

JSACE 1/14

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Foam Ceramics
Manufacturing
TechnologyReceived
2016/05/24
Accepted after
revision
2016/08/12

Development of Foam Ceramics Manufacturing Technology

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 <http://dx.doi.org/10.5755/j01.sace.14.1.15929>

Both dense and porous construction materials are widely used in civil and industrial construction. For example, lightweight concrete, cellular concrete, foam concrete and other types of lightweight concrete are used almost as widely as conventional concrete. However, for the production of walls made of ceramic materials, bricks with dense structure and hollow bricks still are the most popular building materials.

Lower expenses related to the production of building materials having necessary properties will lead to decreased production costs allowing to increase the production capacity due to the economy of raw materials, other materials and fuel. According to the calculations, today, when the thermal resistance for wall structures has increased 2.5-3 times, having an average density of wall material 900-1100 kg/m³ it is no longer possible to meet the requirements for the thermal resistance of the outer walls 460-520 mm in the climatic conditions of the Baltic region. It is necessary either to increase wall thickness or to use more efficient insulation materials which both are not cost-efficient solutions. One of the possible solutions for the given problem is reduction of the average density for the wall material. Economic efficiency due to the reduction of average density for the wall material is the following: production capacity of the material decreases, fuel consumption in the production process of blocks or bricks decreases, energy consumption on bricklaying for 1 m² of the outer wall decreases; less manual work, depending on the wall thickness, heat resistance in walls of the building increases.

One of the most ecological and efficient methods is foaming of ceramic slip mass; therefore this technology has been chosen for the given research focusing on the production method of porous ceramics by using foaming of ceramic slip mass. Porous ceramics with volume mass ranging from 341 – 799 kg/m³, porosity 25-85 % and compressive strength 0.5 – 1.1 MP was obtained. The following physico-mechanical properties of the specimens were tested: compressive strength, bulk density and water absorption. In the framework of this research porous ceramic material with improved properties has been obtained and production technology of the material has been developed.

KEYWORDS: foam ceramics, ecological material, insulation material, porous ceramics.

Introduction



Journal of Sustainable
Architecture and Civil Engineering
Vol. 1/ No. 14 / 2016
pp. 80-86
DOI 10.5755/j01.sace.14.1.15929
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The usage of wall and insulation ceramic materials is in a favorable position today. This is mainly related to the fact that the ceramic wall materials have such properties as strength, durability and resistance to the environment, chemicals and influence of micro-organisms. Therefore it is easy and convenient to use these materials in the construction process. Ceramic wall materials can have moisture-regulating properties thus contributing to healthy microclimate in the building. Due to the increasing focus on the energy saving and efficient use of energy coming from various sources in the last years requirements for the thermal performance of buildings has increased.

As it is known, conventional ceramic bricks has a relatively high thermal conductivity; therefore, in order to successfully compete in the building material market and to provide to the customers

options for building eco houses, manufacturers of ceramic materials have launched new ceramic materials with reduced thermal conductivity. These materials have both advantages and disadvantages compared to the conventional bricks. Increased efficiency for the ceramic wall materials can be achieved by reducing the construction costs, labor intensity and mass of the building as well as by increasing their thermal resistance by developing new types of ceramic materials and technologies allowing to that will reduce the material intensity and energy costs. Among the justifications for this kind of research activities are increase of fuel and energy costs, the finite nature of high-quality clay resources and increased operation and maintenance requirements for the modern wall materials.

One of the most promising trends for the increase of wall ceramic material efficiency is density reduction by creating a porous structure, which is based on the high strength of the baked clay matrix. Currently the wall ceramic material density reduction of up to 800 kg/m^3 is achieved either by raising product hollowness or by introducing burning-out or melting additives in the composition of ceramic paste. However, from the previous experience it is known that the compressive strength of the above mentioned hollow ceramic materials is up to two times lower compared to the initial products, even by using high quality- mortar. High hollowness of the products, pore sizes and shapes as well as mortars exposure character who acts as a wedge, partly flooding in blocks hollows and causing tensile stresses all are among the reasons of strength reduction in these products.

Solution for the wall ceramic materials efficiency problem is related to obtaining of the porous structure of foam ceramic materials and optimization of the technological parameters, which allows to obtain high performance products. The goal of present investigation is elaborating technology and mix content for producing foam ceramic.

Based on the review and analysis of former researches (Кролевецкий, 2005, Максимова, 2002., Хузагарипов, 2014) the raw materials composition for the foam ceramic productions was developed. Bandy clay from the Latvian clay deposit "Ane" was used as raw material. Chemical composition of the clay is presented in **Table 1**, where LOI – loss of ignition. Plasticizers of the ceramic mass - liquid glass and fine soda, foaming agents - PB-2000, and the structure stabilizer - polypropylene fibers or sawdust.

In the first stage it is necessary to create the conditions necessary for the raw clay aggregates dispergation process. It helps to create a stable clay suspension, which is characterized by high fluidity. This is achieved by adding the electrolyte additives. The mechanism of sodium silicate dilution in clay is based on the separation capability with colloidal silicon acid, i.e. polysilicate anion, formation.

In the second stage it is necessary to create porous structure of the obtained suspension. The foam and slip mixing takes place in a low speed mixer in order to avoid air release from the foam mass (Хузагарипов, 2014).

It is known that stable and coagulated clay suspensions are characterized by various sedimentation volumes, because the sediment densities, which are obtained by the stable and coagulated parts of suspension sediments, are significantly differed among themselves. Stable (sustainable) suspension settles slowly, and its sediments have dense structure because the falling particles are not sticky and are rolling free one behind the other while packing more densely, taking the minimum potential energy state. Coagulated suspension settles quickly and the resultant sediments do not have of dense structure, thereof the falling particles clump together into different randomly created positions, creating granulated aggregates.

By analyzing the scientific literature, it has been concluded that the clay materials with high or medium coagulation ability have carbonate impurities - calcite and dolomite - in the composition. Ca^{2+} and Mg^{2+} ions with good coagulation ability contribute to the clay particle coagulation (Кролевецкий, 2005).

Methods

Table 1

The chemical composition of clay, mass %

	Chemical compound	Molecular formula	% by mass
1	Silicon dioxide	SiO ₂	50,0 - 52,0
2	Iron (III) oxide	Fe ₂ O ₃	6,0 - 8,0
3	Aluminum oxide	Al ₂ O ₃	14,0 - 16,0
4	Titanium dioxide	TiO ₂	0,5 - 1,5
5	Calcium oxide	CaO	8,0 - 10,0
6	Magnesium oxide	MgO	4,0 - 5,0
7	Sodium oxide	Na ₂ O	0,5 - 1,5
8	Potassium oxide	K ₂ O	2,5 - 3,5
9	Sulfur trioxide	SO ₃	0,0 - 0,5
10		LOI	14,0 - 16,0

On the stage of ceramic paste preparation, standard 10-% soda and liquid glass solution are used for soaking of raw clay. It has been examined that the best raw material soaking degree is achieved using as an additive liquid glass 0.3-0.4% from the total mass (sodium silicate aqueous solution of silicate module - 2.0) and soda 0.2% from the total mass. In this way, the moisture content on the paste preparation stage can be reduced to 56-70%.

Introducing of calcined soda in the composition provides the strength increase of the plastic mass due to appearing of sodium silicate in the acquisition stage of semi-finished product. In the dehydration process of this product, while it is baked, the water vapour releases contributing to the formation of pneumo-thermal conditions and chipping sintering intensification (Максимова, 2002).

It is necessary to pay attention to the fact that the viscosity and slips fluidity as well are highly dependent from the water temperature. Higher water temperature leads to better fluidity. The optimum value ranges between 40-60 °C (Хузагарипов, 2014).

Regarding the ceramic mass polarization process it should be mentioned that anion active class synthetic surface-active agents were chosen as foaming agents for the experiment, as they are characterized by good stability (sustainability), foam formation and air capturing capacity.

Physico-chemical properties of the surface-active agents:

- _ Density 1000-1200 kg / m³ at the temperature range of +20 ... + 25 °C;
- _ Hydrogen index (pH) range from 7.0 to 10.0;
- _ In the solution with foaming agents volume 4%, the foam expansion is at least 7.0;
- _ Foam stability (steadiness)) at least 360.

It has been established that the surface-active agent absorption on the particle surface causes its hydro-phobisation. The particles which are coated with the hydrophobic surface-active agent sheath loses contact capabilities with each other. Increasing agent content on the particle surface it not only one, but several ion layers can be absorbed. This poly-layer coating causes granulated spatial structure formation, which make impossible partition formation with dense pores. For this reason, the partition formation collapses relatively soon leading to the collapse of the porous structure of the product (Кролевецкий, 2005).

Conducting research it has been concluded that the constant temperature rate drying period results in intense shrinkage of the product. Consequently, its heterogeneity leads to appearance of dangerous stresses and cracks. Therefore, gradual temperature increase is recommended, in this case from 40 °C to 60 °C.

In order to improve the raw product drying properties, e.g. to reduce product shrinkage and other deformations, reinforcing materials, such as sand, cardboard, sawdust, polypropylene fibers and

other fibers have been added to the composition of ceramic paste. Consequently, there is a huge amount of very fine fibers in the product with a maximum fiber length of 6-8 mm and fiber diameter 6-60 microns.

Developing of drying process parameters has been followed by the analysis of firing process characteristics.

Foam ceramic material sintering mechanism is complex; it involves heat and mass exchange as well as physico-chemical processes. It combines physical phenomena determined by the movement of mass and sealing kinetics and chemical processes, which directly related to the heat treatment regime, composition of raw components, gas environment and other factors. Clay sintering proceeds under the impact of optimum temperature. The essence of sintering phenomena is that the substance fills the empty space inside the granules and around them. In addition, grain surface area decreases and the contact zone between them increases in this process. The quantity of grains grows, the grid defects decrease and the existing stresses at the material contact zones are removed during the sintering process. However, exceeding the optimum temperature there is a risk of material porosity reduction due to exceeded sintering.

The bending strength of the ceramics depends on the firing temperature. The crystalline SiO_2 inclusions significantly affect the strength of the ceramics. Exceeding the optimal firing temperature, strength and density of the ceramics increases and the total thermal resistance product is reduced about two times (Матинян, 2011).

According to the research about firing process, an optimal firing temperature is 980-1050 °C (Черных, 2003). Based on previous studies about conducted on the respective clay (see table 1) firing temperature 1000 °C has been selected in a given research.

As mentioned above, the foam ceramic material firing process as well as heat and mass transfer processes occur simultaneously, which are considerably encumbered with the phase and physical and chemical transformations related to the technological factors from one side, including physico-chemical and technological properties of raw materials, nature and quality of impurities and additives, the product porosity and the average density value, etc., –and to the external thermal factors from other side, including temperature, gas environment speed and composition, product dimensions, firing oven type, etc. (Кролевецкий, 2005).

The optimum firing time exposing specimens to the maximum temperature is 2 hours. Extended firing time in the maximum temperature does not lead to the improved results.

After summarizing the theoretical material about foam ceramic production technology development, the compositions of raw materials were designed (see. Table 2). It contain seven components: clay (100%), forming sustainable structure and strength as the main raw material; liquid glass and soda (5% and 0.2% of the clay mass) acting as electrolytes to increase the fluidity of the clay slips, to facilitate the foam introduction into the slips; foaming agent (0.09% of the clay mass) - forming the porous structure of material; polypropylene fi-

Raw material, mass %	Mix Nr.1	Mix Nr.2	Mix Nr.4	Mix Nr.5
Clay	100	100	100	100
Liquid glass	0,3	5	7	7
Sodium carbonate	0,2	0,2	0,5	0,5
„PB-2000”	0,05	0,09	0,09	
„Fairy”				0,09*
Sawdust	2	2	2	2
The mass forming moisture	60	60	60	60

* During foaming process, sugar was added to the solution (0,01 % of the clay mass).

Results

Table 2

Experimental raw material compositions

bers and sawdust (2% of the clay mass) as structural stabilizers reducing drying shrinkage; and forming moisture - water (60% of the clay mass) intended for softening of raw material, dilution of electrolytes and for foaming of foaming agents.

The first two experimental groups of specimens were dried for 2 hours in natural conditions without additional loading pressure to reduce drying shrinkage and then for 24 hours in the oven at 50 °C. The third and fourth groups of specimens were not dried in natural conditions were placed immediately after the molding in the drying oven at temperature of 100 °C for 24 hours instead. The artificial drying is used to completely remove the excess water from foam ceramic mass and to avoid shrinkage cracks during the firing process. During the drying the sample size changes were observed - shrinkage in the horizontal direction and swelling in the vertical direction.

The main problem faced in process of conducting experiments using the foam method is its collapse without being able to create a sufficiently persistent frame, which should resist the force

of gravity. This could be improved in two ways; firstly, based on the pH value, when then anhydrous is added, secondly, considering the temperature increase. To resolve the problems, thermal swelling agent can be used which will prevent foam collapse.

According to the developed firing mode pore ceramic product samples were kept at the temperature range from 0 to 150 °C for 30 minutes to ensure that there is no excess water is left in the samples to facilitate the cracks. In the range of 150-800 °C temperature increase rate was 5 °C/min or 130 min in total, and in the range of 800-1000 °C - 2 °C/ min or 145 min in total. The samples were held at the maximum temperature 1000 °C for one hour, so that all samples were fully fired. Then it was followed by the cooling process with gradual temperature decrease.

The sample firing - cooling process curve (mode) is shown in Fig.1.

The sample of material, which was examined under a microscope, has fine porosity with average pore diameters being 0.70-1.52 mm. There are also some pores with smaller diameter or larger diameter. Pores have regular round shape (see Fig. 2). The walls among pores are about 0.36-0.70 mm thick. Picture of the obtained material in macro mode with a pronounced fine porosity is included in Fig. 3.

Analysis of the research results shows that the obtained ceramic materials whose raw material is characterized by poly-mineral composition has both solid-phase and liquid-phase sintering processes.

Fig. 1

The obtained foam ceramic material firing - cooling mode

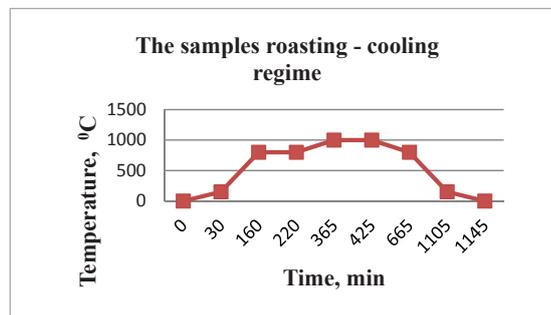


Fig. 2

The obtained foam ceramic material pore arrangement under the microscope. Magnification x4. Microscope lens: 4 / 0.1 _ 160 / 00.17

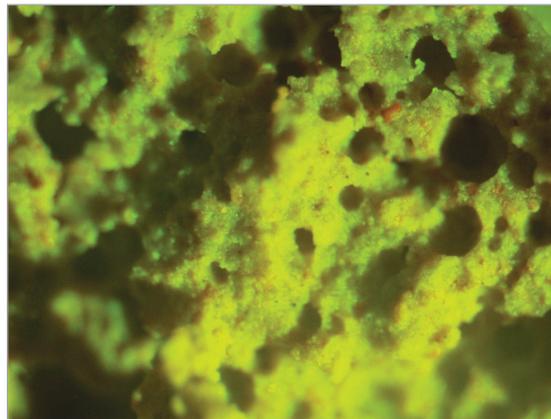


Fig. 3

The foam ceramic material. Photo in macro mode



The water absorption has been performed as passive absorption and measured as relation between weight of absorbed water and weight of dry specimen. The sample density and water absorption ratios are presented in Fig. 4. There is no observed strong correlation between water absorption and porosity of porous ceramic due to differences in the structures of pores. The opened pores have been observed for materials with high density. As results, the water absorption is higher for this specimens and reaches 86%. More light specimens with higher porosity have less water absorption due to closed structure of pores. Formation the closed structure of pores in light materials may be explained by physico-chemical properties and their interaction of clay and additives applied for porization of clay.

Compressive strength was defined in accordance with LVS 358:2003. Compressive strength test results are summarized in Fig. 5. The average strength of the samples varies between 0.3-1.1 MPa for densities 341 – 799 kg/m³. Obtained results are lower in comparison with reached strength by other researchers 3.1-9.3 MPa for densities

450-850 kg/m³ (Кролевецкий, 2005). Low strength may be explained by lacks in the technology of drying and firing, that have to be developed. Water absorption of obtained materials is in the range 13-86%, that is very close to results of other researchers 29-71% (Кролевецкий, 2005).

Increase of the liquid glass amount from 0.3 % of the total mass to 7 % improves fluidity and structural homogeneity of the mass but the third and fourth group of specimens with increased content of the liquid glass have lower strength, than the first and second group of the specimens (see Fig. 6);

In the framework of this research foam ceramic material production technology was designed. After conducting research on the production technological parameters as well as physico-chemical and physico-mechanical properties of raw materials, it has been concluded that clay materials tend to have coagulant structure allowing to develop sustainable raw product structure of foam ceramic material without plaster additives as well as to reduce time of the mass moulding process to 2-3 hours. The compositions of ceramic mass with various content of the liquid glass and surfactants have been developed, their main features have been assessed and optimal parameters for preparation, porization, drying and firing processes have been set.

By varying the content of foaming agents it is possible to obtain products with an average density of 341 – 799 kg/m³ and strength limits in the range of 0.3-1.1 MPa.

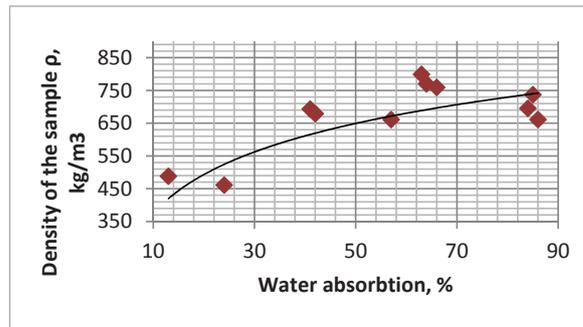


Fig. 4

The sample density and water absorption in graphical interpretation

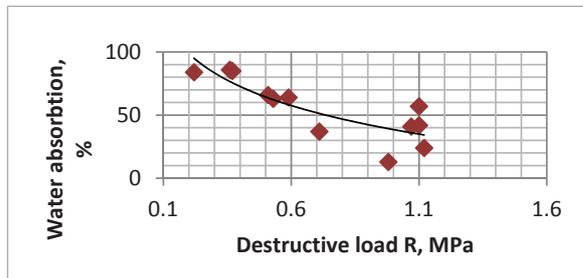


Fig. 5

Destructive load and sample water absorption in graphical interpretation

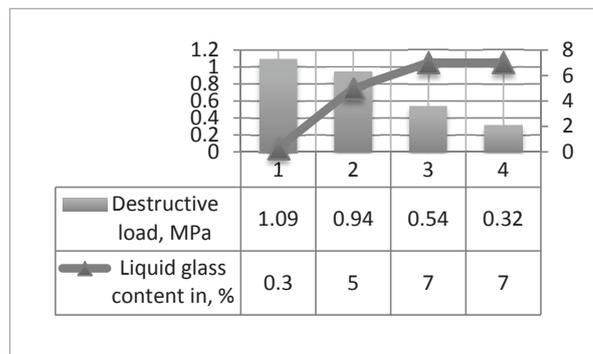


Fig. 6

Destructive load and compositions of specimens with liquid glass in graphical interpretation

Conclusions

Reduction of the foam ceramic material density by increasing the surfactant content is possible up to a certain limit. Excess surfactant content ($\geq 0.05\%$ of the total mass) does not give the expected results, and it is economically unjustified.

Foam ceramic material drying and firing processes are characterized by a number of features related to the production method, physico-chemical and technological properties of the clay raw materials and additives, nature of porous structure, etc. It has been concluded that the strength indicators of specimen groups, which were dried at the temperature $50\text{ }^{\circ}\text{C}$, reached higher values ($0.9\text{--}1.1\text{ MPa}$) when the internal structure of the material slowly releasing its free water has been stabilized leading to lower shrinkage compared to the specimen groups, which were dried at the temperature of $100\text{ }^{\circ}\text{C}$ ($0.3\text{--}0.6\text{ MPa}$) characterised by rapid evaporation of free water, shrinking of the internal structure and micro-cracking. As a result, drying shrinkage of all specimens does not exceed 20% .

In general, the obtained results are evaluated as positive. The direction is considered to be prospective and economically justifiable.

The financial support of the Latvian Council of Science, project Nr.Z12.0412 is acknowledged.

Acknowledgment

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