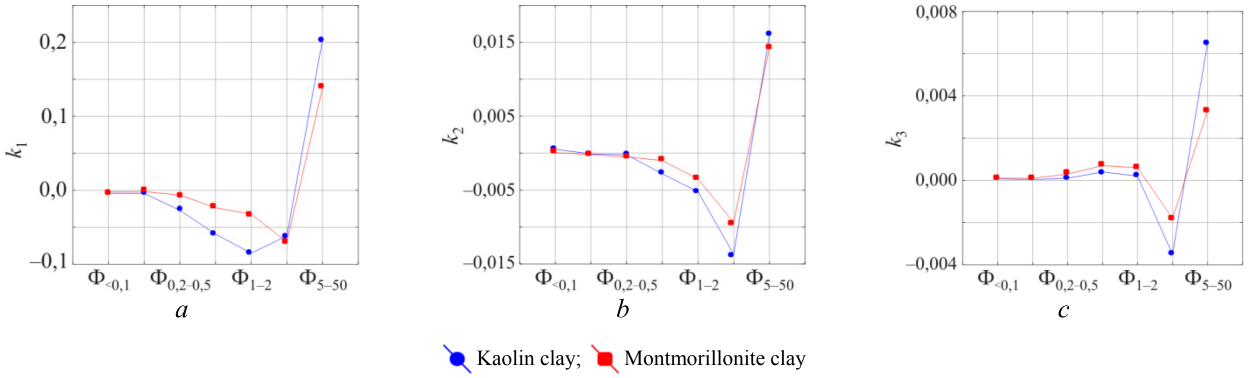

Fig. 2. Dependence of degree of pressure influence on alteration of content of studied fractions of kaolin and montmorillonite clays in classes1 (*a*), 2 (*b*) and 3 (*c*)

In class 2 (P = 150–750 MPa), as in class 1, reduction of $F_{<5}$ clay fraction content and increase of $F_{5–50}$ powdery fraction content is observed (see table 1). Meanwhile for fraction $F_{<0,1}$ other regularity is observed: along with pressure increase content of $F_{<0,1}$ increases, which is determined by dispergation processes of other fractions. Increase of $F_{5–50}$ content is related to processes of clay particles aggregation to powdery fraction sizes.

Of all clay fractions most pressure sensitive is fraction $F_{2–5}$. Meanwhile pressure exerts greater influence on kaolin clay than on montmorillonite clay, which is proved by higher values of k parameter.

In class 3 (P = 800–2200 MPa) influence of pressure on alteration of fractional composition of clays is crucially different from classes 1 and 2. Along with pressure increase content of all fractions except $F_{2–5}$ increases, which is proved by positive values of r. Statistical links are established between P and F, proved by valid pair correlation coefficients. Maximal influence is exerted by pressure on formation of $F_{5–50}$ fraction, and minimal influence – on $F_{<0,1}$.

Of all clay fractions, as in both previous classes, most pressure-sensitive is fraction $F_{2–5}$. Meanwhile pressure exerts more significant influence on kaolin clay that on montmorillonite clay, which is proved by higher values of k parameter.

In the fourth stage influence of pressure on formation of particle specific surface of clay soils was studied. Results of study are given in fig. 3. Calculation of particle specific surface was made per 1 mm^3 of clay.

From fig. 3 it is seen that along with pressure increase area of particle specific surface of kaolin ($S_к$) and montmorillonite ($S_м$) clay reduces.

Table 2

Correlation matrix

Parameter	Kaolin clay			Montmorillonite clay		
	$S_{К_1}$	$S_{К_2}$	$S_{К_3}$	$S_{М_1}$	$S_{М_2}$	$S_{М_3}$
Pressure P, MPa	–0,98	–0,55	0,25	–0,88	–0,61	0,70

Note: $S_{К_i}$, $S_{М_i}$ – area of specific (active) surfaces of particles in classes 1, 2, 3.

Meanwhile the highest speed of S reduction is observed in class 1 (at pressures up to 125 MPa). At pressure over 125 MPa its influence on change of S is less significant. In case of montmorillonite clay on qualitative level interrelation between pressure and surface is observed, while in kaolin clay the relation is not expressed.

For quantitative evaluation of interrelations between P and S correlation and regression analyses were used. Correlation analysis results are given in table 2.

From table 2 it is seen that statistical links are established between pressure and area of clay particle specific surface, which is proven by valid pair correlation coefficients. Calculated value of coefficient of correlation between area of clay particle specific surface in class 3 and pressure equal to r_p = 0,25 is greater than critical value $r_т$ = 0,17 at level of significance α = 0,05 and number of freedom degree n = 160.

In the fifth stage conditions of forming granulometric composition of clays subjected to pressure were studied. During processing clays with pressure particle breaking and aggregation processes are observed in studied samples. Aggregation process is determined by forces of particle interaction between themselves being of physical and chemical nature, and breaking process – by deformations of particles. Let us study influence of pressure on aggregation and dispergation of particles in aspect of their size.

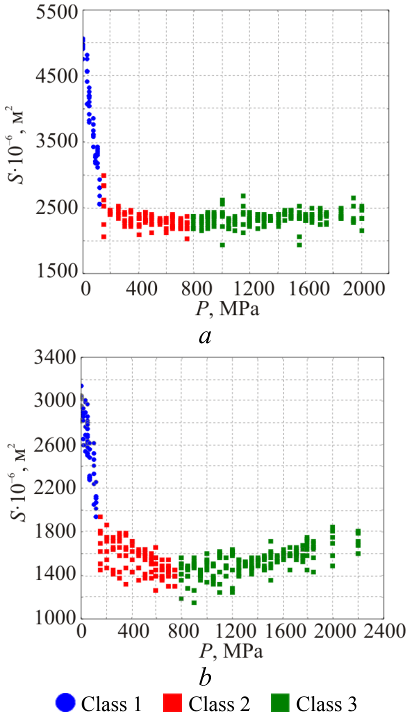

Fig. 3. Alteration of specific surface area in kaolin (*a*) and montmorillonite (*b*) clays depending on pressure

In crystallites aggregation process takes place according to the following scenario: at pressure $P = 0$ MPa (in natural clays) crystallites are interlinked by molecular bonds (through cations of diffuse water layers), i.e. link between crystallites is performed by coagulation contacts. At pressure on soil from 0 to 125 MPa adsorbed water films (diffuse layers) on crystallite surface [45] partially break, that is why crystallites contact between themselves, on one part, in a point-like manner (transition contacts), and on the other part, through diffuse layers (coagulation contacts). At pressure increase from 125 to 2 000 MPa aggregation process is changed by crystallite dispergation process, due to which partial destruction of chemical bonds between crystallites occurs. Reason of this destruction is wedging pressure of adhesive water film, formed by increased energy potential on crystallite surface. That is, we observe reduction of influence in aggregate of point-like contacts (electrostatic and chemical bonds) and increase of weaker coagulation contacts (molecular bonds), and number of contacts between crystallites breaks fully.

In fine colloids (size of 0,1–2,0 μm) at pressure up to 125 MPa aggregation processes follow the above scenario. At pressure from 125 to 2 200 MPa intensity of aggregation and dispergation processes is insignificant, the compensate each other, that is why content of these fractions changes rather weakly.

In coarse colloids (size of2–5 μm) at pressure from 0 to 2200 MPa only aggregation processes happen, with different formation mechanism and intensity. At pressure increase from 0 to 125 MPa number of break-through points in adsorbed water films (diffuse layers) on crystallites surface increases, leading to reduction of coagulation contacts and increase of transition contacts. Therefore, we see reduction of molecular and increase of chemical bonds between colloids. At further increase of pressure from 125 to 800 MPa number of transition contacts increases and individual phase contacts appear. That is, role of chemical bonds between colloids increases. At further pressure increase from 800 to 2200 MPa role of chemical contacts between colloids (phase contacts) prevails. The obtained results do not contradict to nature of forming microaggregate composition of clays.

Conclusions

It is experimentally established that along with pressure increase general tendency of reduction of clay fraction content and increase of powdery fraction content is observed. Together with this regularity, in each class local alterations of clay fraction composition depending on pressure are found. With pressure increase, area of particle specific surface in kaolin and montmorillonite clays decreases. Alterations in granulometric composition are determined by particle aggregation and dispergation processes. In aggregation process, caused by high pressure, coagulation, transition and phase contacts between particles are formed. Breaking and wedging pressure of adsorbed water film around particles are leading factors determining process of particle dispergation.

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