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Engobed ceramic brick used in the energy and resource-saving technologies of the construction industry

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Abstract: The article discusses the production and role of engobed bricks in the construction industry in view of the global trend towards resource and energy conservation. Standard methods for determining properties, as well as X-ray fluorescence, dilatometry, thermal and petrographic analyses, were used in the study. It has been established that for samples of ceramic bricks fired at 950-1000°C, the application of an engobic layer reduces the water absorption of the front surface from 14.5-1.2% to 6-2% and masks the undesirable colour. This makes it possible to use cheaper raw materials for brick production. The thermal conductivity of engobe bricks decreases from 0.48 to 0.43 W/(m·K), which opens up broad prospects for further development of energy-efficient coatings. An engobe was found to be optimal when produced with the following set of properties, wt.%: refractory clay – 65; quartz sand – 10; container glass – 25.

Keywords: ceramic brick, engobe, burning, water absorption, thermal conductivity

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Introduction

Ceramic bricks are one of the most common materials used in the construction of industrial and residential buildings. The products have high physical and mechanical properties, provide an optimal indoor climate and do not emit toxic substances (Ibrahim et al., 2022).

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Due to its porous structure, ceramic bricks have thermal insulation properties, which makes them indispensable in countries with both hot and cold climates. In combination with other structural thermal insulation materials, the use of ceramic bricks allows for effective regulation and optimisation of the heating or cooling needs of a building; an extremely important issue in the current energy-saving climate (Ujma & Jura, 2021; Ujma & Pomada, 2019).

To achieve porosity, organic additives are often introduced into the composition of the ceramic mass, in particular, sawdust (Thalmaier et al., 2020), fly ash (Beal et al., 2019), rice husk ash (Görhan & Simsek, 2013), paper waste (Sutcu & Akkurt, 2009), among other materials. The organic nature of these wastes requires energy consumption to release their calorific value during combustion in the ceramic firing process. However, the fuel consumption during firing is generally reduced. In addition, the combustion products give the fired bricks a more porous microstructure. This reduces the density of the products and improves their thermal insulation capacity.

The porosity of ceramic bricks is also increased by the addition of low-grade lean clay (Desai et al., 2023) and industrial waste, such as marble dust (Segadães et al., 2005) and waste steelmaking slag (Farnood et al., 2018). The practice of using waste in brick masses is quite widespread and solves the issue of resource conservation, as it reduces the consumption of high-quality clay raw materials, the deposits of which are being rapidly depleted.

At the same time, an increase in the porosity of ceramic bricks and their thermal insulation capacity leads to a decrease in their functionality. In particular, the frost resistance of the products decreases, and thus their durability, which often prevents them from being used as a cladding material.

The optimal solution for maintaining the functionality and durability of ceramic bricks can be the use of surface engobation (Yatsenko et al., 2009). Engobe is applied to the front surface of raw ceramic bricks and subjected to a single firing. The presence of such a layer makes it possible to obtain a front surface of the product with a higher density than the ceramic shard itself. Engobes can also be used to create unique decorative effects (Khomenko et al., 2019).

However, in order to fulfil its decorative and protective functions, the composition of the engobe must be carefully matched to the composition of the ceramic tile, in particular in terms of shrinkage and thermal expansion (Khomenko et al., 2023). This is a challenging task, as the compositions of ceramic masses, ceramic brick production technologies, and properties of finished products are very diverse, and the technological parameters that affect the formation of the structure and properties are numerous. The development of dense coatings for ceramic bricks sintered at low temperatures (up to 1000°C) is particularly relevant, as the choice of fusible raw materials for the engobe charge is very limited.

1. Purpose, objectives and research methods

The aim of the following research was to develop an engobe coating for ceramic bricks with porous ceramic shards sintered at temperatures up to 1000°C and to establish their main parameters.

The following tasks were set:

- to justify the choice of the basic raw material system for the engobe charge,
- to study the shrinkage processes during drying of the engobed samples,
- to investigate the ability of engobes to sinter at 950 and 1000°C,
- to investigate the frost resistance of samples of engobed bricks,
- to investigate the thermal expansion of samples of engobed bricks,
- to investigate the thermal conductivity of the samples of the engobed bricks.

For the manufacture of the ceramic brick samples, a typical mass composition was chosen, which included, wt.%: red highly plastic clay – 50 and sandy loam – 50. The raw materials belong to the Sursko-Pokrivske deposit (Ukraine) of low-melting clay. The properties of the ceramic mass and the produced samples are shown in Table 1.

Table 1. Properties of the ceramic mass and fired samples* (*own research*)

Name of the indicator	Properties after heat treatment		
	at 70°C	at 950°C	at 1000°C
Air shrinkage [%]	7.3	–	–
Fire shrinkage [%]	–	5.8	6.2
Water absorption [%]	–	14.5	11.2
Mechanical compressive strength [MPa]	–	120	145
Temperature coefficient of linear expansion [$\alpha \cdot 10^{-7}$ °C^{-1}]	–	56	58
Thermal conductivity coefficient [W/(m K)]	–	0.41	0.48

* the samples were prepared in the laboratory by plastic moulding without the use of a vacuum operation

Engobes were prepared in the form of a suspension with a moisture content of 42-46% by fine wet grinding to a residue of 0.5% on a 63 μm mesh sieve. To determine the properties of the engobes, samples were formed by slicker casting in gypsum moulds, and also engobe slickers were applied to freshly formed ceramic brick samples by casting.

The experimental samples were fired at temperatures of 950 and 1000°C in an electric muffle furnace SNOL 30/1300 (Lithuania) in an air environment according to the modes (Fig. 1). The water absorption was determined by the change in weight of the engobed samples before and after saturation with water in a vacuum according to ASTM C373-18. The frost resistance of the engobed brick samples was determined by the number of freezing and thawing cycles that the front surface of the ceramic sample can withstand without destruction, from +15 to –15, according to EN 539-2.

Chemical analysis of the engobe components was performed by the X-ray fluorescence method using an ElvaX spectrometer (Ukraine). The temperature coefficient of linear expansion for the engobe coating samples was determined on 5x5x55 mm stick samples using a horizontal dilatometer DIL 402 Expedis Classic Netzsch

(Germany) in the range of 20-400°C. The thermal conductivity was measured using an HFM 436 device from Netzsch (Germany). To obtain test specimens with dimensions of 30x30x1 cm, masonry was simulated. The fracture structure of the engobed specimens and the thickness of the coating layer were determined using a stereoscopic microscope MBS-10 (Russia) in reflected light.

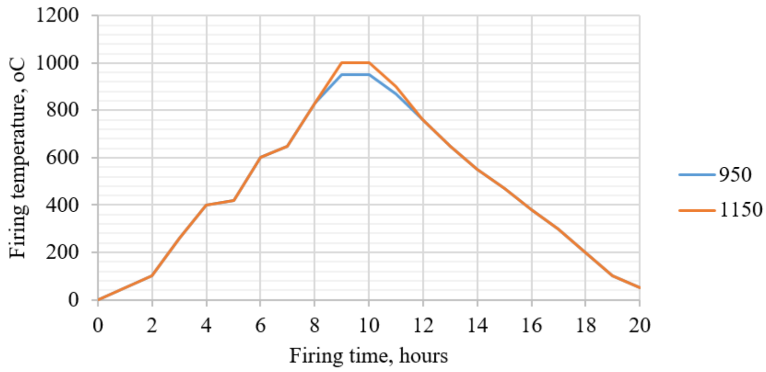

Fig. 1. Firing temperature regimes of the samples (*own research*)

The experiment was planned using the simplex centroid method for 7 points of the triangle of compositions in the system "refractory clay – quartz sand – bottle glass".

2. Results and discussion

2.1. Selection of engobe coating compositions

The compositions of engobe coatings for different types of ceramics are very diverse, but most often they are charge mixtures that include clay, leans and flux components. Clay prevents the deposition of stone particles and helps to form the body of the sintered layer. The lean component creates a "framework" and allows the regulation of shrinkage processes during drying and firing. Flux agents are needed to intensify liquid-phase sintering and produce a dense, durable material.

Engobe compositions were developed on the basis of a three-component system "refractory clay – quartz sand – bottle glass" (Fig. 2). The content of the components in this system was varied within the following limits [wt.%]: clay 80-50; sand 10-40; glass fraction 10-40.

The chemical composition of engobe components is given in Table 2.

The difficulty in the development of engobe coatings for ceramic bricks with a firing temperature of 950-1000°C is that it is necessary to ensure their low-temperature sintering with a sufficiently high content of refractory components – clay and quartz sand. Probably, the use of specially prepared frits with a low melting point in the composition of the engobe could solve the problem (Dal Bó et al., 2014), but the use of such raw materials would increase the cost of production too much.

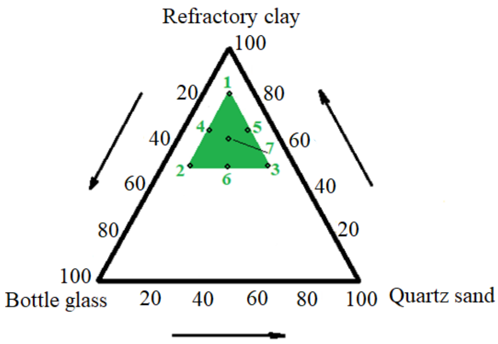

Fig. 2. Experimental compositions of engobe coatings (*own research*)

Table 2. Chemical composition of engobe components [wt.%] (*own research*)

Raw material name	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	losses during calcination
Refractory clay	51.1	34.5	0.7	0.1	0.5	0.5	2.0	0.5	10.2
Quartz sand	97.6	2.1	0.1	0.2	–	–	–	–	0.2
Bottle glass	73.3	3.5	0.6	–	2.9	3.7	–	16.0	0,2

The lower limit of the clay content (50 wt.%) is due to the fact that with its further reduction, engobe coatings tend to delaminate (Khomenko et al., 2023). The deposition of stony particles occurs, which causes additional internal stresses and provokes cracks on the surface of the coatings. For the same reason, the content of quartz sand is limited to 40 wt.%. It is also inappropriate to increase the content of glass fines above 40 wt.%, as this causes localised glazed "spots" on the engobated surfaces.

2.2. Investigation of shrinkage processes during the drying of egobated samples

Shrinkage processes during the drying of an engobed semi-finished product play a crucial role in obtaining a quality product. The engobed layer and the ceramic substrate have different types of ceramic structure (fine and coarse, respectively), so their shrinkage processes must be matched.

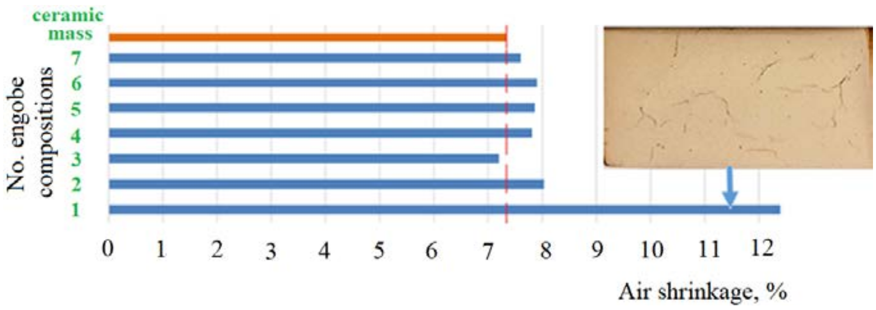

Fig. 3. Air shrinkage of ceramic mass and engobe coatings (*own research*)

Figure 3 shows the air shrinkage rates of the ceramic mass and the experimental engobe coatings when the engobe coating was applied to a freshly formed ceramic sample.

It was found that the largest deviation in air shrinkage was observed for engobe No. 1 with a clay content of 80 wt. %. This is due to the ability of clay particles to absorb water into the interpacket gap of their own crystal lattice (Richard et al., 2022) and release it during heat treatment. On the one hand, this ensures the cohesion of the engobe material to the surface of the ceramic sample. However, an almost twofold increase in the shrinkage rate of the engobe coating relative to the ceramic substrate leads to a high sensitivity of the product to drying and can cause cracks.

2.3. Investigation of the sintering processes of engobe coatings

One of the main tasks of firing engobe bricks is to ensure the formation of a face layer with a denser structure than the ceramic base. It is in this case that the engobe will perform its decorative and protective functions.

Changes in the fire shrinkage and water absorption of the experimental engobes after firing at 950 and 1000°C, depending on the ratio of components in the triangle field, are shown in Figures 4 and 5.

The data shows that the expected increase in the fire shrinkage of engobes occurs with an increase in the content of the sintering component – glass cullet. It intensifies the liquid-phase sintering of the engobe layer, resulting in an increase in its density. The minimum water absorption rate in the experimental three-component system can be achieved at 5.3% in composition No. 2 after firing at 950°C and 2% in compositions No. 2 and 4 after firing at 1000°C. Other engobes contain a fairly high amount of refractory components, such as refractory clay and quartz sand, so sintering at these temperatures is slower. Water absorption for engobes No. 1, 3, 5-7 is in the range of 9-12.8% after firing at 950°C and 8-11% after firing at 1000°C.

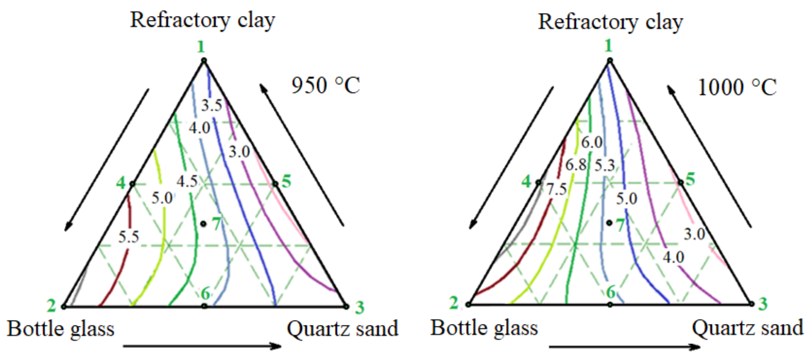

Fig. 4. Isolines of changes in fire shrinkage of experimental engobes depending on their composition at 950 and 1000°C (*own research*)

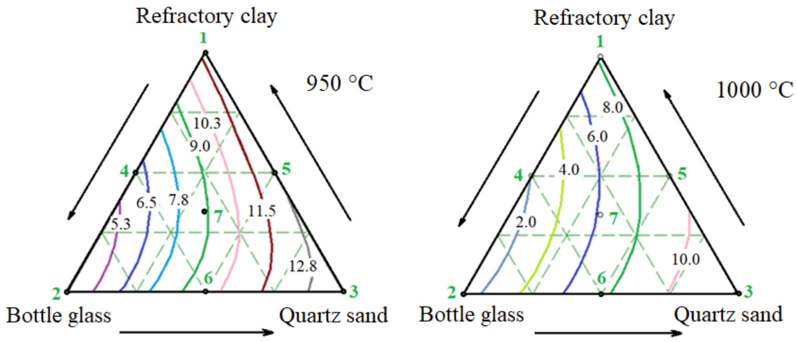

Fig. 5. Isolines of change in water absorption of experimental engobes depending on their composition at 950 and 1000°C (*own research*)

Thus, to obtain a densely sintered layer on the surface of ceramic bricks sintered at a reduced temperature, the most significant are the compositions of engobes No. 2 and 4.

2.4. Investigation of frost resistance in the engobed samples

By coating the face layer of ceramic bricks with an engobe, the goal is to reduce the ability of products to absorb moisture from the ambient atmosphere and extend their service life. One of the most significant properties of brick durability is frost resistance. The results of determining the frost resistance of the engobed brick are shown in Figure 6.

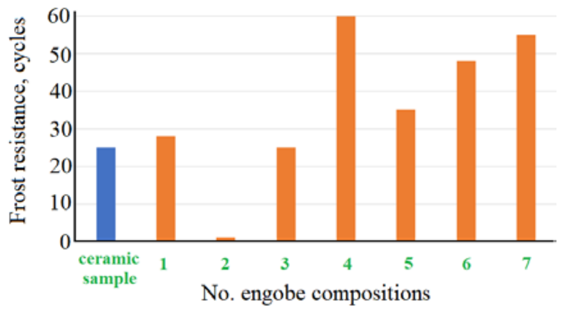

Fig. 6. Frost resistance of the samples after firing at 950°C (*own research*)

Fig. 7. Cracks on the surface of the experimental brick samples with engobe No. 2 after firing at 950°C (*own research*)

The data obtained indicate that the most frost-resistant sample were the brick coated with engobe No. 6. The samples with engobe No. 2 completely failed the test due to the appearance of a cracks on the engobed surface immediately after firing (Fig. 7).

The ceramic brick samples with more refractory engobes No. 5-7, despite the rather high water absorption of the layer, also have higher frost resistance indicators compared to ceramic samples without engobes. This can be explained by the finer structure of the coating layer, some of whose pores are impermeable to liquid droplets due to their small size.

2.5. Investigation of thermal expansion in the engobed brick samples

For any two-layer ceramic, the issue of matching the thermal expansion of the coating and the substrate on which it is applied is relevant. Even with a slight change in ambient temperature, coatings with different densities, phase composition and the ceramic product will expand differently when heated and contract differently when cooled. This will inevitably cause mechanical stresses in the contact layer, which in turn will provoke the appearance of cracks.

The results of determining the temperature coefficients of linear expansion of ceramics and coatings are shown in Figure 8.

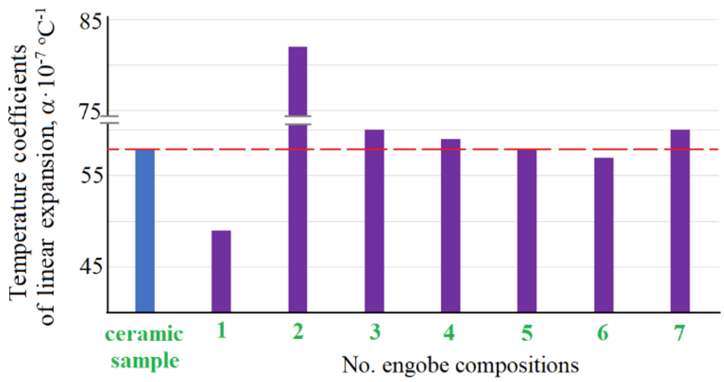

Fig. 8. Dependence of the temperature coefficient of linear expansion of experimental engobes on their composition after firing at a temperature of 950°C (*own research*)

It was found that the highest thermal expansion coefficient $\alpha = 82 \cdot 10^{-7}°C^{-1}$ was observed for engobe No. 2. Compared to the thermal expansion coefficient of ceramic bricks $\alpha = 58 \cdot 10^{-7}°C^{-1}$, the coefficient of engobe No. 2 differs by 30%, which led to the appearance of the cracks shown in Figure 6.

The temperature coefficients of linear expansion of other engobe coatings differ from ceramic samples by no more than 10%.

2.6. Determination of the thermal conductivity of the engobed bricks

Despite the fact that engobe is applied to the surface of a ceramic product in a thin layer, up to 250 μm (Fig. 9), it plays an important role in shaping their thermal and physical properties.

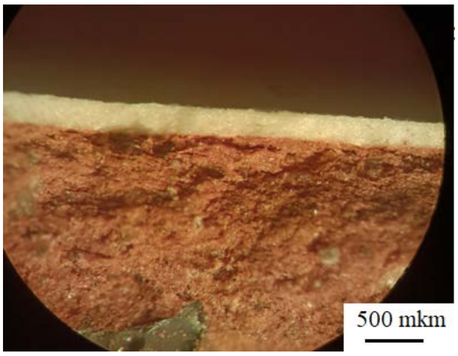

Fig. 9. Microstructure of an engobe ceramic brick specimen in a fracture, reflected light (*own research*)

Denser materials have a greater ability to conduct heat. However, engobed bricks are thicker than bricks without engobe, and the engobic layer actually provides additional thermal insulation. Therefore, in the end, the engobed ceramic samples have a slightly lower thermal conductivity (Table 3).

Table 3. Thermal conductivity of the test materials after firing at 1000°C (*own research*)

Name of property	Sample of ceramic brick without engobe	Sample of ceramic brick with engobe No. 4
Thermal conductivity coefficient [W/(m·K)]	0.48	0.43

In addition, engobe prevents the penetration of moisture into the ceramic tile, which also increases its thermal insulation properties (Azevedo, 2019).

Conclusions

The use of the engobation operation makes it possible to obtain, after firing at 950-1000°C, samples of ceramic bricks with a dense outer layer and a porous inner structure. Thus, the water absorption of the front layer of the samples was reduced from 14.5-11.2% (ceramic shards) to 6-2% (engobe layer). This makes it possible to increase the usage of bricks in energy-efficient construction.

The application of an additional dense outer layer while maintaining the porous structure of the brick samples leads to a decrease in their thermal conductivity from 0.48 to 0.43 W/(m·K) and an increase in the service life of the products due to an increase in frost resistance from 25 to 60 freeze-thaw cycles. The most rational choice for a decorative protective coating for porous ceramic bricks fired at temperatures of 950-1000°C is an engobe that contains, wt. %: refractory clay – 65; quartz sand – 10; bottle glass – 25. The characteristics of the coating are: air shrinkage 7.8%, fire shrinkage 5.4-7.6%, water absorption 6-2%, and a temperature coefficient of linear expansion (at 20-400°C) of $59 \cdot 10^{-7} °C^{-1}$.

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