



# DEVELOPMENT OF EFFECTIVE TECHNOLOGICAL PARAMETERS FOR FORMATION OF A POROUS STRUCTURE OF THE RAW COMPOSITION IN ORDER TO OBTAIN A LIGHTWEIGHT GRANULAR INSULATION MATERIAL

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## ABSTRACT

The goal of the research is to develop the technological parameters for formation of a porous structure of the raw mass in order to obtain a lightweight porous insulation material using oil slime. The results of scientific and experimental studies of oil sludge were obtained. Efficient technological parameters for obtaining a lightweight porous structure of the material were elaborated. Swelling power and the formation of the structures in the studied composition are determined by the optimum combination of the components, determining the rheological parameters of the pyroclastic mass, which is determined by the special nature of the structure and composition of the crystal lattices of the minerals of the constituents. As a result of burning, oil slime forms a gaseous phase during the heat treatment and swells up the softened mass. The distinctive feature of the developed technological solutions is the fact that for the production of the heat-insulating material low swelling loess loams are used as the main raw material with the use of loam composition - a conglomerate mixture with oil slime as a fuel-containing, burning out and reinforcing additive. Addition of oil slime to the raw mixture reduces energy costs and provides swelling power and strength to the finished product using new technological solutions.

**Keywords:** oil slime, loess loam, bulk density, lightweight concrete, strength, porosity, energy efficiency, swelling, energy saving.

## 1. INTRODUCTION

Foreign practice and examples of domestic experience in the use of a large-panel system of buildings construction on the basis of introduction of modern innovative technologies show its high efficiency for accelerating the construction of economy-class housing. Almost in the entire range of buildings altitude, a large-panel house, by its cost, is cheaper by 20-25% if compared to a brick one, and by 15-20% if compared to a cast-in-place house. The most important factor in the successful development of industrial large-panel construction of houses is intensive development of the production of lightweight aggregates and effective thermal insulation and structural lightweight concrete on its basis [1].

Many authors have shown that lightweight concrete strength depends on the used lightweight aggregate, which leads to certain improvements and changes in the mechanical properties of the aggregates [2-5].

Nowadays expanded clay gravel remains the most effective among artificial porous aggregates. There are several reasons for that: cheap enough and abundant raw materials, high porosity and optimal structure of ceramic particles, deep theoretical study of the issue of production of expanded clay gravel and extensive practical experience in its production and use. However, there are a number of issues to be settled, such as expansion of the range of porous aggregates, the possibility to use local and non-swelling and low-swelling clays for production of granular material similar to expanded clay, and reduction of the swelling temperature [6-7].

Some of the issues, constraining the development of the production technology of lightweight granular thermal insulating materials, are high energy output of the technology and limited stocks of raw materials.

One of the aggregate production areas with the use of non-swelling and low-swelling clays is the introduction of swelling additives to the mass composition.

After the literature review and preliminary research work it was revealed that oil slime is one of the most promising components.

Oil slimes, produced in the process of oil and gas extraction, transportation of oil and oil products and their processing, are hazardous pollutants of surface and ground waters, soil and air. The amount of oil slime is constantly increasing. Storage of oil slime in basins causes complicated environmental problems; at the same time its oil part is a valuable organic raw material and its complex processing ensures preservation of natural resources [8-9]. Practical significance of oil slime use in the production of lightweight porous aggregates makes it possible to solve the following tasks on an international scale: energy saving, resource conservation and transition to a "green" economy, as well as, not the least, solving the environmental problem through utilization of oil slime in combination with loess-like loams.

The papers of foreign scientists were devoted to the problem of the use of oil and galvanic slime in the production of building materials [10-12].

Thus, scientists from Vilnius Technical University (Lithuania) have carried out scientific and



experimental research on the use of oil slime in the production technology of ceramic materials. The scientific results of the authors have shown prospectivity in the use of oil slime in ceramic production from the point of view of ceramic masses modification and improvement of physical and mechanical properties of the finished product.

The ceramic composition for production of porous aggregate is known; by weight it includes, in %: solid petroliferous product after oil slime separation - oil cake 10-30, inter-slate clay with the following percentage composition: SiO<sub>2</sub> - 38.3; Al<sub>2</sub>O<sub>3</sub> - 17.4; Fe<sub>2</sub>O<sub>3</sub> - 8.8; CaO - 6.2; MgO - 2.5; R<sub>2</sub>O - 4.1; LOI - 21.2 - 70-90. The technical result is the increase in strength during compression and the decrease in density [13].

The scientists have noted that introduction of industry waste as additives in certain amounts for production of lightweight aggregates improves physical and mechanical properties of the aggregates.

Use of oil slime as a raw material for various industries is one of the promising ways of its use due to the achievement of certain environmental and economic effects.

One of the important sectors in the economy of any country is production of construction materials. In the production of construction materials, high-temperature firing is the obligatory condition being associated with the high cost of energy (coal, gas, diesel, etc.).

These include production of ceramic bricks, expanded clay, agloporite, lime and cement.

That is why the goal of our research was the development of technological parameters of raw mass porous structure formation in order to obtain a lightweight porous insulation material with the use of oil slime.

In order to achieve this goal it is necessary to solve the following tasks:

- study the chemical properties of the loess loam at the Chagansky field;
- study the chemical and rheological properties of the oil slime in order to determine the amount of hydrocarbon and other compounds improving the firing and swelling properties of loess-like loams;
- implement scientific and experimental studies of the properties of ceramic masses and heat-treated samples on the basis of loess loams in combination with oil slime.

## 2. METHODS

As the main raw material were used the loess loam at the Chagansky field and the oil slime of the oil company "ZHAIKMUNAI", LLP in West Kazakhstan region.

The loam sample was taken for laboratory testing of 4 point samples. The weight of the point sample was 4 kg, point samples were mixed to obtain a common sample.

First of all the loam was dried to constant weight, then it was crushed in a laboratory ball mill MSHL-1P to the specific surface area of 1500-2000 cm<sup>2</sup>/g.

The swelling ratio of samples is determined by the following formula:

$$K = V_2 \div V_1 \quad (1)$$

where  $V_1$  - sample volume before swelling, cm<sup>3</sup>,  
 $V_2$  - sample volume after swelling, cm<sup>3</sup>.

In order to determine compressive strength of the materials, the tests were implemented on a PGM-500 MG4 pressure machine.

Thermal conductivity of the samples is measured using a thermal conductivity meter ITP-MG-4 "ZOND". Determination of water resistance is implemented with an instrument for water resistance determination VV-2.

Determination of the specific surface area of the powders is implemented using the instrument measuring the specific surface area of powdery materials T-3.

Determination of crystalline phases, particles and crystals size, the proportion of crystalline and amorphous phases was implemented with a transmission electron microscope JEM- 2100 of the "JEOL" company (Japan).

The study of surface topography and microstructure of different samples, qualitative and quantitative analysis of sample composition in the point area, construction of element distribution profiles along the selected line, receipt of element distribution maps from the selected section were implemented with a scanning electron microscope JSM-6390LV of the LEOL company (Japan).

Qualitative and quantitative phase analysis, determination of cell parameters and crystals orientation, analysis of polycrystals structures, microstrains and textures, quantitative elemental analysis of inorganic substances and materials, isotope analysis were implemented using diffractometric XPert PRO of the "PANalytical" company.

Study of the oil slime hydrocarbon composition was implemented using a gas chromatography-mass spectrometer Agilent 7890A/5975C (USA).

Chromatographic analysis was implemented according to the method specified in this paper [14].

Fractional composition of the studied oil slime was determined using the ARN-LAB-02 unit. Sulfur mass fraction in the studied oil slime was determined using the Spectroscan-Max GF2E unit (Russia).

The calorific value of the studied sample was determined with a calorimeter C2000 of the IKA-Werke company (Germany) with full combustion of the pre-measured weight in a bomb calorimeter in an environment of compressed oxygen and measuring the amount of heat released during combustion and auxiliary substances.

The content of mechanical impurities was determined by filtrating the studied oil slime with preliminary dissolution in petrol and settling washing



through a filter with a solvent, followed by drying and weighing.

The content of chloride salts was determined by titration of the aqueous extract with divalent mercuric nitrate in the presence of the diphenylcarbazide indicator.

### 3. DISCUSSION AND RESULTS

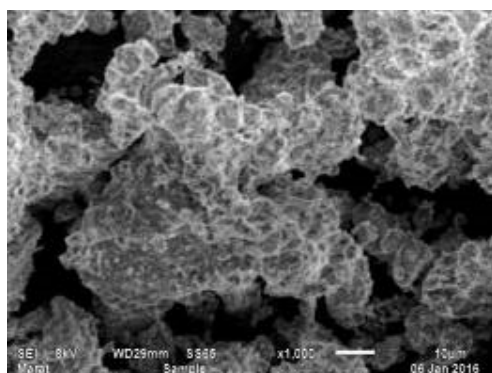
The average chemical composition of the loess loam at the Chagansky field in West Kazakhstan region is shown in Table-1.

**Table-1.** Average chemical composition of the loess loam at the Chagansky field in West Kazakhstan region.

| Raw material | Content of oxides, weight % |                                |                  |      |     |                                |                               |   |                 |                 |                   |                  |      |
|--------------|-----------------------------|--------------------------------|------------------|------|-----|--------------------------------|-------------------------------|---|-----------------|-----------------|-------------------|------------------|------|
|              | SiO <sub>2</sub>            | Al <sub>2</sub> O <sub>3</sub> | TiO <sub>2</sub> | CaO  | MgO | Fe <sub>2</sub> O <sub>3</sub> | P <sub>2</sub> O <sub>5</sub> | F | SO <sub>3</sub> | CO <sub>2</sub> | Na <sub>2</sub> O | K <sub>2</sub> O | LOI  |
| Chagan loam  | 55.1                        | 14.3                           | -                | 13.2 | 3.1 | 7.3                            | -                             | - | 2.8             | -               | -                 | 2.6              | 1.43 |

The loess loam at the Chagansky field contains up to 12% montmorillonite component in the form of mixed layers of formations with hydromica and kaolinite.

From the crystalline phase, clay also contains quartz  $d/n = 4.23; 3.34; 1.974; 1.813; 1.538 \cdot 10^{-10}$  m, feldspar  $d/n = 3.18; 2.286 \cdot 10^{-10}$  m, calcite  $d/n = 3.02; 2.018; 1.912 \cdot 10^{-10}$  m and hematite  $d/n = 1.839; 1.686; 1.590 \cdot 10^{-10}$  m. The loess loam microstructure is shown in Figure-1.



**Figure-1.** Loess loam microstructure.

Bottom oil slime from the tanks of “ZHAIKMUNAI”, LLP is used as a fuel-bearing, burning-out and hardening additive to reduce energy costs and make the maximum swelling and strength of the finished product using new technological solutions for production of lightweight aggregate.

The chemical composition of the oil slime was analyzed by gas chromatography-mass spectrometry method. Group composition of hydrocarbons according to the results of chromatography-mass spectrometry analysis, weight %: paraffins - 46.38; non-condensed cycloparaffins - 27.71; condensed cycloparaffins with 2 rings - 8.45; condensed cycloparaffins with 3 rings - 6.92; benzenes - 2.74; naphthenobenzenes - 0.10; binaphtholbenzenes - 0.10; naphthalenes - 3.66; acenaphthene - 2.96; phenanthrenes - 0.98.

Results of the study of oil slime rheological properties are shown in Table-2.

**Table-2.** Oil slime rheological properties.

| Indicator name                                | Values |
|---|--------|
| Density, kg/m <sup>3</sup> at 200°C           | 836.4  |
| Fractional composition, % vol.                |        |
| 200°C   | 11     |
| 300°C   | 39     |
| 350°C   | 54     |
| Sulfur mass fraction, %                       | 0.024  |
| Heat of combustion, kJ/g                      | 44.987 |
| Content of mechanical impurities, %           | 0.027  |
| Content of chloride salts, mg/dm <sup>3</sup> | 28.46  |

The research results show that the content of oil slime includes paraffinic naphthenic hydrocarbons and resins which improve the rheological properties of the ceramic mass. Due to the fact that with the temperature increase different individual hydrocarbons consecutively evaporate from the oil slime, this property has a positive effect on the firing temperature, i.e., reduces the firing temperature for the aggregates, thus saving energy.

The oil slime sample, obtained as a result of tank cleaning, was previously homogenized by mechanical mixing.

In order to ease introduction in the compositions with loess loam, oil slime with high viscosity is transferred into capillary-porous colloidal state by co-mixing with finely divided loess loam at a ratio of 1:4.

This operation transforms the oil slime into a loose conglomerate with a moisture content of 10-12% and provides a convenient composition for such subsequent technological operations as dosing and uniform distribution while mixing with the ground mass.

Ceramic compositions of the loess loam and conglomerate mixture of the resulting compositions are shown in Table-3.

**Table-3.** Ceramic compositions of the loam and conglomerate mixture.

| Composition number | Component, weight % |                                     |
|--------------------|---------------------|-------------------------------------|
|                    | Loess loam          | Conglomerate mixture with oil slime |
| 1                  | 100                 | 0                                   |
| 2                  | 95                  | 5                                   |
| 3                  | 90                  | 10                                  |
| 4                  | 85                  | 15                                  |

From the studied compositions a ceramic mass with molding moisture of 20-25% was prepared. Then, particles with fractions 5-10, 10-20 mm were produced, which were fired without pretreatment in a revolving

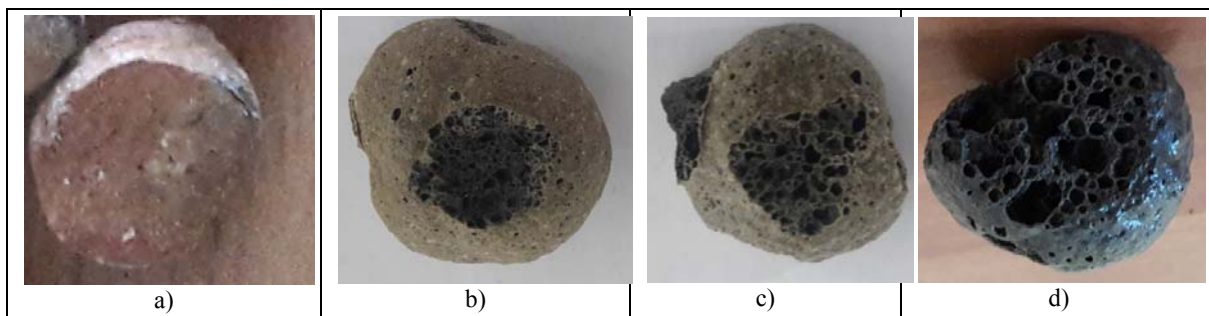
furnace RSR120-1000/13 according to the specially developed regime.

The results of experimental studies are shown in Table-4.

**Table-4.** Physical and mechanical properties of the studied samples.

| Composit ions No. | Swelling ratio | Firing temperature, °C | Bulk density, kg/m <sup>3</sup> | Crushing strength in a cylinder, MPa |
|-------------------|----------------|------------------------|---------------------------------|--------------------------------------|
| 1                 | 0              | 1150 ± 20              | 840                             | 5.7                                  |
| 2                 | 1.3            |                        | 642                             | 5.1                                  |
| 3                 | 1.5            |                        | 508                             | 4.9                                  |
| 4                 | 1.8            |                        | 490                             | 4.3                                  |

Figure-2 shows the fired materials with different content of conglomerate.

**Figure-2.** Fired materials with different content of conglomerate, %: a) of pure loam; b) 5; c) 10; d) 15.

The results showed that the fired material obtained from pure loess loam almost did not swell due to the fact that loess loam is low-swelling. According to the results of experimental studies, with the increase in the content of conglomerate mixture of the loess loam - oil slime by reducing the content of loam, a decrease in the bulk density from 840 to 490 kg/m<sup>3</sup> is observed. Low indexes of bulk density are observed in compositions No. 3 and 4 being within 508-490 kg/m<sup>3</sup>. Similar changes take place regarding the crushing strength in a cylinder. The minimum strength values are also observed in compositions No. 3 and 4 being within 4.3-4.9 MPa.

As for the swelling factor, the following is observed: with the increase in the content of conglomerate mixture of the loess loam - oil slime and by reducing the content of loess loam, the swelling ratio increases from 1.3 to 1.8.

Swelling of the loess loam in the composition of conglomerate mixture - loess loam with rapid firing

according to the elaborated regime determines their most important physical and mechanical properties and the structure of the finished product. As a result, a lightweight porous material with fine-mesh structure is obtained, which has low density with significant strength and high heat-shielding properties.

Swelling and structure formation in the studied composition are determined by the optimum combination of components, determining the rheological parameters of the pyroplastic mass. This is determined by special nature of the structure and composition of crystal lattices of the minerals of the components included in the group of quartz, kaolinite, hydromica and other.

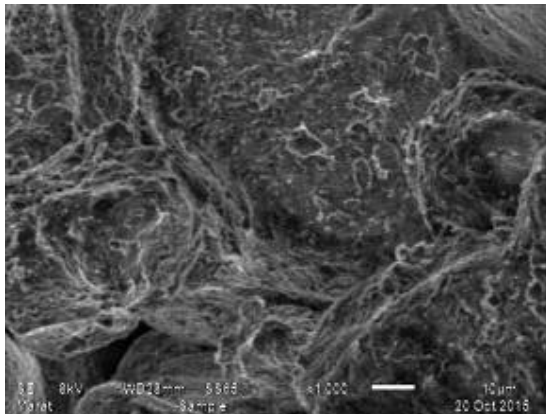
In addition, an important role in the processes of swelling and structuring is played by such accompanying minerals as mica, feldspar, iron and alkali-containing minerals as well as organic substances, which are present in the composition of loess loam and oil slime.





The regularity of the structure formation and swelling processes in the studied object was confirmed by the studies of the pores sizes (Figure 2), as well as its dense surface with the help of an electron microscope (Figure-3).

It should be noted that the dense sintered surface of the porous granular material represents the very positive effect, consisting in the fact that there is formed a waterproof surface.



**Figure-3.** Microstructure of the surface of the granular thermal insulation material.

Herewith, a special place during structure formation and swelling of raw material composition is taken by the content of oil slime, which forms a gaseous phase during heat treatment and swells the mass in the pyroplastic state.

The importance of chemical-mineralogical composition of the raw material composition in the system of loess loam - oil slime is in the fact that it predetermines a complex physical and chemical process of structure formation of the finished product, including phase transformations at the main stages of heat treatment.

The components of the considered system (clay minerals, organo-mineral part based on oil slime) directly take part and interact with each other in the formation of a porous structure with separation of the gaseous phase by combustion of oil slime, without which swelling and pore formation is impossible.

#### 4. CONCLUSIONS

The use of oil slime gives new technological advantages and physical and mechanical properties according to the following indicators:

- improves the rheological (structural-mechanical) properties of the ceramic mass by 50-60%;
- reduces the fuel - energy costs by 30-40% due to complete combustion as part of the ceramic mass; that is, reaching the temperature exceeding 400°C the mass generates heat due to combustion of oil slime and makes it possible to reduce the energy delivered from the outside;

- a porous structure of the finished product is obtained due to the swelling effect of oil slime. The swelling ratio is 1.3-1.8, and the finished product has a bulk density of (490-508 kg/m<sup>3</sup>).
- in the composition of loess loam – conglomerate (oil slime - loess-like loam), low-melting eutectic melts appear, providing the decrease in the temperature of pyroplastic state formation. As a result, the temperature of the pyroplastic state in the offered composition is within the temperature range of  $\pm 1150^{\circ}\text{C}$ .

As a result of the research, effective technological parameters for formation of a porous structure of the raw composition were developed in order to obtain a lightweight granular insulation material.

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