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Development of engobe coating compositions for clinker and facing bricks

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Abstract: This article deals with the basic principles of developing engobe compositions for facing and clinker ceramic bricks. The microstructure features of ceramic bricks have been studied, which must be considered when choosing the engobe composition and engobe products' technology. The expediency of using alkaline and substandard kaolins as the main raw material, which improves the conditions for sintering ceramic coatings by applying the single annealing technology of construction ceramics, has been studied. Due to its higher annealing reactivity, compared to traditional clay materials, the experimental raw material in the engobe composition improves the adhesion of the coating to the ceramic base and increases the strength of the decorative and protective layer.

Keywords: ceramic brick, engobe, burning, alkaline kaolin, water absorption

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Introduction

Ceramic brick is a building material implemented in a many design solution due to its wide colour palette, texture, and various laying techniques. One of the promising developments in the production of construction ceramics is the bricks' engobing (Becker et al., 2017). Even though engobed products gained popularity more than a decade ago (Nestertsov, 2004), many production processes remain insufficiently

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studied. This, in turn, limits the possibilities of ceramic brick manufacturers to produce quality products.

The process of matching a ceramic body and an engobe coating has not been sufficiently studied (Khomenko et al., 2019). Neither have the physicochemical processes occurring during single annealing of an engobe product, nor the algorithms for choosing the main raw materials and sintering additives, etc. Therefore, scientific research must be conducted into these processes for creating and implementing new types of decorative ceramic bricks, which are indispensable in terms of sustainability, reliability, and durability (Luangnaem et al., 2014).

The issue of shrinkage processes requires careful and complex processing (Khomenko et al., 2019) because the quality of an engobed brick is determined by several factors: the microstructure of the ceramic body; composition of engobes; slip parameters; application method, burning frequency, etc. In addition, the problems of the cost and availability of raw materials, energy consumption and the possibility of implementing engobing technology in traditional brick production are quite acute.

1. Purpose, aims, and research methods

The purpose of the article is to develop engobe coating compositions based on available domestic raw materials for ceramic bricks obtained by single annealing.

The aims of the article:

- to study the building ceramics microstructure and further application of engobe coatings on its surface;
- to establish the possibility of obtaining engobe coatings based on alkaline and unenriched kaolin;
- to study the consistency of the shrinkage indicators of ceramic masses and engobes.

Engobe slips were prepared by thin wet grinding of the components in a ball mill at a suspension humidity of 46.5%. Readiness was controlled by the residue on sieve No. 0063 (no more than 0.5%). To estimate the engobes' properties, cube samples were made from experimental slips by casting them into gypsum moulds. Engobe slips were applied to dried ceramic samples by the pulverization method. The engobe samples were dried at 70-90°C, after which they were burned at appropriate temperatures depending on the type of ceramic mass.

Standard methods for assessing properties: water absorption, shrinkage, and mechanical compressive strength have been used to reach the research aims. A Zetium X-ray fluorescence spectrometer (PANalytical B.V., the Netherlands) was applied to determine the kaolin chemical analysis. The mineralogical composition of raw materials was determined by a calculation method. A MBS-10 optical microscope with a magnification of up to 32 times was used to study the microstructure of the burned samples.

2. Results and discussion

2.1. Study of the construction ceramics microstructure

It is important to study the microstructure of the ceramics when selecting engobe coatings and providing product quality (Khomenko et al., 2018; Ryshchenko et al., 2009). The common, facing and clinker bricks' microstructures of different manufacturers were studied using an optical microscope.

The greatest porosity and the smallest amount of vitreous phase are found in common brick samples (Fig. 1): they are characterized by a rough structure and often the presence of visible and hidden defects. Considering the above, it is obvious that the common brick for engobing is not used.

The facing brick also has a rough structure, but it is characterized by a denser sintered ceramic body. The phase composition of such ceramics is more diverse since artificial and natural fluxes are specifically added to the ceramic mass.

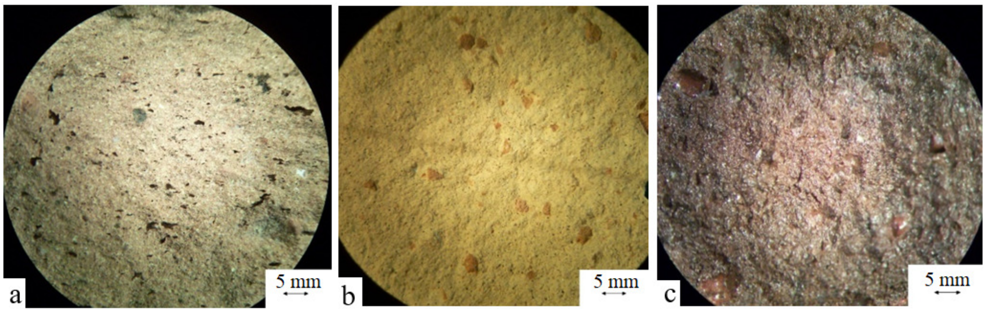

Fig. 1. Microstructure of ceramic brick: a) common, b) facing, c) clinker, optical microscope, magnification ×32 (*own research*)

We can say the same about the structure of clinker ceramics, with the difference that it contains an even more vitreous phase after sintering. As a result, after burning, a ceramic body is formed with minimal water absorption for coarse ceramics – no more than 5%.

Here, the additional vitreous phase on the surface of the ceramic body has a positive effect on the formation of a strong transition layer between the coating and the ceramic base, which increases the strength of adherence.

Therefore, when developing compositions and manufacturing technologies for engobe coatings, it is necessary to study the nature of ceramic mass sintering.

2.2. Study the possibility of obtaining engobe coatings based on alkaline and unenriched kaolins

The main focus in the development of engobe coatings was related to the use of available and inexpensive raw materials; ensuring high physical and ceramic properties of engobed products and the possibility of implementing engobed technology in large capacity production.

Engobe coatings for facing and clinker bricks must differ in sintering since annealing temperatures for clinker ceramics are usually 1150-1180°C and is higher than for facing bricks (950-1050°C). In this regard, we have chosen different sets of raw materials for the slips' composition in the engobe coating development.

2.2.1. Development of engobe coatings for clinker ceramics

Engobe coatings are a three-component system: clay material – skinny component – flux (Khomenko et al., 2019). Varying the quantitative and qualitative filling of the engobe composition allows them to be adapted to different types of ceramic bricks.

Alkaline kaolins are a more promising raw material for the implementation of engobe coatings in clinker bricks (Fedorenko et al., 2018). These are natural materials (Table 1), containing the above-mentioned three components – kaolinite, microcline and quartz in their chemical and mineralogical composition. Such kaolins are quite widespread and do not require enrichment.

Table 1. Chemical and mineralogical composition of alkaline kaolin [wt. %] (*own research*)

Deposit (region)	Oxide content							Mineral content		
	SiO_2	Al_2O_3	Fe_2O_3 + TiO_2	CaO + MgO	Na_2O	K_2O	l.o.i	kaolinite	quartz	microcline
Katerynivske (Donetsk)	74.3	15.2	0.6	0.9	0.7	4.7	3.6	26	42	32

Due to the low content of colouring oxides Fe_2O_3 and TiO_2, alkaline kaolins can provide high whiteness of the engobe coating after annealing, giving wide possibilities for obtaining various colour characteristics by implementation pigments without distorting the desired tone.

Alkaline kaolin from the Katerynivske deposit was chosen as the main engobe component (Table 2).

Table 2. Compositions of engobe coatings for clinker ceramics [wt. %] (*own research*)

Raw material	1	2	3	4
Kaolin alkaline	80.0	78.5	78.5	75.0
Refractory light clay	20.0	18.5	18.5	15.0
Glass cullet	–	5.0	–	5.0
Technical alumina	–	–	5.0	5.0

Studies have shown that unenriched alkaline kaolin has a complex effect during the formation of engobe coatings during burning. Firstly, the presence of quartz and kaolinite contributes to the expansion of the engobe coating sintering interval, which leads to a more complete course of the annealing process. Secondly, with the presence of an alkali mineral – microcline, the raw material acts as a “flux”: small feldspar grains begin to melt, increasing the amount of the liquid phase and contributing to the sintering of engobe coatings, which, in turn, results in their high density. Thirdly, the combination of these minerals in alkaline kaolin provides high engobe reactivity in a thin layer. A more liquid phase is formed and a densely sintered intermediate layer is developed when interacting with the ceramic base. In turn, the densely sintered intermediate layer strengthens the adhesion of engobe coatings to the ceramic product.

Figure 2 shows the main properties of the engobe coating materials’ samples. Samples from the clinker mass were prepared by the plastic method. The burning was carried out at 1180°C.

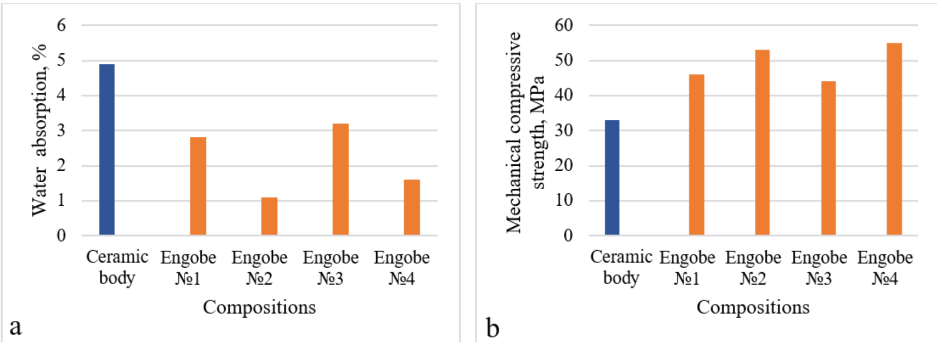

Fig. 2. Main test samples’ operational properties of clinker ceramics and engobes: a) water absorption, b) mechanical compressive strength (*own research*)

The given results indicate that after sintering the samples of engobe materials, water absorption and mechanical strength indicators are better than those for the ceramic body. So, for example, applying an engobe layer on the spoon and header surface of a ceramic clinker brick will reduce its water absorption by 1.6-4.5 times. This will increase the frost resistance and durability of products.

Therefore, the requirements of ДСТУ Б В.2.7-245:2010 standards, which apply to clinker ceramics, will be met for engobed bricks.

2.2.2. Development of engobe coatings for facing ceramics

When developing engobe coatings for facing ceramics, whose burning temperatures of which are usually lower than those for clinker ceramics, the most difficult issue is the choice of not only the main clay raw material but also the flux component. This is because the melting point of the most common and available natural fluxes (feldspars, nepheline-syenites) is above 1100°C (Esposito et al., 2005). Ultimately, the only effective and available flux at temperatures of 950-1050°C is a glass cullet.

Unenriched (or non-conditioned) kaolins can be used as the main raw material for engobes. These are cheaper than their enriched analogs because, after mining, they are immediately sent to production. Preference must be given to secondary kaolins, which have a relatively small amount of quartz component (up to 10-15%) as impurities. The clay mineral kaolinite in such raw materials has an imperfect crystal lattice, which in turn improves the sintering conditions of the ceramic material (Fedorenko et al., 2018).

Table 3 shows the chemical and mineralogical compositions of unenriched kaolin used in this study.

Table 3. The chemical and mineralogical composition of unenriched kaolin [wt. %] (*own research*)

Deposit (region)	Oxide content							Mineral content	
	SiO_2	Al_2O_3	$Fe_2O_3 + TiO_2$	CaO + MgO	Na_2O	K_2O	l.o.i	kaolinite	quartz
Polohivske (Zaporizhzhia)	49.0	35.5	1.8	0.2	0.3	0.3	12.9	86	14

Secondary unenriched kaolin plays the role of a "structure-forming" component and ensures the formation of a dense coating structure without signs of over-annealing and deformation. Refractory light clay in the engobe helps to improve the adhesion of the coating to the ceramic base.

Table 4 shows the **c**ompositions of engobe coatings for facing ceramics.

Table 4. Compositions of engobe coatings for facing ceramics [wt. %] (*own research*)

Raw material	1	2	3	4
Unenriched kaolin	55	55	65	65
Refractory light clay	30	25	20	15
Glass cullet	15	20	15	20

Figure 3 shows the main properties of engobe coating materials' samples. Samples from the mass of the facing ceramics were made by the plastic method. The burning was carried out at 1000°C.

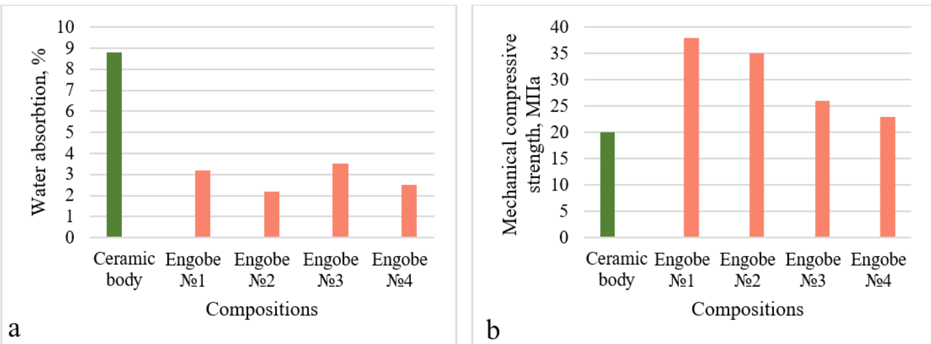

Fig. 3. Main test samples' operational properties of facing ceramics and engobes: a) water absorption, b) mechanical compressive strength (*own research*)

The given results are similar to studies on the development of engobes for clinker bricks. As a whole, after sintering samples of engobe materials at 1000°C, water absorption and mechanical strength indicators were better than those for the ceramic body.

Therefore, when applying these coatings to a ceramic product, the requirements of the DSTU Б В.2.7-61:2008 (EN 771-1:2003, NEQ) standards, spread to the facing bricks, will be met.

2.3. Study of the consistency of shrinkage processes of ceramic masses and engobes

To obtain a high-quality engobed brick, the most important thing is the consistency of the shrinkage indicators of the ceramic mass and the engobe. In this case, during annealing, the engobe is firmly "burnt" and adheres to the ceramic base through the creation of a developed transitional layer between the ceramic and the coating. Obviously, during such a process, the internal stresses arising from shrinkage deformations must be minimal, and the mass and coating shrinkage must be as close to each other as possible.

Figure 4 shows the ratio of ceramic masses and engobe coating shrinkage for clinker and facing bricks.

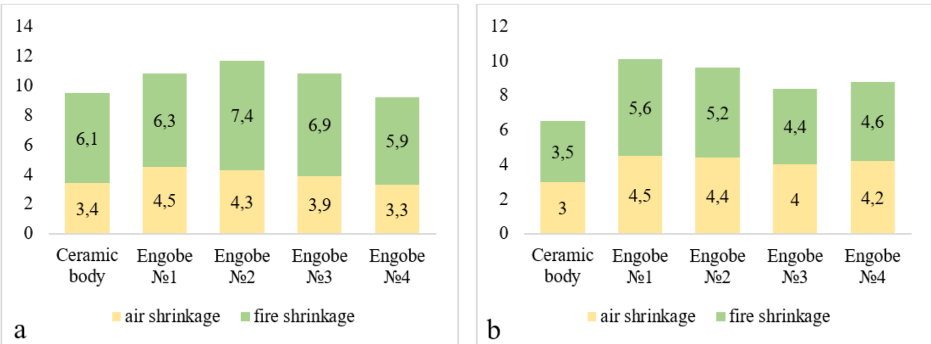

Fig. 4. Main test samples' operational properties of clinker ceramics and engobes: a) water absorption, b) mechanical compressive strength (*own research*)

From the above figure for clinker ceramics (Fig. 4a), we can conclude that engobe No. 4 is closest to the ceramic mass in terms of shrinkage processes. The difference in air and fire shrinkage of ceramic mass and engobe differs by no more than 10%, which is quite acceptable from the point of view of minimizing internal stresses that can cause coating defects.

For similar reasons, engobe No. 3 was chosen for the facing brick (Fig. 4b), although achieving consistency of shrinkage processes for the facing brick is more difficult than for the clinker due to the ceramic body's higher porosity.

The best engobes were applied to the appropriate dried ceramic materials and annealed at temperatures [°C]: clinker brick – 1180, facing brick – 1000. The samples had no defects and were distinguished by high whiteness.

Conclusions

In conclusion, we have shown promising directions for the development of engobe coatings for facing and clinker bricks.

Compositions of engobe mixtures have been developed in interaction with the ceramic base. The proposed coatings are designed for single annealing of construction products, which allows them to be produced using energy-saving technology. When applying the compositions of the developed engobe coatings and observing the established conditions of the technological process, it is possible to obtain high-quality products that meet the requirements of the standards.

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