DEVELOPMENT OF ENGOBE COATINGS BASED ON ALKALINE KAOLINS

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1. Introduction

Facing and clinker ceramic bricks are currently one of the most demanded construction materials [1, 2] and en Grove coatings make it possible not only to diversify significantly the assortment of this product, but also to enhance its physical and mechanical properties.

Production of high-quality en Grove coatings for construction ceramics is a rather complex process and largely depends on the choice of raw materials for the en Grove composition. The main requirements for raw materials are availability for multi-tonnage production and relatively low cost.

Alkaline kaolins, which along with quartz-feldspar materials [3, 4] found their use in manufacturing many types of fine ceramics, can be also promising complex raw material for the production of en Grove coatings. However, taking into account the diversity of alkaline kaolins, it is necessary to carry out complex studies of peculiarities of their application and state the basic dependences of the properties of en Grove coatings on the content of the raw material.

Thus, scientific research into the expansion of resource base for the industry of construction materials is relevant, since the existing formulations of ceramic masses and coatings include mainly high-quality clays, kaolins, feldspars, quartz sands and others. Natural supplies of such raw materials are rapidly exhausted, which negatively affects manufacturing the products. The choice of alkaline kaolins as promising raw materials for the construction industry takes into account their prevalence, availability, cost and ability to ensure large-tonnage production.
2. Literature review and problem statement

Traditionally, to manufacture fine ceramics and decorative coatings, including engobe coatings, clay or kaolins, quartz and feldspar are used as basic raw materials [5]. Clay materials provide the suspension forming ability of slips during fine wet grinding of the composition, adhesive ability of the coating to the ceramic base, the strength of the material in the dry and annealed state. Quartz sand is necessary to create a frame of the material, and during annealing it increases the viscosity of the melt and prevents the deformation of products. Feldspar or pegmatite act as fusing agents, i.e. ensure liquid phase sintering of ceramic material or a layer of engobe coating.

However, deposits of feldspar materials, especially of high quality, with a minimum content of colorizing impurities, are quickly exhausted, especially when they must meet the needs of large-tonnage technologies [6, 7]. That is why the issues of replacing feldspar materials in the compositions of ceramic masses or coatings are sufficiently acute.

Papers [8, 9] contain the results of research into replacement of feldspar materials in ceramics with cullet. It was shown that this component is an effective fusing agent in the engobe composition, which intensifies sintering of ceramic material. However, the issues of availability and the high cost of this raw material remain unresolved. The reason for this is that the needs of cullet by far exceed its amount; in this case, it takes selected cullet of transparent glass to obtain white engobe.

It is indicated in paper [10] that effective substitutes of feldspars are granite siftings. They also contain feldspar minerals that contribute to the intensification of sintering of ceramic material. But the problem of the content of colorizing impurities in this raw material remains unresolved. Due to the increased content of colorizing oxides Fe₂O₃ and TiO₂ in granite siftings, their application is limited in order to obtain white engobe.

In articles [3, 4], the use of alkaline kaolins in the production of tiles was shown, and in the studies of authors [12–14], raw materials were introduced into ceramic masses for the production of domestic, sanitary and electro-insulating porcelain. It is noted that the alkaline kaolins act as a sintering additive to the masses with the annealing temperatures of 1,180–1,200 °C and above. But the issue of using alkaline kaolins in the compositions of engobe coatings for construction ceramics remains unresolved in scientific literature. The fundamental possibility of the application of this raw material as the sintering component at lower annealing temperatures was not established and shrinkage processes in terms of the necessity of coordination of the coating and the ceramic base were not studied.

The above allows arguing that it is advisable to conduct research into alkaline kaolins as the alternative raw material in the production of engobes for construction ceramics. It is necessary to study the rheological properties of engobes based on raw materials, conformity of the coating with the ceramic base in the dry and annealed state, as well as the properties of the coating after annealing.

3. The aim and objectives of the study

The aim of this study is to develop the composition and to establish the physical-chemical foundations for the formation of the engobe coating based on alkaline kaolin intended for decorating ceramic bricks of single annealing.

To achieve the aim, the following tasks were set:
- to explore the basic properties of alkaline kaolins of Katerynivka, Maidan-Vilsky, Yosypivsky and Nemylniansky deposits;
- to study the rheological properties of engobe slips and physical-ceramic characteristics of engobes after sintering;
- to study conformity between engobe coatings and the ceramic base.

4. Materials and methods to study the production of engobe coatings based on alkaline kaolins

Alkaline kaolins of different deposits were selected as the basic raw semi-finished product for research. Other components of the engobes, in % by weight, included: 15 – light burning clay of Druzhkivka deposit (Ukraine), 10 – quartz sand of Avdivka deposit (Ukraine), 3 – cullet.

Raw semi-finished product, obtained by the extrusion method from the mixture of fusible clays was used in the studies as a ceramic base.

The grinding fineness of the engobe slip was determined by a change in the dimensions of samples – semi-finished products before and after drying and annealing. Water absorption of sintered materials was assessed by the amount of absorbed water in the open pores of the sample after saturation in vacuum and with the help of hydrostatic weighing [15].

Chemical analysis of alkaline kaolins and engobes was made with the help of X-ray fluorescent spectrometer Zetium (PANalytical B. V., Netherlands), and the mineralogical composition of raw materials was determined by the calculation method based on oxide content and qualitative X-ray phase analysis.

X-ray phase analysis was performed using the X-ray diffractometer DRONE-3 («Burevsnynk», Russia) using X-ray tubes with the copper electrode.

The character of the thermal transformations of ceramic masses was determined using differential-thermal analysis using the derivatograph by the system F. Paulik, J. Paulik, L. Erdey Q-1000 (company «MOM», Hungary).

To study the micro-structure of the annealed samples, the optical microscope MBS-10 was used at magnification x32 and the raster electron microscope «REM-106-I» at magnification x1,000–3,000.

5. Results of studying engobe construction ceramics

5.1. Studying the basic properties of alkaline kaolins

Kaolins are the products of weathering of granitoid rocks containing potassium feldspars and their partial kaoliniza-
tion. During these processes, due to the intensive removal of oxides of iron, calcium, magnesium and partly of sodium, alkaline kaolins are enriched with potassium oxide. The degree of changes of maternal rocks and the depth of their transformation into alkaline kaolin determines the chemical and mineralogical composition of the latter.

The chemical composition of the studied alkaline kaolins is given in Table 1. The studied kaolins contain from 3.4 to 5.4 % by weight of masses of oxides of alkali metals (Na₂O+K₂O), that is, in the composition of engobe coatings they will contribute to their sintering during annealing. Katerynivsky and Maidan-Vilsky kaolins have the smallest content of colorizing oxides (Fe₂O₃+TiO₂).

The results of determining qualitative mineralogical composition of alkaline kaolins using the X-ray phase analysis are shown in Fig. 1 and the calculated quantitative content of minerals is shown in Table 2.

The studied kaolins contain kaolinite, quartz and albite or microcline. The ratio of these minerals in each kaolin is different, so it is necessary to take this factor into account when designing the engobe compositions.

Differential-thermal analysis showed (Fig 2) that when kaolins are heated, in the thermograms, there are endo- and exoeffects, which differ by various temperatures and intensity.

Endoeffect with the maximum of 540–575 °C is associated with the removal of residual crystal-bound water from kaolinites. The area and the depth of the effects depend on the amount of kaolinite in the studied raw material: the greatest depth of the endoeffect is observed for Maidan-Vilsky deposit, in which the amount of kaolin is up to 46 % by weight. The smallest area of endoeffect is on the thermogram of Katerynivka kaolin (with the kaolin content of 25–27 % by weight). Yosypivsky and Nemylniansky kaolins additionally have the endoeffect associated with the destruction of the structure of mica minerals (610 °C).

The exoeffect at 930–970 °C testifies to crystallization of the primary mullite in raw materials. Maidan-Vilsky kaolin has the most intense exoeffect, since it has the highest content of kaolin, as well as Nemylniansky kaolin, which contains greater amount of titanium oxide, which is a catalyst of mullite crystallization. For the same reason, the temperature of the exoeffect in Nemylniansky kaolin is the lowest (930 °C).

### Table 1

| Deposit                         | SiO₂ | Al₂O₃ | Fe₂O₃ | TiO₂ | CaO | MgO | Na₂O | K₂O | L. a. f* |
|--------------------------------|------|-------|-------|------|-----|-----|------|-----|---------|
| Katerynivsky (Donetsk oblast)  | 74.3 | 15.2  | 0.3   | 0.3  | 0.7 | 0.2 | 0.7  | 4.7 | 3.6     |
| Maidan-Vilsky (Chmelnytska oblast) | 67.2 | 21.5  | 0.8   | 0.1  | 0.1 | 0.2 | 0.1  | 3.3 | 6.7     |
| Yosypivsky (Zhytomyr oblast)   | 72.5 | 16.3  | 0.8   | 0.4  | 0.8 | 0.4 | 3    | 1.2 | 4.6     |
| Nemylniansky (Zhytomyr oblast) | 69.1 | 18.2  | 0.9   | 0.8  | 0.6 | 0.6 | 1    | 3.7 | 5.1     |

*Note: L. a. f. – losses at firing*

### Table 2

| Deposit                | kaolinite | quartz | albite | microcline | mica | impurities |
|------------------------|-----------|--------|--------|------------|------|------------|
| Katerynivsky           | 25–27     | 39–42  | 0–1    | 30–32      | 0–1  | 0–0.5      |
| Maidan-Vilsky          | 42–46     | 18–20  | 0–1    | 27–29      | 0–0.5| 0–1        |
| Yosypivsky             | 26–30     | 37–40  | 19–22  | 5–8        | 0–1.5| 0–0.5      |
| Nemylniansky           | 27–32     | 33–37  | 4–6    | 27–30      | 1–3  | 1–2        |
Thus, different temperatures and intensities of endo- and exoeffects in the studied kaolins show their different activity and the ability to sinter during annealing.

5.2. Studying the rheological properties of engobe slips and physical and chemical characteristics of engobes after sintering

The engobe compositions of the same composition were prepared for the studies. Kaolin of Katerynivka deposit was introduced to engobe No. 1, kaolin of Maidan-Vilsky deposit was introduced to engobe No. 2, kaolin of Yosypivsky deposit was introduced to engobe No. 3, and kaolin of Nemylniansky deposit was introduced to engobe No. 4. At this, the chemical composition of engobes differed mainly (Table 3) by the content of refractory oxide Al₂O₃ from 17.91 to 23.08 % and oxide of alkaline metals Na₂O 1.55–3.68 % by weight and K₂O 1.16–3.73 % by weight, which cause different activity of sintering of engobes during annealing of the samples.

Table 3

| No. of engobe composition | SiO₂ | Al₂O₃ | Fe₂O₃ | TiO₂ | CaO | MgO | Na₂O | K₂O |
|---------------------------|------|-------|-------|------|-----|-----|------|-----|
| 1                         | 73.82| 17.91 | 0.42  | 0.50 | 1.09| 0.57| 1.95 | 3.73|
| 2                         | 70.19| 23.08 | 0.8   | 0.36 | 0.67| 0.58| 1.55 | 2.77|
| 3                         | 73.03| 18.86 | 0.79  | 0.58 | 1.18| 0.72| 3.68 | 1.16|
| 4                         | 70.77| 20.35 | 0.87  | 0.88 | 1.03| 0.87| 2.2  | 3.03|

Engobe slips having the moisture content of 40 % were ground in a ball mill to the residue on sieve No. 0063 of less than 0.5 % and their ability to be diluted by liquid glass and rheotan (Fig. 3).

Regardless of the type of electrolyte, the ability of engobe slips to dilute is preserved. The lowest content of electrolytes for ensuring the liquid state of suspension was observed in the engobes based on Maidan-Vilsky and Katerynivka kaolins. Thus, to obtain fluidity of 18–20 s, it was enough to introduce 0.48 % by weight of liquid glass or 0.41 % by weight of rheotan to engobe No. 2 based on Maidan-Vilsky kaolin. Similar figures were received for the engobes based on Katerynivka kaolin (0.5 and 0.41 % by weight of electrolytes, respectively).

Fig. 2. Thermograms of the studied alkaline kaolins of: a – Katerynivsky; b – Maidan-Vilsky; c – Yosypivsky; d – Nemylniansky deposits

Fig. 3. Indicators of fluidity of the studied slips at dilution: a – with liquid glass; b – rheotan

In order to determine the ability of the engobes to sinter, the samples-tiles were cast from slips, dried at 100 °C, annealed in a laboratory furnace at temperatures of 1,050–1,150 °C, and the indicators of water absorption of samples were measured.

Fig. 4. Indicators of water absorption for engobe samples after annealing at different temperatures

The most active sintering is characteristic for engobe No. 3 (water absorption after annealing in the studied range of temperatures is 7.9–3.1 %). This can be explained by the high content of sodium oxide in Yosypivsky raw materials, which promotes sintering of ceramic materials to a greater extent than potassium oxide. In addition, high reactivity of Yosypivsky alkaline kaolin is proved by the existence of endoeffect, associated with the destruction of the crystalline lattice of kaolinite at the lower temperature (540 °C, Fig. 2).

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Engobe No. 2 based on Maidan-Vilsky kaolin among all the studied kaolins is capable of sintering less actively – the indicators of water absorption of samples vary in the interval of 10.1—7.7 %, which is associated with the increased content of aluminum oxide in the engobe (23.08 %, Table 3). Engobes number 1 and 4 based on Katerynivka and Nemylniansky kaolin have the identical character of sintering.

The results of measurement of parameters of shrinkage of ceramic mass and engobe coatings are shown in Fig. 5.

The above figure shows that the indicators of air shrinkage of the ceramic mass and coatings differ slightly (maximum by 20 % for engobe No. 2). At the same time, fire shrinkage of the ceramic mass and engobe coatings differ more significantly. This is due to the fact that the coatings contain fusible agents, which contribute to the formation of strong engobe layer on the surface of the ceramic product. That is why the method of applying the engobe should take into account such difference to avoid internal stresses between the coating and the ceramic base.

5.3. Studying conformity between engobe coatings and ceramic base

The studied engobe slips (Table 3) were applied on freshly molded ceramic semi-finished product by pulverization method, which allows getting a thin layer of coating of equal thickness [18]. The use of this method was determined by the difference of fire shrinkage of ceramic mass and the coatings (Fig. 5).

The studied samples of the engobe ceramics were dried at 100 °C and annealed in a laboratory furnace at the temperature of 1,100 °C. The results of analysis of the engobe samples are shown in Table 4.

| Indicator                        | No. 1 | No. 2 | No. 3 | No. 4 |
|---------------------------------|-------|-------|-------|-------|
| Existence of microcracks on the surface | –     | single | –     | –     |
| Existence of chips on the surface | absent|       |       |       |
| Whiteness, %                    | 85    | 82    | 79    | 76    |

Fig. 5. Indicators of shrinkage of ceramic mass and studied engobes

The results of the influence of annealing temperature on the formation of the structure of engobe layer of composition No. 3 (the most compact) is shown in Fig. 7. It is clear that at 1,150 °C, such a dense structure of the layer is formed that there are almost no open pores. Those remaining single micropores with the dimensions of less than 5 microns, are safe, since water virtually does not get into the pores of this size during the operation, which prolongs the service life of products. At 1,050 °C, a rather dense structure is also formed, but the number and the size of the open pores is larger. Thus, in terms of forming the microstructure of the covering layer, it is an insufficient annealing temperature for the engobe to ensure the lengthy operation of the products.

Fig. 6. External view of engobe surface of the samples after annealing at 1,100 °C, magnification ×32: a – No. 1, b – No. 2, c – No. 3, d – No. 4

In general, the coatings looked good, except for engobe No. 2, which had recorded single microcracks (Fig. 6) that may have appeared due to the significant difference of shrinkages of the ceramic mass and the coating. Other defects were not detected.

The coatings had different whiteness indicators – engobe No. 1 (based on Katerynivsky kaolin) had the highest whiteness, engobe No. 4 (based on the Nemylniansky kaolin) had the lowest whiteness, which is associated with the existence in the latter of the largest amount (1.7 %) of colorizing oxides Fe2O3+TiO2.
6. Discussion of results of developing the engobe coatings based on alkaline kaolins

As a result of the conducted studies, the appropriateness of using alkaline kaolins as complex raw material in the preparation of engobe coatings for construction ceramics was established. Since these kaolins contain kaolinite, quartz and microcline/albite, their use in the compositions of engobe coatings will make it possible to a great extent to replace each of the separate components of the engobe.

It was found that alkaline kaolins, depending on the chemical-mineralogical composition, differ when it comes to thermal transformations (Fig. 2). Thus, the intensive destruction of the crystalline lattice of kaolinite at the lowest temperature (540 °C) occurs in Yosypivsky alkaline kaolin. That is, the particles of this kaolin have a low degree of orderliness and partly are «prepared by nature» to active sintering. Katerynivka alkaline kaolin has the highest degree of orderliness – active dehydration occurs at 575 °C. At the same time, active formation of primary mullite in Yosypivsky kaolin occurs at the temperature of 980 °C, which is the highest among experimental kaolins, it happens exactly for the same reason – a low degree of orderliness of kaolin particles. That is, the particles are more prone to dehydration and sintering than to the formation of a crystalline lattice of the new phase – mullite.

From the perspective of early mullite formation, Nemylniansky kaolin is the best. The cause of a low mullite crystallization temperature (930 °C) at a relatively low degree of orderliness of the structure of kaolin lattice can be related to the high content of titanium dioxide in the raw material, which is usually a catalyst of mullite crystallization [19]. But, given the conditions of operation of products, as well as the ability of mullite to re-crystallize after further heating at the temperature of annealing of products, mullite formation is not the goal in the formation of the covering layer of the engobe.

Comparison of the rheological characteristics of engobe slips that had the same quantitative composition, but different nature of alkaline kaolin, showed that the engobe slips are capable to be diluted by electrolytes. Having identical influence on all experimental engobe suspensions – optimum fluidity (17–20 s) was achieved when introducing 0.43–0.57 % by weight, rheotan appeared to be most effective. When using liquid glass as electrolyte, its amount for achievement of the specified fluidity indicators reached 0.6–0.7 % by weight for Nemylniansky and Yosypivsky kaolins. This is probably due to the existence of hydro-mica impurities in the composition of kaolin, which additionally adsorb moisture and decrease the effectiveness of the electrolyte.

But according to the most important indicator – water absorption, the engobe coating No. 3 based on Yosypivsky alkali kaolin is the best. This kaolin ensures the formation of the most compact structure in the studied temperature range: during annealing of the samples in the temperature interval of 1,050–1,150 °C, water absorption varies from 7.8 to 3.0 %.

The highest indicators of water absorption belong to engobe No. 2 based on the Maidan-Vilsky kaolin, which has the highest indicator of the amount of refractory aluminum oxide – 23.08 % by weight versus 18.86 % by weight for No. 3 based on Yosypivsky kaolin.

Engobe No. 3 is also the best by the shrinkage parameters. Air shrinkage for this engobe coating is almost completely consistent with the ceramic base (5.5 against 5.3 %) in the process of drying and forming a ceramic crock at the initial
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stage. At heating to temperatures of 1,100 °C, the indicator of fire shrinkage of the coating significantly exceeds fire shrinkage of the ceramic mass (3.4 versus 0.9 %) due to the more active liquid phase sintering, which deliberately flows on the surface of the samples. Engobe No. 2 based on Maidan-Vilsky kaolin has the greatest difference in air shrinkage with ceramic mass – up to 20 %, which may have caused the appearance of cracks on the surface of the sample of construction ceramics covered with this engobe. The merit of the conducted research is that it established the correlation between the chemical-mineralogical composition of alkaline kaolins and its influence on a number of the most important indices of engobe slips and coatings. The little known Nemyalianksy and Yosypivsky kaolins were studied, which allowed recommending virtually new raw material for ceramic industry. In addition, all parameters of production were carefully selected and explained.

The findings of these studies are useful and can be applied in the production of construction materials, including engobe ceramic bricks. Applying the formulations of the developed engobe coatings and observing the conditions of the technological process, it is possible to obtain interesting high-quality products. The above, in turn, allows expanding the assortment of construction ceramics and improving its competitiveness. In the future, this work can be directed to the expansion of the color range of engobe coatings.

7. Conclusions

1. The studies of the basic properties of alkaline kaolins showed that they represent the integrated raw material containing kaolin, quartz and feldspar minerals in different ratios. By the chemical composition, the studied kaolins differed mainly in the content of refractory aluminum oxide of 15.2–21.5 %, as well as the content of alkaline earth metal oxides – Na2O from 0.1 to 3 % by weight and K2O from 1.2 to 4.7 % by weight. The difference in the chemical and mineralogical composition of kaolins causes their different reactivity in heat treatment, recorded by different temperatures of endo- and exoeffects on the thermograms of raw materials.

2. The research into rheological properties of engobe slips revealed that the engobes based on Katerynivka and Maidan-Vilsky kaolins have the best dilution by liquid glass, the engobe based on Yosypivsky raw materials demands larger amount of kaolin. The most effective electrolyte for all the engobes was rheostan. The research into physical-ceramic properties of engobe revealed that the values of air shrinkage of engobes and ceramic masses differ insignificantly (except for composition No. 2). This minimizes the occurrence of internal stresses in the intermediate layer between the coating and the ceramic base. Engobe No. 3 based on Yosypivsky kaolin has the lowest indicators of water absorption during sintering under the same conditions, because it has an increased number of oxides of alkaline metals, which act as fusing agents.

3. The analysis of conformity between the ceramic base and engobe coatings showed high quality of coatings No. 1, 3 and 4, which had no defects associated with inconformity of the mass and the coating. According to the whiteness indicator, engobe No. 1 based on Katerynivka alkaline kaolin appeared to be the best.

Thus, according to the complex of the specified indicators for the studied alkaline kaolins and engobe coatings, the engobe coatings based on Katerynivka and Yosypivsky alkaline kaolin were found to be the best.

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