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Comparison of technological properties of clay raw materials during the tests using plastic and soft methods of brick molding

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Abstract. The article presents the results of studies of technological properties of clay raw materials using the example of highly dispersed refractory clays of Vladimirovskoye deposit and low-dispersed fusible loams of Aksayskoye deposit in relation to plastic and soft methods of product molding. Research in this area is being conducted due to the lack of testing methods for clay raw materials in relation to the wet method of product molding, despite the fact that ceramic bricks produced using this production technology are in high demand in modern construction and architecture. In Russia, only a few companies produce bricks using this technology, and many companies have not yet been able to master this technology. Comparative tests on typical water-soaked clay raw materials showed that the existing accepted methods approved by regulatory documents cannot be applied to soft molding technology and that a separate method needs to be developed. Thus, with soft molding, molding materials are characterized by an increased degree of deformation under load, reduced plastic strength and critical compressive stress, as well as increased stickiness. It has been shown that a simple increase in the moisture content of molding materials leads to the deterioration in the basic pre-firing technological properties: air shrinkage, sensitivity to drying, cohesion; and to achieve the required degree of sintering, an increased firing temperature is required. Therefore, increasing the moisture content of molding materials is not a simple solution. Molding materials for soft molding must contain increased amounts of leaners with grain compositions approaching the densest packing and the presence of the required amount of sand fraction particles. Otherwise, obtaining high-quality facing ceramic bricks with high decorative properties becomes extremely problematic from a technological perspective. The results of the conducted research will allow to move closer to the development of a method for selecting raw materials for soft molding of ceramic bricks and, in practical terms, will help in organizing production using this technology.

Keywords: ceramic brick, soft molding, clay raw materials, technological properties, product properties, testing methods

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1. INTRODUCTION

Currently, wall ceramics industry is experiencing a general decline in production which was expected and is due to several interrelated reasons including a rather limited range of manufactured products. However, there is a steady demand for some types of ceramic products, and the domestic industry is currently unable to satisfy it. Such products primarily include soft-molded facing ceramic bricks, which in domestic practice have acquired a stable name - hand-molded bricks. The increased demand for this type of brick is primarily due to its aesthetic properties. The method of soft molding and various technological techniques make it possible to obtain bricks with a very diverse aesthetically attractive front surface, the basis of which is special, unique types of relief brick surfaces, onto which other decorative methods are applied – volumetric coloring, engobing, texturing, glazing (Fig. 1).

Fig. 1. Various types of soft-molded bricks.

In the production of facing ceramic bricks, we have identified various technological methods for product molding that currently do not have generally accepted established names in the “ceramic practice” of Russia – acceleration, pressing in, vibration-casting, manual molding, water-cutting technology, etc. (Fig. 2), but what they all have in common is that the molding materials for this method, unlike the plastic method, are characterized by an increased degree of deformation under load and stickiness, reduced plastic strength and critical compressive stress [1-9]. Depending on the technological method of soft molding, the composition and structural strength of the molding masses, various types of simple or combined brick surfaces are obtained, which also do not yet have generally accepted established names – this is a crumpled surface, wrinkled, with varying depth and shape of depressions, rough, granular, tuberculate, furrowed, wavy, cavernous, cratered, rocky, etc., on which other decorative methods are applied – texturing, engobing, glazing. Considering the diversity of factors, technological search for certain aesthetic solutions in the soft molding of ceramic bricks is a difficult task and requires a lot of experience.

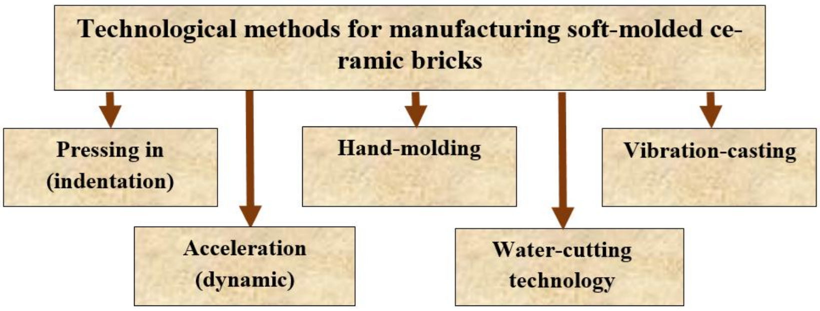

Fig. 2. Technological methods for the production of soft-molded ceramic bricks.

One of the factors limiting the increase in the production of soft-molded facing ceramic bricks is the lack of any recommendatory documents, methods for testing clay raw materials and selecting raw material masses for this technology. GOST 21216-2014 "Clay Raw Materials. Test Methods" provides for the determination of coarse-grained inclusions and fine fractions, chemical and mineral composition, and water-soluble salts. The complex study of technological properties includes the determination of plasticity, refractoriness, air shrinkage, molding moisture, and sensitivity to drying. In this case, normal molding humidity is considered to be the humidity “at which clay mass, exhibiting plastic and molding properties, retains the shape given to it without deformation and does not stick to hands or metal when rolled out.” The molding properties of clay raw materials are assessed quite subjectively according to the following scale: well molded (+++); satisfactorily molded (++); poorly molded (+); not molded (-). It is noted that “for highly plastic clays, the amount of added sand can exceed 50%, for medium-plastic clays it can be 20%-50%, and for moderately plastic clays it can be less than 20%.

As our previous studies have shown, it is not possible to evaluate clay raw materials and select the optimal composition of raw materials for soft molding based on the above parameters. This is due to the fact that the molding moisture content (moisture content) during soft molding is always higher than with the plastic method, therefore the molding materials stick to your hands and to the metal, they always have greater air shrinkage and are more sensitive to drying [7-17]. The assessment of molding properties cannot be carried out subjectively. Criteria that are assessed in specific numbers are needed. Moreover, the introduction of quartz sand in the quantities recommended by GOST 21216-2014 (“For highly plastic clays, the amount of added sand may exceed 50%, for medium-plastic clays it may be 20%-50%, for moderately plastic clays it may be less than 20%) cannot be considered rational, since the introduction of quartz sand in quantities greater than 30% reduces the strength of the shard and makes it brittle. Having analyzed numerous experiments and technological methods, we have established that when testing clay raw materials for soft brick molding, in addition to the above-mentioned technological properties, the degree of deformation of the mass under a certain load, the critical compressive stress, plastic strength (degree of penetration) and stickiness must be determined. The methods of these tests can be used in testing clay raw materials in engineering geology, as well as in determining plasticity using other methods.

2. METHODS AND MATERIALS

The chemical and mineral composition of the clay raw materials was determined using generally accepted methods in accordance with GOST 21216-2014 "Clay raw materials. Test methods" and NSAM method No. 138-X "Determination of rock-forming elements in rocks and ores using accelerated photometric and titrimetric methods." The mineral composition of the clay raw material was studied using X-ray analysis on an ARL X´TRA device with a slit width of 2-4-1-0.5 on oriented, ethylene glycol-saturated and calcined samples with an interval of 5 to 60°, a speed of 4-6 degrees/min, a voltage of 40 kV, and a current of 30 mA. The interpretation of radiographs was carried out using appropriate methods, using international databases and by comparison with similar studies. Determination of coarse-grained inclusions and fine fractions was also carried out in accordance with GOST 21216-2014 "Clay raw materials. Test methods."

Plasticity, molding moisture, air shrinkage, cohesion, and sensitivity to drying were determined according to GOST 21216-2014 "Clay raw materials. Test methods", taking into account that the molding moisture during soft molding is higher than during plastic molding.The degree of deformation of the mass under a certain load and the critical compressive stress were determined using the Pfefferkorn method and device. Plastic strength, which in ceramics technology is defined as the ultimate shear stress that a molding material can withstand under load without breaking its integrity, was determined by the degree of penetration using the methodology adopted in engineering geology according to GOST 34276-2017 "Soils. Laboratory Methods for Determining Specific Penetration Resistivity," as well as using a KP-3 cone plastometer which is used to study the structural and mechanical properties (ultimate shear stress) of viscoplastic materials in many industries. The difference between these methods is that the penetrometer according to GOST 34276-2017 has a conical probe with an opening angle of 30°, a height of >50 mm and a mass of 300 grams, while in the conical plastometer KP-3, the load can be changed by installing weights on the plastometer disk. For each molding mass, 6-7 measurements are carried out with different loads. In any case, the essence of both methods is to determine the depth of immersion of the cone into a plastic viscous mass under the action of a certain load.

Since the stickiness of molding materials during soft molding is an important characteristic, and there is no generally accepted method for testing clay raw materials, it was determined using the method adopted in engineering geology according to GOST 34259-2017 "Soils. Laboratory Method for Determining Stickiness." Firing process properties (sinterability, strength and other physical and technical properties of samples) were determined in accordance with the methods set out in GOST 21216-2014 "Clay raw materials. Test methods", OST 21-78-88 "Clay raw materials (rocks) for the production of ceramic bricks and stones. Technical requirements. Test methods", as well as according to the methods set out in specialized literature [14-22].

We compared the technological properties of clay raw materials during tests using the plastic and soft methods on various types of clay. This article presents the results using highly dispersed refractory clays from Vladimirovskoye deposit and low-dispersed fusible loams from Aksayskoye deposit as examples. The chemical composition of Vladimirovskoye deposit clays and loams is presented in Table 1. The granulometric composition is presented in Table 2. Based on its mineral composition, the clay from Vladimirovskoye deposit can be classified as polymineral with a predominance of kaolinite and the presence of hydromica and montmorillonite. The loams of Aksay deposit, based on the type of clay component, can be classified as belonging to the montmorillonite-hydromica group with the presence of finely dispersed calcite and quartz of the sandy-dust fraction. (Fig. 3, 4).

Table 1. Chemical composition of clays and loams.

Sample	LOI	SiO_2	AI_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	P_2O_5	TiO_2
Clays	11.85	54.14	26.52	1.44	1.27	0.60	0.16	2.37	0.74	0.08	0.82
Loams	11.94	57.71	12.73	4.65	7.60	1.62	0.33	1.79	0.81	0.10	0.71

Table 2. Granulometric composition of clays and loams.

Sample	Content of fractions, mm, %					Name of the group
	> 0.06	0.06-0.01	0.01-0.005	0.005-0.001	< 0.001	
Clays	3.83	7.37	8.90	15.22	64.68	highly dispersed
Loams	12.78	29.65	8.93	14.03	34.61	low dispersed

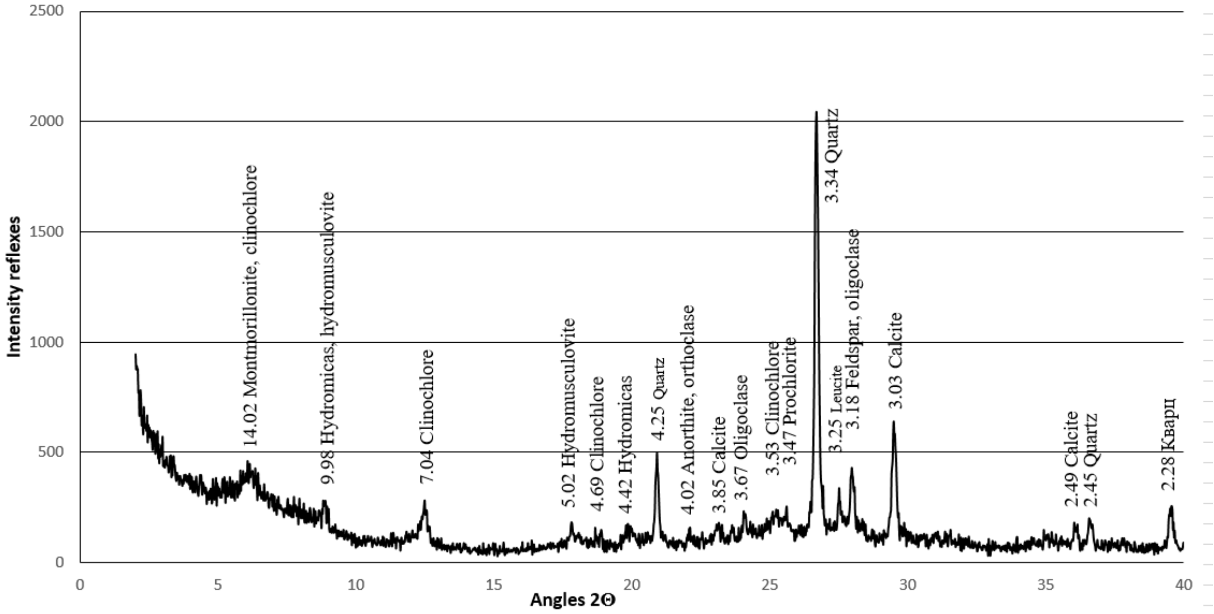

Fig. 3. X-ray diffraction pattern of the clay from Vladimirovsky deposit.

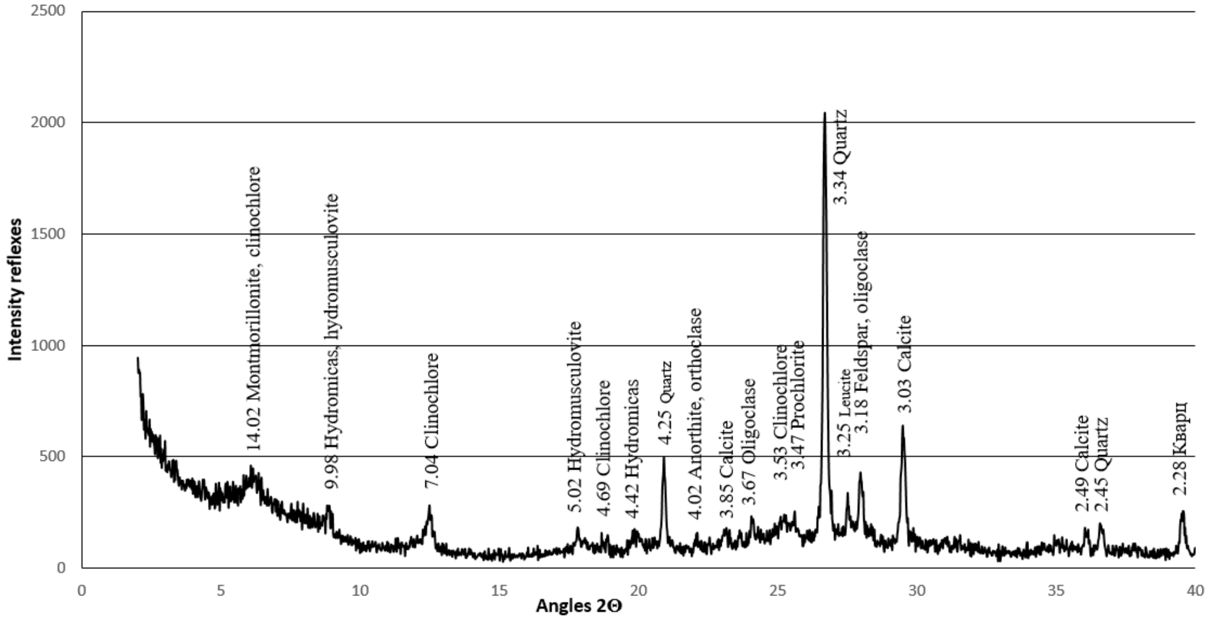

Fig. 4. X-ray diffraction pattern of the loam from Aksay deposit.

As can be seen from the presented data, clays and loams differ significantly from each other in chemical, mineral and granulometric composition. The clays contain a significantly large amount of clay minerals, which is reflected in the granulometric composition. The loams do not contain kaolinite but contain calcite and a larger amount of quartz of the sandy-dust fraction. A higher amount of alumina in the form of kaolinite makes the clays more refractory. Such differences in composition naturally affect the technological properties, which will be discussed below.

3. RESULTS AND DISCUSSION

Our experiments allowed us to establish that with soft molding using the most common methods – acceleration and manual molding, the degree of deformation of the molding masses determined on a Pfefferkorn device should be 3-5 units, while the critical compressive stress is less than 1.0 kg/cm2. (Fig. 5, 6). During plastic molding, the main method of which for wall ceramic products is extrusion (non-vacuum, with mass vacuuming, rigid extrusion), the degree of deformation of the molding masses is on average 1.2-2.5 units, the critical compressive stress in this case can reach 20.0 kg/cm2 and higher – during rigid extrusion.

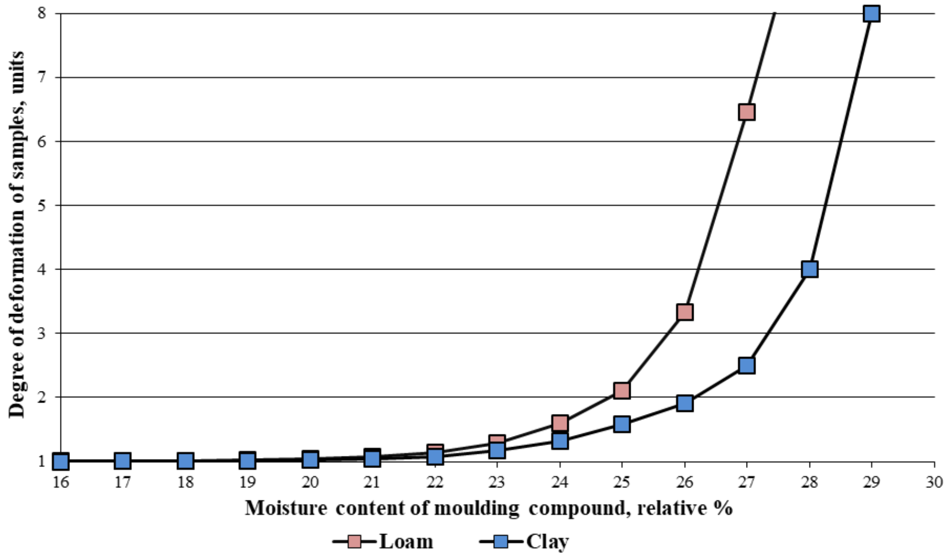

Fig. 5. Dependence of the degree of deformation of molding mixture samples based on loam and clay on moisture content.

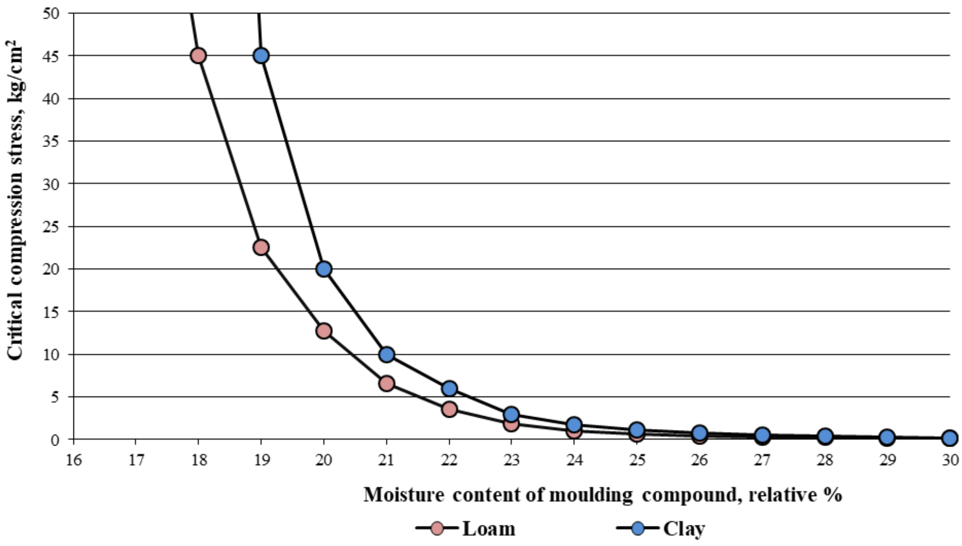

Fig. 6. Dependence of the critical compressive stress of molding samples based on loam and clay on moisture content.

Based on this, we prepared test samples based on molding materials with different moisture contents – conditionally for plastic and soft molding. For plastic molding, the moisture content for clay was 22%, and for loam, 21%. For soft molding: moisture content for clay – 28%, for loam – 26%. Based on the specified molding masses, comparative tests were carried out to determine the pre-firing technological properties with the determination of air shrinkage, binding capacity, determined by the bending strength of dried samples, sensitivity to drying, plastic strength and stickiness (Fig. 7-11).

As can be seen from the experimental results, the sensitivity to drying and the binding capacity do not change significantly with changes in the moisture content of the molding masses. Drying properties deteriorate slightly with increasing moisture content, but if the loam was highly sensitive to drying during plastic molding, it remained the same during soft molding. Also, if the clay was moderately sensitive to drying during plastic molding, it remained the same during soft molding. With soft molding, a small (≈ 5.5%) increase in the bending strength of dried clay samples is observed. Apparently, with increased moisture content, the effect of capillary forces is greater, which leads to greater density of the samples and, accordingly, strength. However, in our opinion, the granulometric composition plays a major role here, which either contributes to or does not contribute to the densest packing of particles during drying.

A significant difference is observed in air shrinkage. While air shrinkage for loam under plastic molding is 8.14%, under soft molding it is 9.88%. For clay, these figures are 8.83% and 10.56%, respectively. Such a significant increase in air shrinkage can be explained by the fact that if the moisture content of the raw mix by weight for loams during soft molding increases by 5% (from 21 to 26%), and for clay by 6% (from 22 to 28%), then by volume the increase in moisture content for loam already amounts to almost 7% (from 41.8 to 48.7)%, and for clay by 8.2%. In any case, air shrinkage values are quite high. In ceramic technology, the goal is to keep air shrinkage of products no higher than 7.0%; otherwise, drying conditions must be sufficiently gentle and prolonged. To reduce air shrinkage various technological methods are used, the most common of which is the introduction of leaners of a certain grain composition. It should be noted that generally accepted drying regime for ceramic bricks using the plastic (extrusion) molding method is 72 hours.

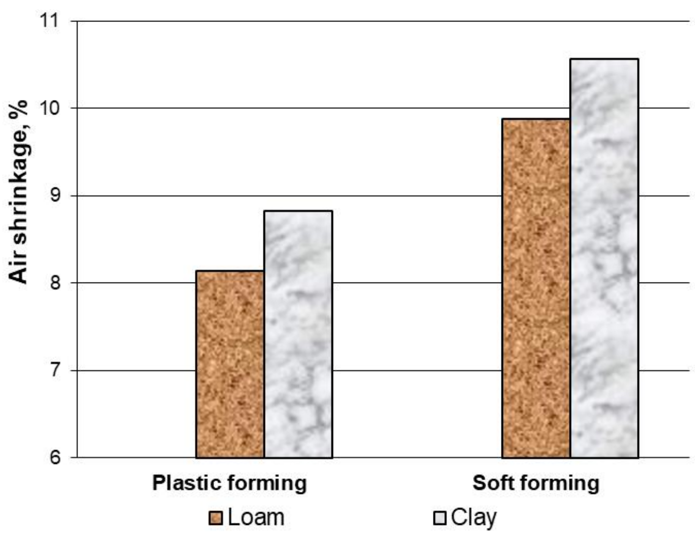

Fig. 7. Dependence of air shrinkage on the moisture content of the molding material.

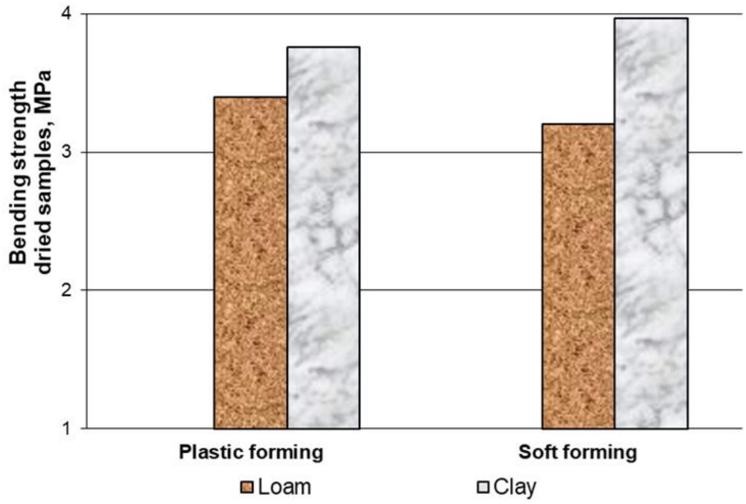

Fig. 8. Dependence of the bending strength of dried samples on the moisture content of the molding material.

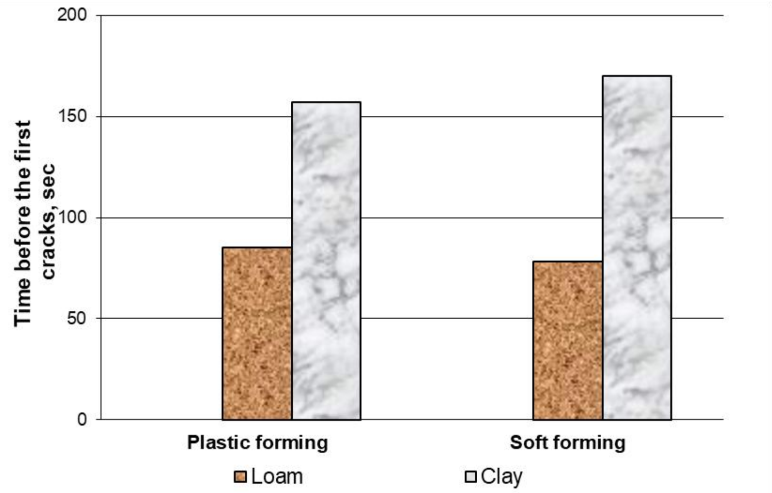

Fig. 9. Dependence of the drying sensitivity on the moisture content of the molding material.

Determination of the plastic strength of molding materials, as indicated above, can be carried out in various ways. Fig. 10 shows the results of determining the plastic strength according to GOST 34259-2017 adopted in engineering geology for clay raw materials for loams and clays with moisture content of molding masses for plastic and soft molding. As can be seen, plastic molding materials with the strength according to the classification of GOST 34259-2017 belong to the high-strength group. Molding masses for soft molding belong to the group with very low strength. However, it is important to keep in mind that, for both plastic and soft molding, plastic strength can vary significantly depending on the molding method (technological approach). For example, for plastic molding – vacuum-free extrusion and rigid extrusion molding materials. For soft molding – compression molding and vibration molding materials. From a technological point of view, a method for reducing plastic strength is important for soft molding. Simply increasing the moisture content is not entirely rational, since this will deteriorate other technological properties. Therefore, in our opinion, an important technological task is to achieve the required plastic strength with minimal moisture content. A reduction in moisture content can be achieved by introducing certain leaners, their optimal grain composition, the introduction of surfactants, electrolytes, and even the temperature of the molding mass. Also, in our opinion, it is important, based on practical and experimental data, to develop a method for determining the plastic strength of molding masses specifically for soft molding with specific loads and cone angles generally accepted for the plastometer. The plastic strength of molding materials is determined by the formula:

$$P_m = K_a \frac{F}{h_2},$$

where Pm is the plastic strength, F is the load, h is the cone penetration depth, and Ka is the cone constant. The constant depends on the cone angle and is calculated using the formula:

$$K_a = \frac{1}{\pi} cos^2 \frac{\alpha}{2} ctg \frac{\alpha}{2},$$

where α is the angle of the cone.

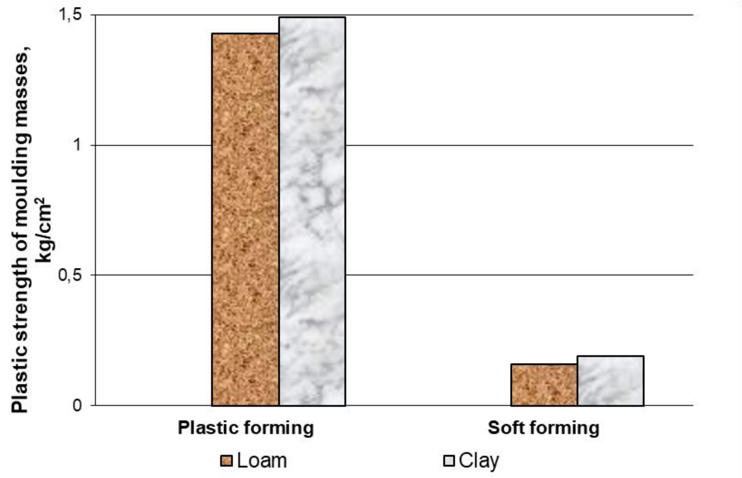

Fig. 10. Dependence of the plastic strength of molding compounds on moisture content during plastic and soft molding.

Stickiness of the molding masses is important in soft molding, which is not determined in standard tests of clay raw materials during plastic molding of samples and, accordingly, there is no approved method for determining this characteristic. The choice of technological method (technological approach) for molding, technological features of the molding process and the appearance of the brick's front surface depend on the stickiness. The stickiness of the molding ceramic masses depends on many factors which can be conditionally divided into internal and external factors. Internal factors include the chemical and mineral composition of raw materials, grain size (granulometric composition), quantity and composition of water-soluble salts, moisture content of the mass, etc. External factors include the material of which the adhesive object is made, the nature of its surface, the magnitude of the pressing load, and a number of other factors. The main technological task in selecting raw materials for soft molding is to achieve minimum stickiness at a given plastic strength. This is a rather difficult task, as it seems at first glance, since the specified plastic strength should, if possible, be achieved with minimal moisture content. Fig. 11, as an example, shows the dependence of the adhesion value (L) on the moisture content of clay and loam at a moisture content adopted for plastic and soft molding. The tests were conducted according to GOST 34259-2017 "Soils. Laboratory Method for Determining Stickiness," which is accepted in engineering geology for clay rocks. The stickiness index for soft-molding ceramic masses, as well as for clay soils, is the force required to tear off an adhered object from the molding mass at its certain moisture content. In engineering geology, stickiness is assessed based on the moisture content of clay soil at the onset of adhesion and the moisture content at maximum adhesion. The relationship is natural: with an increase in moisture content, the amount of stickiness increases to certain values and then decreases. Maximum stickiness is achieved in various types of clay soils, as well as in molding masses at different humidity levels. As can be seen from the results of the experiments, the stickiness of soft molding compounds is significantly higher in comparison with plastic molding compounds. Based on GOST 25100-2020

"Soils. Classification," plastic molding compounds can be classified as low-adhesive, while soft molding compounds are classified as moderately adhesive, or closer to highly adhesive. Moreover, the stickiness of loams is higher, which is apparently due to the large number of minerals from the montmorillonite group, as well as the increased content of a complex of exchange cations, namely calcium and magnesium.

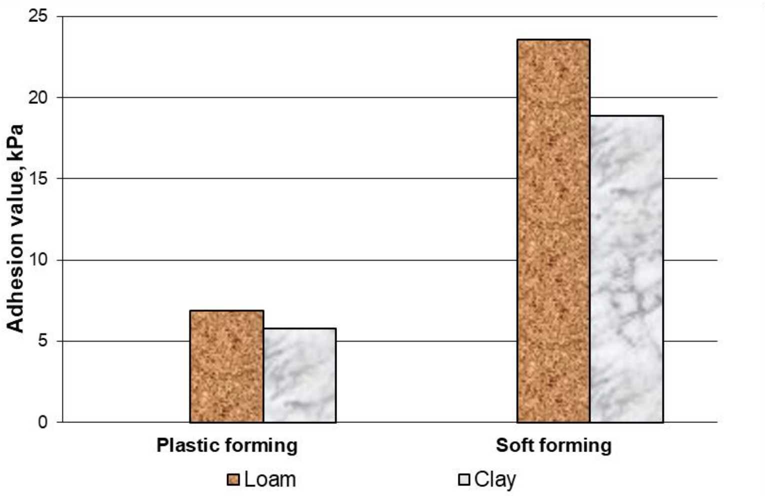

Fig. 11. Dependence of the stickiness of molding materials on moisture content during plastic and soft molding.

If we conditionally classify clay raw materials for the production of ceramic bricks by stickiness, taking into account the quantity and type of clay minerals, as well as dispersion and degree of lithification, then the following patterns can be identified. The more clay minerals clay rock contains, the stickier the rock itself is. Minerals with the highest degree of adhesion are those from the montmorillonite group, followed in descending order by hydromica, kaolinite, and minerals from the chlorite group. The highest stickiness is found in non-lithified water-soaked clays, and stickiness decreases with increasing degree of lithification: argillite-like clays → argillites → clay shales. It should be noted that the greatest stickiness for various types of clay rocks is observed in a certain humidity range. The stickiness depends on the dispersion of the rock, which is consistent with the mineral composition, since different clay minerals differ in size (Table 3) [15-27].

Table 3. Average particle size of different types of clay minerals.

Mineral group	Particle length, μm	Particle width, μm
Minerals of the kaolinite group	0.1-5.0	0.05-2.0
Minerals of the hydromica group	0.1-3.0	0.03-0.1
Minerals of the montmorillonite group	0.001-0.1	0.001-0.05
Minerals of the chlorite group	0.1-8.0	0.05-1.0
Mixed-layer minerals	0.05-1.0	0.01-0.1

As can be seen from the results of the experiments during testing of clay raw materials using the plastic and soft molding scheme of samples, the indicators for individual types of tests differ significantly. In this case, for soft molding, when testing clay raw materials and selecting raw material masses, it is highly desirable to introduce additional types of tests that are not used in tests according to the plastic scheme – the degree of deformation of molding masses, critical compressive stress, plastic strength, stickiness.

4. CONCLUSIONS

The analysis and the results of the experiments allowed us to draw certain conclusions and identify further directions for research.

1. When testing clay raw materials using the soft molding scheme of samples, it is necessary to develop a separate, generally accepted method that, in addition to determining the chemical-mineralogical and granulometric composition, determining plasticity, cohesion, air shrinkage, and sensitivity to drying, should include additional indicators: plastic strength (specific penetration resistance), degree of deformation, and critical compressive stress.

2. When selecting raw materials for soft molding, it is necessary to ensure that, for given plastic strength, degree of deformation, and critical compressive stress, the molding materials have as little air shrinkage as possible, are less sensitive to drying, and have a smaller degree of adhesion. Reducing these indicators can be achieved in various ways. As a rule, the best results are achieved with a combined approach.

3. When using traditional soakable clay raw materials for soft brick molding, molding masses, in order to achieve the specified technological properties, must contain an increased amount of leaners with a certain grain composition to achieve the densest packing at both the macro and micro levels, taking into account the grain composition of the clay raw material itself.

4. Hydromicaceous clays with a fairly high content of chlorites and relatively large particle sizes can be considered the most suitable for soft molding based on their mineral composition.

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