

**REPUBLIC OF AZERBAIJAN**

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**ABSTRACT**

of the dissertation for the degree of Doctor of Philosophy

**MODIFICATION OF CLINKER BRICKS  
WITH ULTRADISPERSE ADDITIONS**

Specialty: 3305.07- Construction materials and products

Field of science: Technique

Applicant: **Bulud İbrahim Baghirov**

**Baku - 2024**

The dissertation work was performed at the "Material Science" department of the Azerbaijan University of Architecture and Construction.

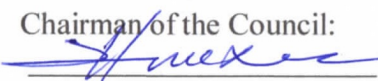
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## GENERAL CHARACTERISTICS OF THE WORK

### **The relevance of the topic and degree of development:**

Ceramic materials with high ecological and technical properties have been widely used in construction and household at all times and are still being used today. This is due to the resistance of ceramic materials to moisture, frost and atmospheric effects, high thermal-physical properties, and long service life. At the same time, ceramic materials have irreplaceable aesthetic properties such as texture and color. In the era of rapid development of construction works in our country, the demand for ceramic cladding materials from high-quality building materials is also increasing. As in other fields, in the ceramic materials industry, as the variety of products increases, the competition increases and the necessity of improving the quality of materials comes to the fore.

Clinker brick is one of the highest-quality ceramic facing materials used in paving the courtyards of parks and recreation centers. Also, one of the solutions to the actual problem of saving heat consumption in residential buildings is the use of facing bricks with high thermal insulation properties. It is known that the materials used in the cladding of buildings should be selected in such a way that they are sufficiently strong and frost-resistant, and at the same time have low thermal conductivity. Of course, the warmth and comfort created by ceramic materials cannot be created by any natural or artificial stone material. Clinker brick is one of the facing materials that are successfully used in the facing of building facades and sidewalks.

However, it should be noted that there are almost no high-quality hard-melting clays suitable to produce clinker bricks in our country (or such clay deposits have not yet been discovered). In order to obtain high-quality ceramic coating materials, the enrichment of clays based on local soft clays or kaolinized clays is currently one of the urgent issues for the ceramic materials industry in our Republic. It is advisable to use industrial products containing aluminosilicate imported from foreign countries to enrich the solid composition for clinker brick. However, to obtain high-quality clinker bricks based on local polymineral clays and ultradispersed additives

containing aluminosilicate, the theoretical, practical and scientific basis of these processes must first be developed.

**Object and subject of the study:** The object of the study is clinker brick. The subject of the study is the investigation of methods of improving the properties of clinker brick by modifying it with ultradisperse additives.

**Aims and objectives of the research:** The aim of the research work is to study the composition of clinker bricks, production technology and properties of the aggregate based on local polymineral clays and ultradisperse additives containing aluminosilicate.

To achieve this goal, the following issues should be resolved:

- Analyzing the modern state of the influence of the characteristics of clinker brick technology on its properties and formulating the research task;
- Studying the composition and properties of local raw materials (clays) and ultradisperse additives;
- Studying the dependence of the properties of clinker brick on the amount of ultradisperse additives used;
- Developing the optimal burning mode for clinker brick;
- Development of mathematical equations that determine the dependence of the properties of clinker brick on its composition;
- To confirm the reliability and longevity of the obtained material, studying its composition through modern physico-chemical analysis methods.

**Research methods:** In the research work, the physico-mechanical and physico-chemical (x-ray spectroscopy, radiography, differential-thermal analysis, microprobe and electron microscopy) research methods used in modern construction material science, and the processing of experimental results using the Box-Benkin method, which involves conducting active experiments was conducted. Experimental studies were carried out in the "Construction Materials Scientific Research and Testing Laboratory" accredited under the ISO 17025-2020 system under the "Materials Science" department of AzUAC.

**The main propositions defended:** The following theoretical and empirical propositions are defended:

- Requirements for the composition of ceramic slag for clinker bricks;
- Optimum aggregate composition of local polymineral clays and ultradisperse additives for clinker brick;
- The results of the study of physical and mechanical properties of clinker brick;
- The results of the active experiment of the processing of slag residue for clinker brick;
- Chemical, mineralogical and phase composition of ceramic material with high technical properties.
- The mechanism of formation of new minerals in the ceramic mass selected for clinker brick during thermal processing.

**Scientific innovation of the research:** For the first time, the technological principles of obtaining clinker bricks with high technical properties and the regularity of the formation of the structure of ceramic material based on ultradisperse additives containing local easy clays and aluminosilicate were determined.

1. The improvement of physical and mechanical properties of clinker brick obtained as a result of modification of the composition of ceramic slag with ultradisperse additives has been proven experimentally.
2. After experimentally determining the dependence of the properties of clinker brick on the composition, a mathematical model of this dependence was established, and the aggregate composition was optimized for high-quality brick.
3. The possibility of obtaining high-quality clinker brick with the use of ultradisperse additives has been proven by physico-chemical research methods. It was determined that the ceramic material with the optimal composition has a semi-crystalline phase composition and a mineralogical composition typical of densely structured ceramic pottery, which proves that the artificial stone material obtained on its basis has high mechanical properties.

**Theoretical and practical significance of the research:** The clinker brick technology was developed based on local softened clays and ultradispersed aluminosilicate-containing additives and a trial run was carried out in industrial conditions. As a result of the conducted research, the possibility of organizing the production of clinker bricks, imported from foreign countries by modifying local raw materials has been proven.

A technological regulation of clinker brick production was developed based on local polymineral clays and ultradisperse additives.

**Degree of integrity of research work.** Degree of reliability of research works. The reliability of the results of the conducted studies is confirmed by modern methods of physical and chemical analysis, widely used in materials science. Thus, mutually confirming methods of X-ray structural, X-ray spectral and electron microscopy in the composition of clinker brick revealed the presence of minerals in the composition of clinker brick, which impart high strength, density and frost resistance to ceramic materials.

The integrity of the results obtained in the dissertation work is confirmed by the application of research methods that have been widely approved in this field. Also, the obtained results do not contradict the general results in this field. In special cases, the results are confirmed by coincidence with the results obtained by other authors using other methods and methods.

The results of the conducted research were applied in the production of external floor tiles with high construction and technical properties at the "Ceramics and Pottery" factory.

Approbation and application: The materials of the conducted research were discussed at the following conferences:

**International conferences:**

1. "Radiation and chemical safety problems" International Scientific-Practical Conference, Baku. 2019.
2. "III International Azerbaijan-Ukraine Conference". Baku-Poltava. 2020. No. 3
3. "Proceedings of the 3rd International Conference on Building Innovations". Poltava. 2022.
4. International Conference on "Architectural Heritage of Karabakh and Eastern Zangezur". Baku. 2022.

5. "Russian science in the modern world" LX International Scientific and Practical Conference. Moscow. 2024. **National conferences:**
1. Republican Scientific-practical Conference on "Isolation problems in construction". Baku. 2022.
  2. Scientific-practical conference on "Prospects of the development of the transport-road complex of the Republic of Azerbaijan". Baku. 2017.
  3. Materials of XXIII Republican scientific conference of doctoral students and young researchers. Baku. 2019.
  4. "XXV Republican Scientific Conference of Doctoral Students and Young Researchers Dedicated to the Year of Shusha". Baku. 2022.
  5. "Transport, road and logistics in the Republic of Azerbaijan Development prospects of the complex", Materials of the Scientific Practical Conference, Baku. 2022.

**Personal participation of the author:** analysis of literature sources in the fulfillment of the dissertation work, setting the issue, forming new ideas, planning and carrying out experimental work, harmonizing principle-based results obtained by various research methods, the main leading role in the writing of articles and dissertation belongs to the author.

**The name of the organization where the dissertation work was carried out:** The dissertation work was carried out at the "Material Science" department of the Azerbaijan University of Architecture and Construction.

**The total volume of the dissertation is indicated by indicating the volume of the structural sections of the dissertation separately:** The dissertation consists of an introduction, four chapters, a bibliography with 117 names, and an appendix. The dissertation work is represented in 143 pages of computer text, including 42 tables and 22 figures and 3 chart. The main part of the research paper consists of 160654 characters, including 9084 45032 characters characters in c the introduction, hapter I, 38426 characters in chapter II, 51666 characters in chapter III, 11190 characters in chapter IV, and 5256 characters in the general results.

## MAIN CONTENTS OF THE WORK

**In the general description of the work**, the relevance of the topic of the research is justified, the practical importance of the work is emphasized, the main provisions defended, the research methods used during the performance of the work, the scientific innovation, approval and application of the work are explained.

**In the first chapter of the dissertation**, the characteristics of the production of clinker bricks - the literature research conducted in the direction of the characteristics of the raw materials and materials used in the production of clinker bricks and the effect of technological factors on the properties of the material were presented.

This section also examines the characteristics of high-quality clinker bricks and identifies the advantages of their use. Research on the application of ultradisperse particles in the production of construction materials, as well as in all directions of the industry, have been investigated in modern times, and the positive results of their modification of ceramic materials have been emphasized. At the end of this section, the works to be carried out in the direction of creating the physico-chemical and theoretical basis to produce high-quality clinker bricks in our country were defined, and the task of the research was determined based on this.

**In the second chapter of the dissertation**, the characteristics of raw materials and materials used for the obtaining of clinker bricks (their technological properties, chemical and mineralogical composition), physical-mechanical, and physical-chemical research methods used in the work are presented.

**In the study, clay from several fields was used to produce clinker bricks:** Ashagi Guzdak clay (Absheron region); Umbakı clay (Gobustan region); Pirigol clay (Shahbuz district, Nakhchivan MR); Kuku clay (Shahbuz district, Nakhchivan MR). Also, 2 types of ultradisperse mineral additives - fireclay and fly ash - were used.



The chemical composition of the used clays and ultradisperse additives is presented in table 1.

The study of the chemical composition of clays is of great importance in the selection of ceramic slag for clinker bricks. Because the suitability of clays for the production of clinker mortar is determined based on the amount of  $Al_2O_3$  in their content.

Clays with a content of this oxide in the range of 17-25% are considered suitable for clinker bricks, and the studied clays of Umbakı and Pirigol deposits belong to this type of clays.

**Table 1. Chemical composition of clays and ultradisperse additives used in research work .<sup>1</sup>**

№	Name of clay deposits	Chemical composition of clays								
		SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O +K <sub>2</sub> O	SO <sub>3</sub>	TiO <sub>2</sub>	Yİ
1	Ashagi Guzdak Clay	56,82	14,15	7,32	1,97	3,50	5,53	1,25	-	9,56
2	Umbakı clay	62,90	20,0	1,20	0,45	1,50	1,50	0,18	0,50	11,74
3	Pirigol clay	63,50	16,70	2,00	1,60	2,40	2,50	0,70	0,60	10,00
4	Kuku clay	65,50	13,50	1,20	1,30	1,75	6,30	1,50	0,10	8,85
5	Chamotte	64,38	17,58	3,07	3,21	5,17	4,76	0,46	0,63	-
6	Fly ash	62,67	28,38	1,63	0,56	4,10	1,06	-	1,6	-

The use of clays from other deposits without modification with high aluminosilicate mineral additives is not considered suitable for the purpose. However, the use of the clays of AgaGüzdak and Kuku fields used in the research in the composition of ceramic slag for clinker bricks is also related to their high content of alkali metal

<sup>1</sup> Study of the chemical composition of raw materials for the production of clinker bricks. Bagirov B.I., Shirinzade I.N.

oxides. During heating, an alloy is formed due to the interaction of alkaline oxides with  $\text{SiO}_2$ . In addition to ensuring the compaction of this ceramic mass, at the same time it intensifies the formation of mullite.

Chapter II also presents the classification of studied clays according to the amount of  $\text{Al}_2\text{O}_3$ . This classification was done for the first time for the clays of the studied deposits. As a result of the analysis, it was determined that the clays of the Umbakı and Pirigöl deposits are semi-acidic clays, and they can be included in the difficult-to-melt clay group due to their refractoriness.

At the same time, these clays are considered medium coloring clays due to the amount of coloring oxides in their contents. Because  $\text{Fe}_2\text{O}_3 + \text{TiO}_2 = 1.50 + 0.50$  in Umbakı clay; In Pirigöl clay, it is  $\text{Fe}_2\text{O}_3 + \text{TiO}_2 = 2.40 + 0.60$ . The amount of coloring oxides in Lower Guzdak clay is  $\text{Fe}_2\text{O}_3 + \text{TiO}_2 = 3.50 + 0$ . Lower Guzdak clay belongs to the group of clays with high coloring oxides.

In Kuku clay,  $\text{Fe}_2\text{O}_3 + \text{TiO}_2 = 1.75 + 0.10$ . The amount of  $\text{Al}_2\text{O}_3$  and carbonates in Kuku clay is less than other studied clays. The amount of  $\text{SiO}_2$  is higher. The clay of this deposit belongs to sandy clays, and for this reason, the fire resistance of this clay is lower than the clays of other deposits. It was determined by experiment that the fire resistance of the clay of the Pirigöl field is  $1520-1540^\circ\text{C}$ , and that of the Kükü field is  $1420-1460^\circ\text{C}$ .

In this section, the mineralogical composition of the used clays and ultradisperse additives is also presented. The X-ray method was used to study the mineralogical composition. It has been determined that the composition of clays is mainly composed of illite, kaolinite, vermiculite, albite and quartz minerals.

The radiosensitive part of the clays of each deposit is made up of the quartz mineral. The amount of quartz mineral in the clays used in the research varies between 30-60%.

Improving the properties of materials using ultradisperse particles is one of the promising directions in modern materials science.

This is due to the unique properties of small particles, which is explained by the increase in their surface energy. The absence of poorly soluble clays in the territory of Azerbaijan or the lack of minerals in the composition of clays that give them this property necessitates the use of materials containing aluminosilicates as ultradisperse additives.

The mineralogical composition of the ultradisperse additives used in the research was also studied. Through X-ray analysis, it was determined that the main part of the composition of fly ash consists of amorphous  $\text{SiO}_2$ .

This is evidence that it is chemically active and can easily interact with other components that make up the composition of the ceramic mass during thermal processing. At the same time, from the derivatograms of fireclay, the collapse of their crystal lattice is also observed.

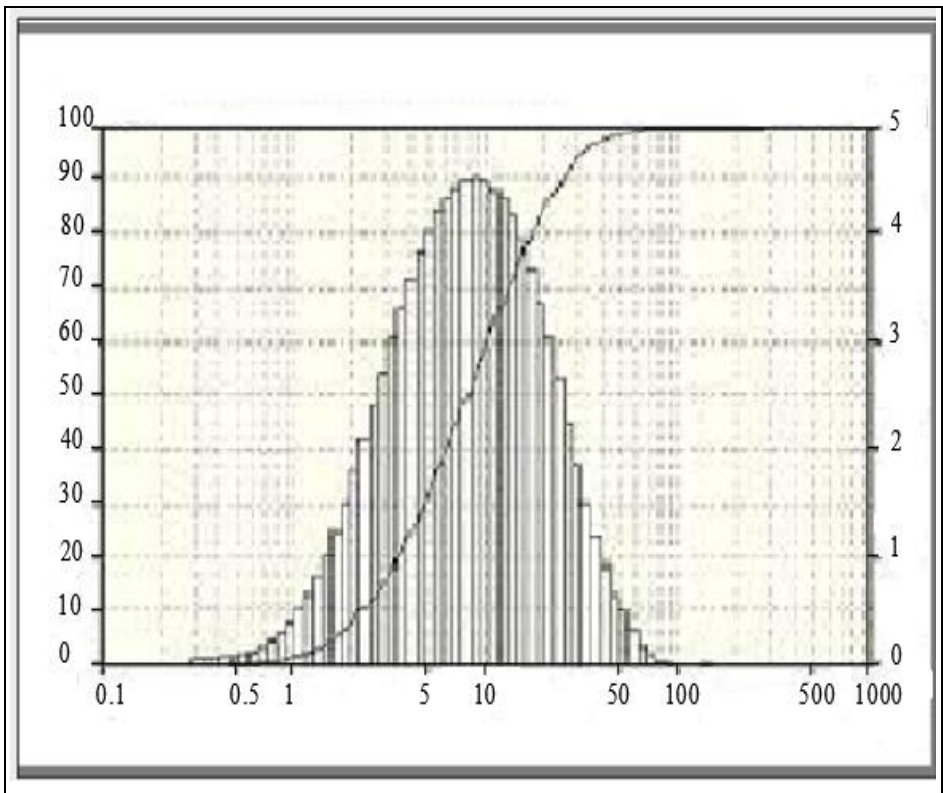
The analyses show that the composition of chamotte and some fly ash is composed of aluminosilicate minerals. The diffractogram of fly ash shows that it is an acid ash, that is, it is obtained from the burning of coal or brown coal. The diffractogram clearly shows that the composition of fly ash is mainly composed of the mineral sillimanite. The composition of sillimanite consists of  $2\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$  and it is formed at temperatures above  $1200^\circ\text{C}$ . This mineral is one of the minerals that give high physical and mechanical properties to ceramic materials.

One of the ultradisperse additives is chamotte. Chamotte is a material obtained by burning kaolin clays at temperatures above  $1000^\circ\text{C}$ . It is widely used in the production of fine ceramics and refractory ceramic materials. Its composition consists of dehydrated aluminosilicate-containing clay minerals.

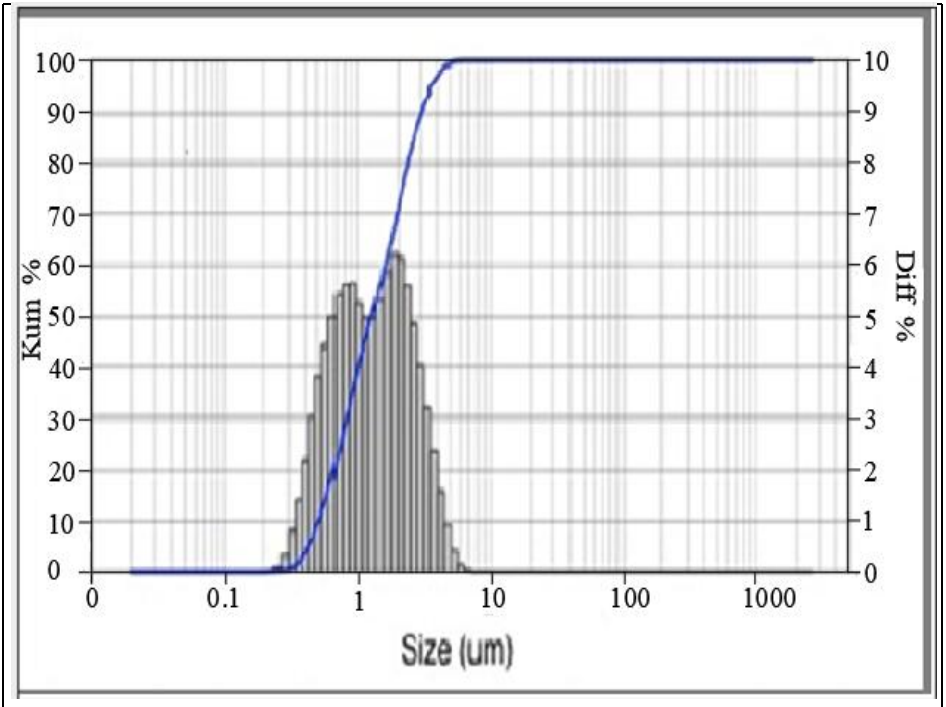
In chapter II of the dissertation, the results of the study of several technological properties of clays were also presented. The results of their granulometric composition are presented in figure 1, and the results obtained from the study of coagulation temperature, drying and binding property are presented in table 2.

**Table 2. Technological characteristics of the clays used in the research work**

No	The name of a clay deposit	Shrinkage of clay, %	Solidification temperature, °C	Binding property, MPa
1	Ashagi Guzdak clay	9,5	1340	4,7
2	Umbakı clay	5,0	1390	4.1
3	Pirigol clay	7,0	1380	4,9
4	Kuku clay	9,0	1250	3,5



**Graphics 1. a) Granulometric composition of chamotte:**



**Graphics 1. b) Granulometric composition of fly ash.**

**Table 3. Granulometric composition of ultrafine additives:  
a) Chamotte, b) Fly ash.**

a)

D, $\mu\text{m}$	%
0.000-0.020	0.00
0.020-0.050	0.00
0.050-0.100	0.60
0.100-0.200	13.00
0.200-0.500	74.00
0.500-1.000	10.00
1.000-2.000	2.00
2.000-5.000	0.40
5.000-10.000	0.00

b)

D, $\mu\text{m}$	%
0.000-0.020	0.00
0.020-0.050	0.00
0.050-0.100	0.00
0.100-0.200	9.00
0.200-0.500	60.00
0.500-1.000	30.00
1.000-2.000	3.00
2.000-5.000	0.05
5.000-10.000	0.00

One of the most important technological properties of the studied clays is the determination of the coefficient of sensitivity in drying. This is more important for clinker brick, which is given special

attention to its decorativeness. The drying sensitivity coefficient of all four types of clay samples was determined experimentally. This indicator is for Lower Guzdak clay - 1.06 (clay moderately sensitive to drying), for Umbaki clay - 0.99 (clay less sensitive to drying), for Pirigol clay - 1.34 (clay moderately sensitive to drying), Kuku clay for – 2, 42 (clay highly sensitive to drying).

Since the clays used in the obtaining of clinker bricks are polymineralclays, attention should be paid to their environmental cleanliness, especially their radioactivity. Due to the presence of some K compounds in this type of clay, their radioactivity was checked, and it was determined that their radioactivity is within the norm (for LowerGuzdak clay - 155 Bq/kg).<sup>2</sup>

Chapter III of the dissertation work is devoted to the selection of ceramic mass composition for clinker bricks based on several clay deposits located in Azerbaijan and the study of the properties of the obtained ceramic material. On the basis of local raw materials and ultradispersed additives containing aluminosilicate, the optimal composition of the raw material mixture and the optimal burning mode for the production of clinker bricks were determined, and the construction and technical properties of the obtained ceramic material were studied.<sup>3</sup> It is very important to determine the optimal combustion mode of the ceramic mass in the study of cladding materials such as clinker bricks, which have special requirements for their density. Because the structure of ceramic material is formed during combustion.

It is known that during the firing of ceramic material, the product should remain at the coagulation temperature for an optimal period. At the first moments of coagulation, the ceramic mass becomes dense, but after a certain period, the product is deformed and loses its shape. At the same time, too much burning can cause defects such as foaming, which is an important defect for clinker brick, which is considered a facing material.

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2 Study of radioactivity of raw materials for clinker brick production. Bagirov B.I., Shirinzade I.N.

3 Study of the effect of fly ash on the properties of clinker brick. Bagirov B.I., Shirinzade I.N.

To achieve the goal set in the research work, various ultradisperse materials were added to the selected clays and the combustion mode of the obtained ceramic masses was determined.

Table 4 presents the results obtained from the firing of the ceramic mass made on the basis of a mixture of Lower Guzdak clay and fireclay.

**Table 4. The effect of the combustion mode of the ceramic mass on its properties**

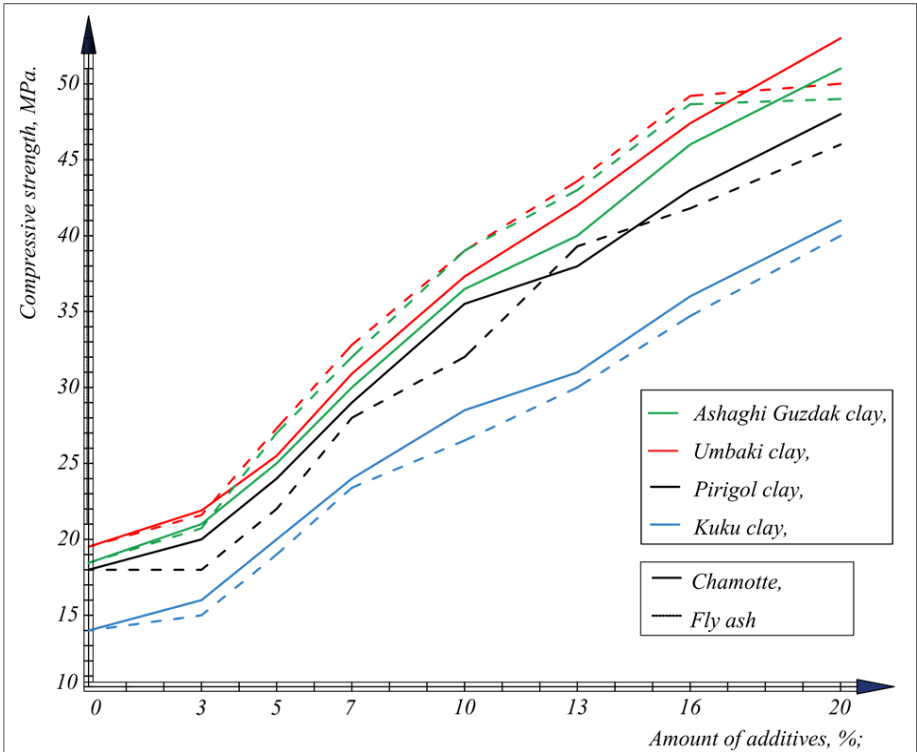
Combustion temperature , °C	Shrinkage during combustion , %	Water absorption , %	Medium density , q/sm <sup>3</sup>
950	3.3	15.5	1.63
1000	3.7	10.8	1.70
1050	4.2	9.7	1.80
1100	4.9	8.6	1.85
1150	5,2	8,0	1,90
1200	5,5	6,5	1,95
1250	5,8	5,5	2,0
1300	6,9	5,0	2,2

As can be seen from the tables (table 2 and table 3), the coagulation temperature of the Lower Guzdak clay without additives was 1340<sup>0</sup>C, and when it was fired with fireclay, it was 1250<sup>0</sup>C.

The coagulation temperature of ceramic materials made on the basis of other clays and aluminosilicate-containing additives used in the research work was determined, which was 1390<sup>0</sup>C and 1300<sup>0</sup>C for Umbakı clay, 1380<sup>0</sup>C and 1280<sup>0</sup>C for Pirigol clay, and 1250<sup>0</sup>C and 1190<sup>0</sup>C for Kuku clay.

As it can be seen from Table 3, with the increase in temperature, the absorption of the samples decreases. Since the decrease in water absorption causes changes in other properties of stone materials (for example, increase in strength and frost resistance, decrease in friction, increase in density), it can be considered based on this property that

1250<sup>0</sup>C is the optimal temperature for making clinker bricks based on Lower Guzduk clay.



**Graphics 2. Effect of aluminosilicate-containing ultradisperse additives on the strength of ceramic material.**

For clinker bricks, the moisture content of the material (according to the standard) should be below 6%. This indicator is also provided in samples fired at 1250<sup>0</sup>C.

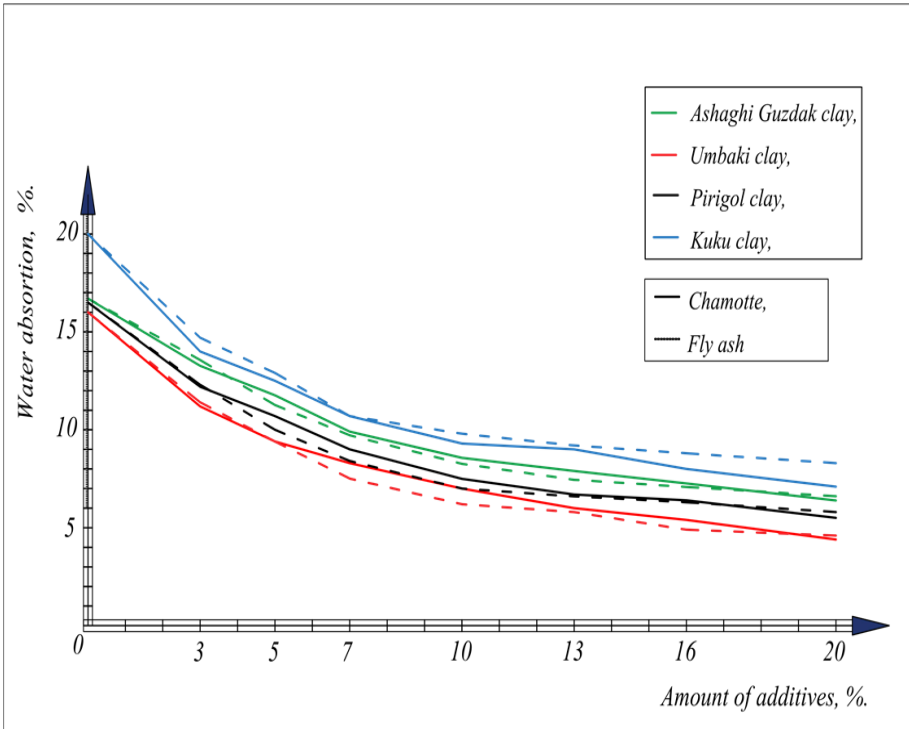
Determination of the optimal amount of ultradisperse additives was determined experimentally for each of the 4 selected types of clay, and the results of experiments are presented in figure 2 and figure 3.<sup>4</sup> The compressive strength of the ceramic materials obtained by adding 10% aluminosilicate additive (fireclay and fly ash) to the clays of the

<sup>4</sup> Selection of Ceramic Mass for Clinker Brick by Modification of Azerbaijan Clays with Ultradisperse Additives. Baghirov B.I., Mammadova I.H., Shirinzade I.N.



Lower Guzdak, Umbakı and Pirigol fields from the studied clays meets the norms set for the strength of clinker bricks. This is ensured by adding 13% to the material obtained on the basis of the clay of the Kuku bed.

Also, materials with higher strength properties were obtained on the basis of Umbaki and Ashagi Guzdak clays. The limit of compressive strength of materials with ultradispersed additives containing aluminosilicate was 2.5-2.9 times higher than the material without additives.



**Graphics 3. Effect of aluminosilicate-containing ultradisperse additives on the drying of ceramic material.**

As mentioned above, the compressive strength of the ceramic material obtained by using 10% ultradisperse additive meets the standards for clinker bricks, but this amount was not considered as optimal. This is achieved by adding 13% to the material obtained from the clay of the deposit.

Also, materials with higher strength properties were obtained based on Umbaki and Aşağı Güzdek clays. The compressive strength of materials with ultradisperse additives containing aluminosilicate significantly differs from the material without additives. Thus, a 20% chamotte additive increased the strength of the material based on Aşağı Güzdek clay by 2.8 times, the strength of the material based on Umbaki clay by 2.7 times, the strength of the material based on Pirigöl clay by 2.5 times, and the strength of the material based on Küku clay by 2.9 times.

Also, fly ash addition increased the strength of materials based on the mentioned clays by 2.6; 2.5; 2.4 and 2.8 times, respectively. As can be seen from the results of the experiments, there is almost no difference in the use of fireclay, which is a relatively high energy-intensive material, and fly ash, which is an industrial waste.

This is due to the fact that the requirements for drying, which is another important property of ceramic bricks, are not met when using an additional 10%.

The results of the experiment show that the required index of water absorption is provided when using 15-16% dispersing additive.

Most often, the frost resistance of clinker bricks is determined based on the saturation coefficient. Although the frost resistance grade of clinker bricks is not precisely determined by this method, it is possible to determine whether the material is frost resistant or not. The saturation coefficient is usually determined by the ratio of the dry shrinkage of material samples by mass to the dry shrinkage of the samples after boiling.

If the saturation coefficient of the material is higher than 0.8, then the material will be frost resistant.

As can be seen from Table 3.1.5, the saturation coefficients of the ceramic material obtained by modifying all types of clays studied with aluminosilicate-containing ultradisperse additives were determined for samples with the optimal composition determined in the previous paragraph. In this case, samples were prepared using 84% of all 4 types of clay (separately), 16% of aluminosilicate-containing ultradisperse additives, 82% clay and 18% (9+9) of each additive. The dry weight of the materials under normal conditions and after boiling

was determined, and the saturation coefficient was calculated based on these indicators.

The saturation coefficient of all samples was above 0.8. The samples with the best saturation coefficient were clay and ultradisperse additives from the Umbaki deposit, while the samples with a relatively low saturation coefficient were samples prepared on the basis of clay from the Kuku deposit.

**Table 5. Water Saturation coefficient of ceramic material for clinker bricks**

№	Composition of materials, %			Water absorption by mass of materials, $W_k$ , %	Water absorption of materials after boiling, $W_q$ , %	Saturation coefficient of materials
	Clay	Chamotte	Fly ash			
Based on Lower Guzdek clay						
1	84	16	-	6,0	6,8	0,88
2	84	-	16	5,8	6,5	0,89
3	82	9	9	5,7	6,4	0,89
Based on Umbaki clay						
4	84	16	-	6,0	6,8	0,88
5	84	-	16	5,5	6,1	0,90
6	82	9	9	5,6	6,1	0,91
Based on Pirigol clay						
7	84	16	-	6,4	7,3	0,87
8	84	-	16	6,3	7,4	0,85
9	82	9	9	6,1	7,0	0,87
Based on Kuku clay						
10	84	16	-	6,0	6,9	0,87
11	84	-	16	6,8	8,2	0,82
12	82	9	9	6,5	7,4	0,87

In chapter III of the research work, the influence of the molding method and the optimal burning temperature on the properties of the material was also studied. For this, 2 optimal compositions were selected: composition I (clay - 84%, fireclay - 16%), composition II (clay - 84%, fly ash - 16%).

Samples of the selected composition were prepared by both plastic molding (moisture 23%) and semi-dry pressing (moisture 12%). As a result of the experiment, it was determined that the strength of the ceramic mass molded by the semi-dry pressing method was 40% higher than the strength of the material molded by the plastic method. Also, other technical indicators (density, water absorption, water resistance) of the material obtained by molding by semi-dry pressing method were significantly higher.<sup>5</sup>

At a moisture content of 23%, the preparation of samples is technically not difficult. However, at lower moisture contents, the molding of the material becomes more difficult. To prevent deformation during firing, the moisture content of the material should be reduced to 3-5% after drying. Reducing moisture improves the quality of the ceramic material. Therefore, the lower the moisture content of the clay mass, the higher the quality of the finished product.

Research has been conducted to eliminate this contradiction in the process of preparing ceramic mass. The conducted research shows that, analogously, surface-active additives (SAA) used to reduce the water requirement of concrete mix are also effective in eliminating this contradiction in the production technology of ceramic materials.

At the same time, it is considered advisable to use chemical additives to prevent the formation of white salts on the surface of ceramic facing material. Since such defects in materials such as clinker bricks (especially clinker bricks intended for walls) impair the appearance of the product, chemical additives were used.

Clays from the Umbaki (Gobustan region) and Ashagi Guzdek (Absheron region) deposits were used in the research work. C-3 (naphthalene sulfate acid and sodium salt of formaldehyde) was used

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as a surface-active additive in the amount of 0.3% of the ceramic mass.

The amount of ultradisperse additives used in the preparation of these samples was 20%. As a result of the experiment, it was determined that the use of surfactants reduces the moisture content of the ceramic mass from 25% to 20%. Reducing the moisture content gives more effective results when using clay from the Lower Guzdek deposit, which has a relatively high plasticity. Increasing the amount of surfactants in the preparation of ceramic batches was not considered advisable. The use of small amounts of surfactants (especially chemical additives containing alkali cations) improves the molding of the ceramic batch, but an increase in the amount of surfactants leads to agglomeration of clay particles, which in turn worsens the compaction of the mass.

As a result of the experiment, it was determined that the compressive strength of the ceramic material based on Umbaki clay with chamotte additives increased from 49.6 MPa to 54.5 MPa when using a chemical additive. This was from 48.6 MPa to 52.3 MPa in the ceramic material prepared on the basis of Lower Guzdek clay, respectively.

In the ceramic samples in which surface-active additives were used, the number of cracks after firing was significantly reduced, the uniformity of the fired ceramic material increased, and as a result, the strength of the material significantly increased. Also, low humidity leads to a decrease in the fuel consumed in production, which reduces the cost of the product in an era when energy carriers are rapidly increasing in price.

One of the important technological properties of ceramic materials is their shrinkage during drying and firing. This indicator is especially important in materials where decorativeness is important, such as clinker bricks.

It has been experimentally established that ultradisperse additives containing aluminosilicate significantly reduce the shrinkage of ceramic materials both during drying and during firing. The air-drying shrinkage of ceramic material made from the Lower Güzdek clay decreased from 8% to 3.5% with the use of additives, and the shrinkage during combustion decreased from 10% to 2.3%. The air-

drying shrinkage of ceramic material made from the Umbakı clay decreased from 6% to 2% with the use of additives, and the shrinkage during combustion decreased from 6% to 4%. The air-drying shrinkage of ceramic material made from the Pirigöl deposit clay decreased from 6% to 4% with the use of additives, and the shrinkage during combustion decreased from 8.5% to 4.5%. The air-drying shrinkage of ceramic material made from the Kükü deposit clay decreased from 8% to 5.6% with the use of additives, and the shrinkage during combustion decreased from 12% to 7.2%.

The study of the influence of the combustion mode, which is one of the most important factors affecting the formation of the technical properties of ceramic materials, is presented in this section.

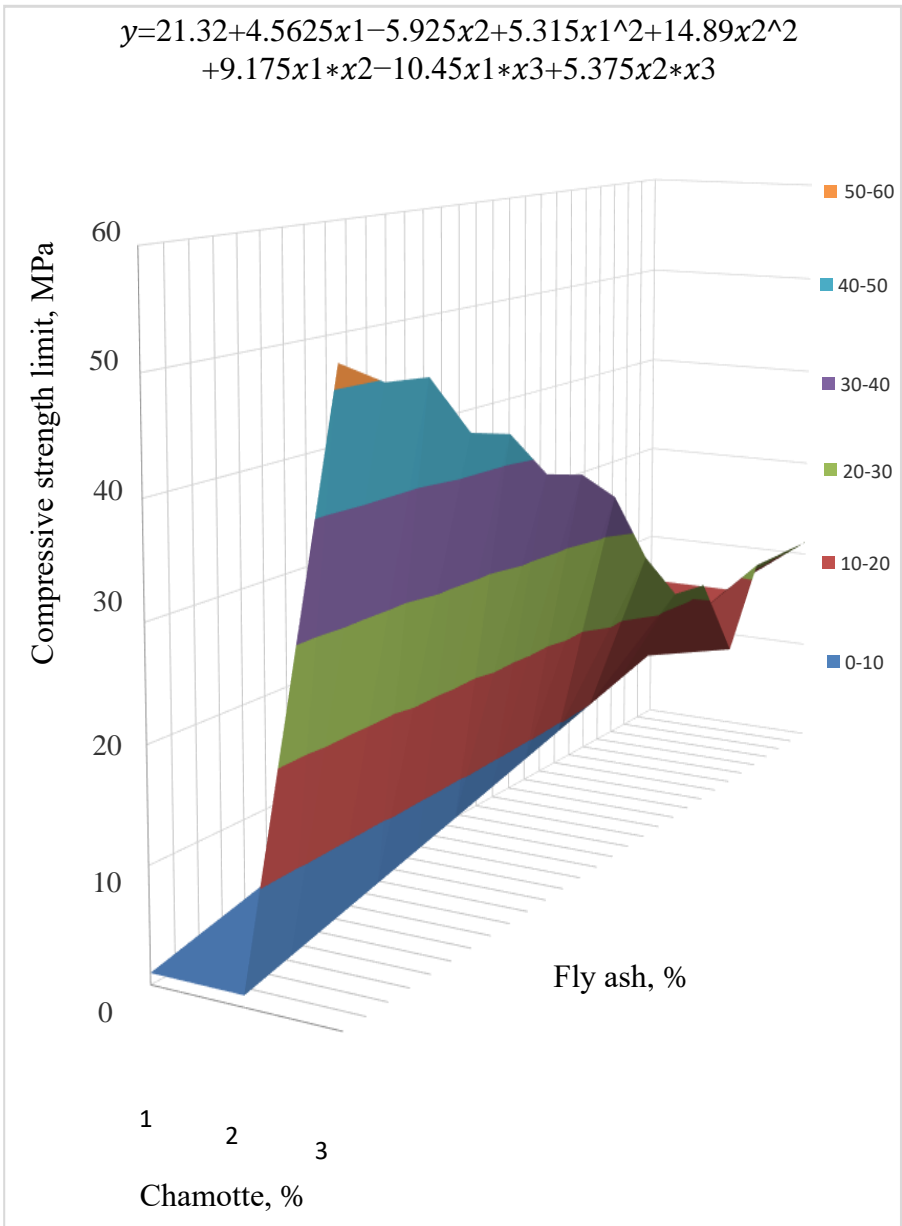
The selection of the optimal combustion mode in ceramic materials with ultradisperse additives was determined based on the drying of the burned material. The firing temperatures corresponding to 6% lower drying of ceramic materials were as follows for the studied clays:

1250<sup>0</sup>C was determined based on the clay of Lower Guzdak, Umbakı and Pirigol clays, and 1100<sup>0</sup>C was determined in the material obtained based on Kuku clay.

2-component ceramic mass compositions are usually used in the ceramic materials industry of our country. More modern systematic approaches should be used in the study of 3-component ceramic mass compositions.

Conducting such a systematic study of clinker brick is an important issue for more efficient organization of its production.

Mathematical-statistical analysis was carried out by applying the Box-Benkin plan in studying the separate characteristics of each of the components that make up the composition of the 3-component ceramic mass and the characteristics formed due to the interaction of these components.



**Graphics 4. Dependence of the compressive strength limit on the amount of ultradisperse additives**

It was determined that the optimal composition of the clay is determined according to the coefficients of the regression equations obtained as a result of the active experiment, and this optimal composition provides the maximum value of the compressive strength limit of 58.66 MPa.<sup>6</sup>

The last paragraph of Chapter III of the research study is devoted to the study of the composition of clinker bricks made on the basis of ultradispersed ceramic mass using modern physico-chemical research methods.

Since the properties of ceramic materials depend on the ratio of the amorphous and crystalline phases that make up its composition, the phase composition of the material was determined based on the chemical composition and is presented in table 6.

This was carried out on samples prepared from Lower Bag clay and ultradisperse additives containing selected aluminosilicates and fired at 1250<sup>0</sup>C.<sup>7</sup>

**Table 6. Mineralogical composition of ceramic material for clinker brick**

Components included in the composition of the ceramic mass, %			Mineralogical composition of ceramic material, %		
Clay	Chamotte	Fly ash	Mullite	SiO <sub>2</sub>	Glass phase
80	10	10	34,67	52,0	12,7

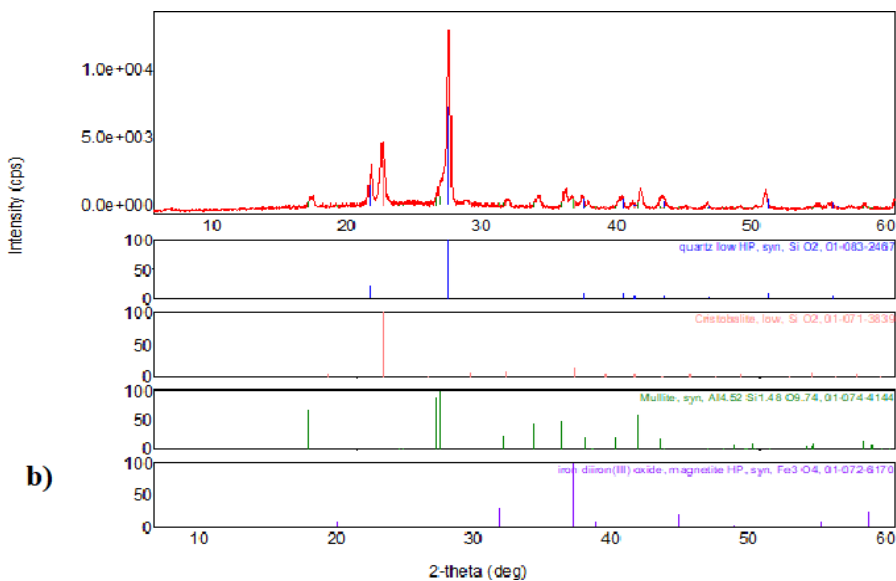
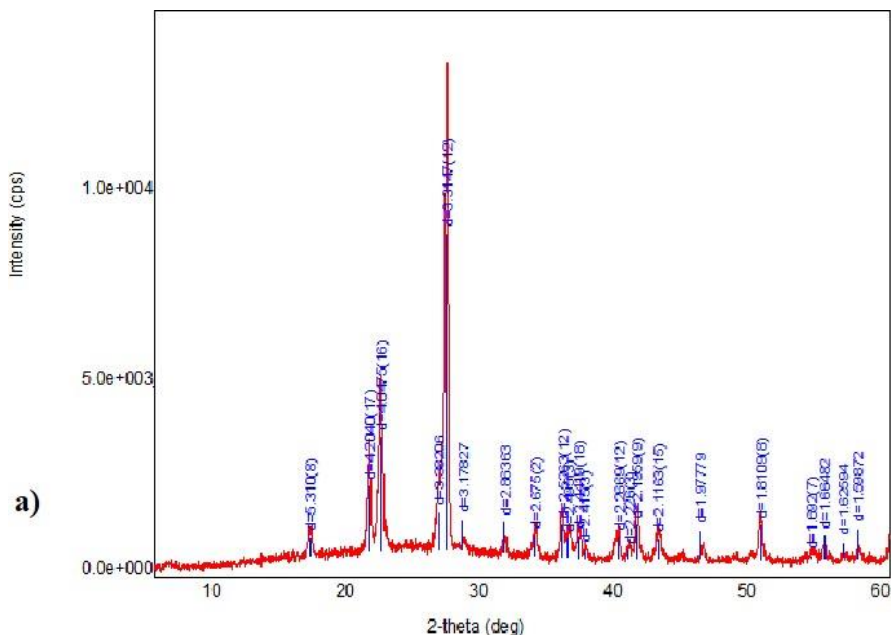
It can be seen from the table that the composition of the studied material mainly consists of crystalline phase (87.3%). The crystalline phase is composed of mullite mineral and free SiO<sub>2</sub>. A small amount of glass phase (12.7%) leads to further densification of the ceramic material.

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<sup>6</sup> Проведение активного эксперимента при разработке состава шихты для производства клинкерного кирпича на основе местных глин. Багиров Б.И., Мамедова И.Г., Аббасова Н., Ширинзаде И.Н

<sup>7</sup> Study of chemical mineralogical composition of clinker brick. Bagirov B.I., Shirinzade I.N., Mammadova I.H., Khalilov E.V.





**Figure 1. Ashaghi Guzdek diffractogram of ceramic material based on clay and chamotte.**

This is evident from their dervatograms presented in Figure 1 and their micrographs (Figure 2)

Chapter IV of the thesis work is devoted to the development and feasibility study of clinker brick technology based on ceramic mass modified with ultradisperse additives. Here, first of all, the technological operations for the production of clinker bricks are mentioned, the raw materials are selected, and the raw material mixture is prepared.

Lower Guzdak clay, which is also used in laboratory tests, was used because it is close to the enterprise where experimental testing is carried out (Ceramics and pottery factory. Khirdalan city). Both chamotte and fly ash were used as ultradisperse additives.

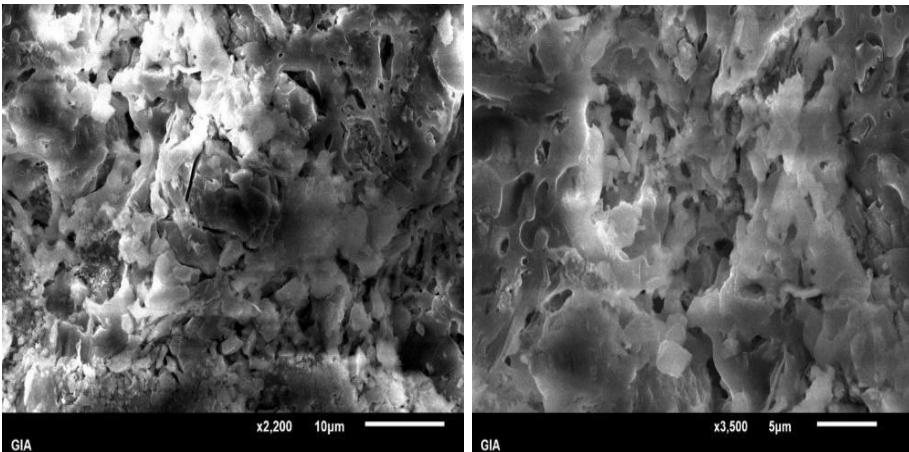
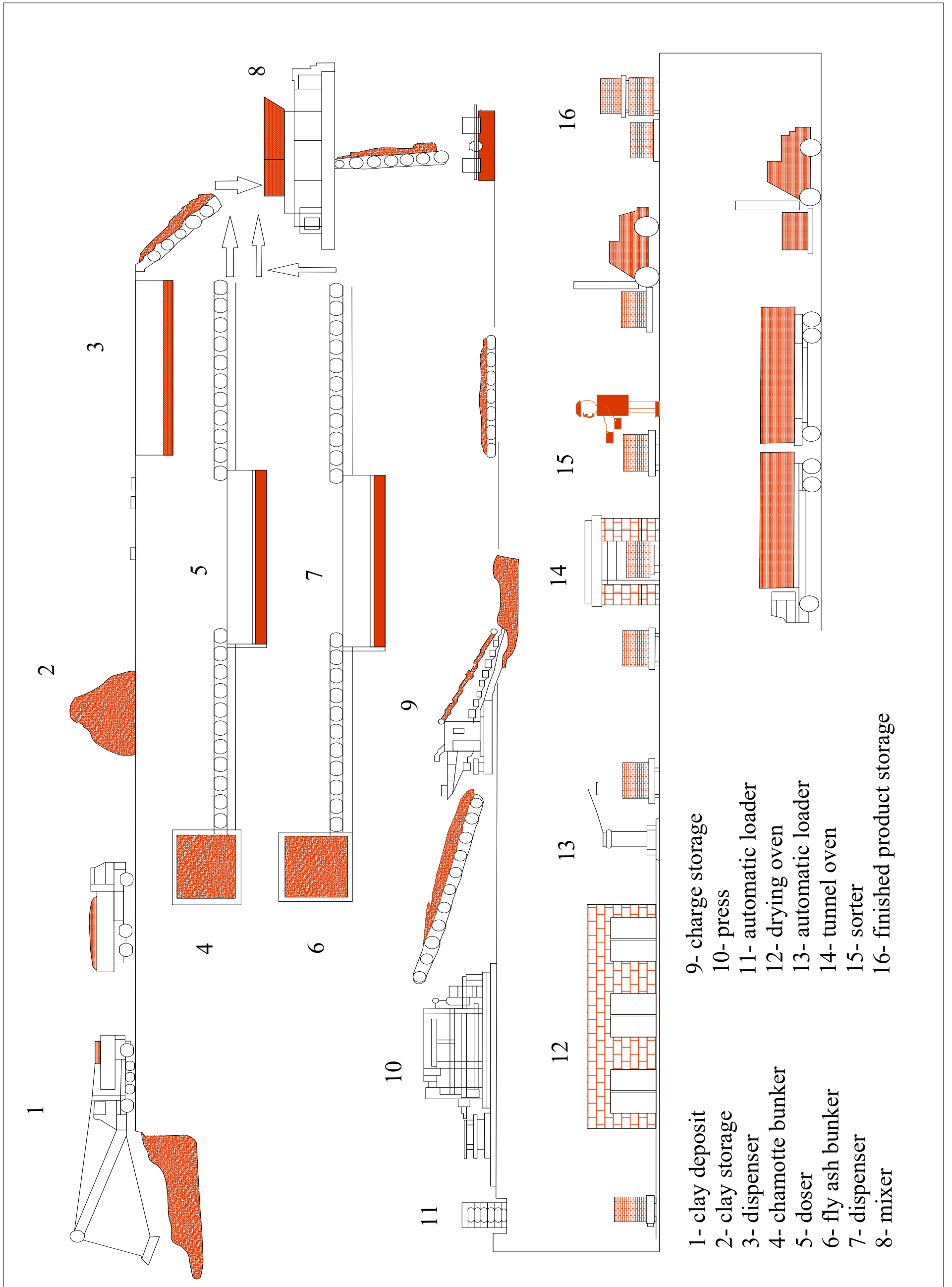


Figure 2. Ashaghi Guzdak Micrographs of ceramic material containing clay, chamotte and fly ash.

Technological operations for the production of clinker brick are defined as follows:

- Grinding clay - 2 hours;
- Dosing of raw materials – 2 minutes;
- Dry mixing of the ceramic mass - 5 minutes;
- Mixing the ceramic mass with water - 5 minutes;
- Clinker brick pressing – 1 min;
- Drying of bricks at 80-90<sup>0</sup>C - 7 hours;
- Burning bricks at 1250<sup>0</sup>C – 2 hours.



**Figure 3. Technological scheme for the production of clinker bricks modified with local polymineral clays and ultra-dispersed additives containing aluminosilicate.**

Construction-technician properties of clinker bricks obtained under production conditions, were determined according to GOST 32311-2012 and it was determined that it meets the requirements of the standard.

The results of technical and economic indicators of clinker brick production are also presented in chapter IV of the dissertation. First, the cost of 1 brick was calculated, and then the economic efficiency was calculated by comparing it with the price of clinker bricks brought to our country from Russia and Iran. Annual economic efficiency is equal to 1 million manats.

## MAIN RESULTS

1. The features of the clinker brick manufacturing technology were analyzed, and it was determined that it is possible to obtain a facing product with high technical properties by modifying local polymineral clays with aluminosilicate-containing ultradisperse additives.
2. The composition of clays and aluminosilicate-containing ultradisperse additives used in the production of clinker bricks was studied through modern physico-chemical research methods and it was determined that the clays of the Umbaki and Pirigol deposits belong to the semi-acidic clay group, and the clays of the Lower Guzdak and Kuku deposits belong to the sour clay group. Also, the clays of the Umbakı and Pirigöl deposits are hard-to-melt clays due to their refractoriness. According to the X-ray analysis of the composition of ultradisperse additives containing aluminosilicate, it was determined that the composition of fireclay, fly ash and dehydrated clay consists of amorphous mass ( $\text{SiO}_2$ ), which ensures their high activity in the thermal process.
3. The technological properties of the clays used in the production of clinker bricks were also studied and experimentally determined that the amount of clay particles is less in the clay of the Kuku deposit than in other deposits (46% in the Kuku deposit, 50-60% in others). Also, the coagulation temperature of the clays was determined: it was  $1340^\circ\text{C}$  for Ashagi Guzdak clay,  $1390^\circ\text{C}$  for Umbaki clay,  $1380^\circ\text{C}$  for Pirigol clay, and  $1250^\circ\text{C}$  for Kuku clay.
4. Based on 4 types of clay used in the research (clays of Agashi Guzdak, Umbakı, Pirigol and Kuku deposits) and ultradisperse additives containing aluminosilicate, the effect of the amount of additive on the properties of ceramic material was studied. It was determined that when 15-20% ultradisperse additive is added to the ceramic slag prepared on the basis of the clays of Agashi Guzdak, Umbaki and Pirigöl deposits, the physical and mechanical indicators of the obtained material meet the norms given by the standard for clinker brick. That is, the absorption of these materials is less than 6%, and their compressive strength is more than 30 MPa. The drying rate of the ceramic material obtained on the basis

of the clay of the Kuku bed and up to 20% addition is higher than this norm.

5. As a result of the experiment, it was proved that the technical properties of the ceramic material obtained on the basis of fireclay and fly ash do not differ sharply from each other. The compressive strength of the ceramic material obtained using 20% fireclay is 5-6% higher than the compressive strength of the ceramic material obtained using the same amount of fly ash.
6. The dependence of the properties of the clinker brick on the molding method of the ceramic mass was studied, and it was determined that the compressive strength limit of the ceramic material molded with a pressure of 15 MPa at 12% moisture content (semi-dry pressing method) is 30% higher than the compressive strength limit of the material molded by the plastic method (at 23% moisture content).
7. To increase the effectiveness of plastic molding, surface- active additives were used in the preparation of the selected optimum ceramic slag composition. With the use of 0.3% C-3 plasticizer, the moisture content of the ceramic mass was reduced from 25% to 20%, which led to an increase in the density and, accordingly, strength of the ceramic material (10-15%), and a significant decrease in shrinkage.
8. Based on local polymineral clays and ultradisperse additives containing aluminosilicate, the firing of ceramic mass at 1250<sup>0</sup>C was experimentally determined to obtain clinker brick with high technical properties.
9. Mathematical-statistical analysis was carried out by applying the Box-Benkin plan in the study of the separate properties of each of the components that make up the composition of the three-component ceramic mass and the properties formed due to the interaction of these components. It was determined that the optimal composition of the clay was determined according to the coefficients of the regression equations obtained due to the active experiment, and this optimal composition provides the maximum value of the compressive strength limit of 58.66 MPa.
10. The composition and structure of the ceramic material for the purchased clinker brick was studied using modern physico-

chemical analysis methods and it was determined that the composition of the high-quality ceramic material consisted of 25-30% crystalline phase and 70-75% glass phase. The crystalline phase consists mainly of the mineral mullite and  $\text{SiO}_2$  (crystalite).

11. Production of clinker bricks in industrial conditions was carried out at the "Ceramics and Pottery" factory located in the city of Khyrdalan. The physical and mechanical properties of the obtained clinker bricks were in accordance with the requirements of GOST 32311-2012. The economic indicators of the clinker brick obtained under production conditions were calculated and it was determined that compared to the clinker brick imported from foreign countries, the product obtained with the proposed technology is 2 times cheaper.

**List of published scientific works**  
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**The applicant's personal contribution to published works:**

Scientific works No. [1; 5; 10; 11] were independently carried out by the Claimant

In scientific works [2; 3; 4; 6; 7; 8; 9; 12; 13; 14; 15; 16], the formulation of the problem, the conduct of experiments, the integration of the results and the preparation of the article for publication belong to the Claimant.

The defense of the dissertation will be held at the meeting of the One-time Dissertation Council BFD 2.37, which was established on the basis of the FD 2.37 Dissertation Council operating under the Azerbaijan University of Architecture and Construction, on the date *January 24, 2025 at 12:00.*

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It is possible to get acquainted with the dissertation work in the library of the Azerbaijan University of Architecture and Construction.

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