**Eco-Efficient Sulfate-Resistant Cement Using Karakalpakstan Raw Materials**

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**Abstract.** This study is devoted to the production of sulfate-resistant Portland cement clinker based on local raw materials found in the Aral Sea region of the Republic of Uzbekistan and the study of its properties. The results obtained highlight technological, economic and environmental advantages. The raw material mixtures consisted of limestone, barren sand, kaolin and iron-rich industrial waste, resulting in a clinker with an optimized oxide composition (LSF = 0.82, SR = 2.15, AR = 0.94). X-ray phase analysis shows a high content of alite (C₃S ≈ 38.6%) and belite (C₂S ≈ 41.0%) phases, which provides high mechanical strength. The low content of tricalcium aluminate (tricalcium aluminate < 5%) and the sufficient content of ferrite (C₄AF ≈ 16.2%) increased sulfate resistance and phase dominance. Sulfate resistance tests performed in accordance with ASTM C1012/C1012M showed that sulfate-resistant Portland cement had the lowest swelling index among samples placed in a 5% Na₂SO₄ solution, proving its high resistance compared to ordinary Portland cement and mixed cement types. The results obtained provide an opportunity to produce high-quality sulfate-resistant Portland cement clinker based on local raw materials and demonstrate its suitability for the sulfate-rich conditions of the Oral region. Economically, the use of local raw materials reduces production costs, reduces the need for imports, and increases the competitiveness of the cement industry. From an environmental perspective, the long service life of sulfate-resistant Portland cement and the use of industrial waste are compatible with the green economy, help reduce waste, and contribute to sustainable development.

**Keywords:** sulfate-resistant Portland cement, clinker synthesis, mineral resources, sulfate resistance, green economy, sustainability.

**INTRODUCTION**

In recent years, the rapid development of the construction industry — in particular, hydraulic structures, industrial facilities and housing construction — has sharply increased the demand for durable and long-lasting cements. In the Republic of Karakalpakstan, especially in the Aral Sea region, high concentrations of sulfate ions (SO42-) in soil and groundwater pose a serious threat to concrete structures. Ordinary Portland cement is rapidly corroded in a sulfate environment, since sulfates interacting with the three calcium aluminate phases in the composition form expanding ettringite, resulting in cracking and a decrease in strength. Therefore, the development of sulfate-resistant Portland cement is an urgent task for this region.

According to the standards of UzM St 337:2024 and GOST 22266-2013, the content of tricalcium aluminate (tricalcium aluminate) in the cement composition should not exceed 5%, and in some cases it should be reduced to 3%. This is because tricalcium aluminate reacts with sulfate ions and forms harmful expansion products. Therefore, reducing the content of tricalcium aluminate by reducing the content of Al₂O₃ or increasing Fe₂O₃ increases sulfate resistance.

The territory of Karakalpakstan has large reserves of high-quality limestone (CaCO₃ ≈ 85–90%), kaolin (Al₂O₃ ≈ 36–38%), basalt and iron-rich rocks. These raw materials allow for the production of low-cost, high-quality sulfate-resistant Portland cement clinker and are important for the complex sulfate-rich conditions of the Aral Sea region. The rational use of local resources not only provides a technological advantage, but also increases economic efficiency.

The main objective of this study is to obtain sulfate-resistant Portland cement clinker based on mineral raw materials from the Republic of Karakalpakstan, determine its chemical and mineralogical composition, and evaluate the effect of phase structure on sulfate resistance. Ordinary Portland cement is subject to sulfate attack, reducing the service life of concrete structures. Therefore, many researchers are focusing on reducing the amount of tricalcium aluminate to improve sulfate resistance. Recent studies have shown that reducing the amount of Al₂O₃ or increasing the amount of Fe₂O₃ is an effective way to reduce the amount of tricalcium aluminate phase [1-5].

Scientific studies conducted by Uzbek scientists also show that local clay mineral deposits have great potential. In particular, the possibility of using kaolin from the Khojakul deposit located in Karakalpakstan as a high-quality aluminosilicate additive for the cement industry has been scientifically proven. Metakaolin obtained from Aral Sea raw materials has been found to increase the sulfate resistance and heat stability of cement composites [6, 7]. It has also studied the mineralogy and processing technology of Khojakul kaolins, confirming their industrial significance [8]. According to the information provided in the literature, it has been studied the possibilities of producing glass and ceramic products based on kaolin and feldspar, emphasizing their role in creating stable silicate materials [9].

Research is also being conducted in the region on silicate materials. High-strength ceramic materials for the restoration of historical monuments have also been obtained based on loess-lime. The production of binders based on mechanically activated clay has been proposed as an effective alternative in construction [10].

The synthesis of sulfate-resistant Portland cement clinker based on local raw materials of Karakalpakstan is technologically and economically important. From an economic point of view, the use of limestone, barren sand, kaolin, and iron-rich industrial waste reduces the need for imports, reduces costs, and increases the competitiveness of the regional cement industry. From an ecological point of view, the high durability of sulfate-resistant Portland cement clinker extends the service life of buildings, reduces costs, and reduces environmental impact. As a result, it is fully consistent with the principles of the modern "green economy" and "circular economy".

In addition, the addition of iron-rich industrial waste to the raw material composition serves to recycle industrial waste and contributes to the reduction of carbon dioxide emissions into the atmosphere in the cement industry. As a result, the production of sulfate-resistant Portland cement clinker not only helps to achieve technological superiority, but also economic efficiency and environmental sustainability, and makes it possible to produce it as a promising material on a national scale.

**METHODS**

The production of sulfate-resistant Portland cement clinker was carried out using local raw materials from the Republic of Karakalpakstan. The main composition of the raw material mixture consisted of limestone, barren sand and kaolin, with basalt and iron-rich industrial waste used as additives.

Laboratory-scale samples were prepared at Karakalpaksement LLC, and the analysis was carried out at the STROM Research and Testing Center under the Institute of General and Inorganic Chemistry of the Academy of Sciences of Uzbekistan.

**Chemical Composition.** The chemical composition, determined according to UzM St 337:2024 and GOST 5382-2019 using classical titration and gravimetric methods, was as follows (wt.%): CaO – 61.09, SiO₂ – 22.10, Al₂O₃ – 4.98, Fe₂O₃ – 5.32, MgO – 2.07, SO₃ – 0.80, LOI – 1.14, Free CaO – 0.94.

Calculated module values were: LSF = 0.82, SR = 2.15, AR = 0.94 — all within SULFATE-RESISTANT PORTLAND CEMENT normative limits.

**Mineralogical composition (XRD).** X-ray phase analysis (Rigaku MiniFlex, Cu-Kα radiation) and Rietveld recalculation revealed the main phases in the cement clinker: C₃S – 38.6%, C₂S – 38.0%, tricalcium aluminate – 4.2%, C₄AF – 14.8%. These results are consistent with Bogue calculations and the solid phase composition of sulfate-resistant Portland cement clinker.

**Test conditions.** All analyses were performed in accordance with the requirements of UzM St 337:2024 and GOST 5382-2019 [11, 12]. Sulfate resistance was assessed according to ASTM C1012/C1012M: samples were immersed in a 5% Na₂SO₄ solution for testing and their expansion was compared with reference samples stored in distilled water [13].

To ensure reliability, two control samples were used in each case, which allowed to accurately determine the effect of sulfate ions on dimensional stability and strength.

**RESULTS AND DISCUSSION**

The selection of raw materials for the production of sulfate-resistant Portland cement clinker is of particular scientific and practical importance. Therefore, the economical use of local raw materials is an urgent issue. In the Republic of Karakalpakstan, dune sands, kaolin, iron-rich industrial waste and limestone deposits are considered promising sources of raw materials. By analyzing their chemical composition, it is possible to determine the mineralogical composition of sulfate-resistant Portland cement clinker.

The ratio of the main oxides in the raw material mixture is an important factor in the synthesis of sulfate-resistant Portland cement clinker. Calcium oxide (CaO) serves to form alite (C₃S) and belite (C₂S) phases, and silicon dioxide (SiO₂) is the basis for silicate phases; aluminum oxide (Al₂O₃) serves to form tricalcium aluminate (tricalcium aluminate). Also, to ensure sulfate resistance, it is necessary to reduce the content of tricalcium aluminate to ≤5%, preferably to 3%. Iron oxide (Fe₂O₃) is part of the ferrite phase (C₄AF) and is important in reducing the content of tricalcium aluminate.

Also, achieving the correct distribution of oxides is an important factor in ensuring the strength and long-term stability of cement in sulfate environments. The chemical composition of local raw materials in the Republic of Karakalpakstan by main oxides is presented in Table 1.

**TABLE 1.** Chemical composition of local raw materials selected for the synthesis of sulfate-resistant portland cement

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Raw Material** | **Chemical composition by major oxides (wt.%)** | | | | | | | | | |
| **SiO₂** | **Al₂O₃** | **CaO** | **MgO** | **Fe₂O₃** | **Na₂O** | **K₂O** | **TiO₂** | **SO₂** | **LOI** |
| **Limestone** | – | 0.15 | 53.14 | 2.56 | 0.12 | – | – | – | – | 42.49 |
| **Barxan sand** | 89.22 | 3.15 | 0.68 | 2.32 | 0.97 | 0.94 | 0.70 | 0.24 | 0.20 | 1.61 |
| **Kaolin** | 54.07 | 16.47 | 0.85 | 2.57 | 9.93 | 1.72 | – | 0.17 | – | 9.23 |
| **Iron-rich by-product** | 40.40 | 6.00 | 2.05 | 4.00 | 45.24 | – | 0.40 | – | 0.22 | 3.10 |

The chemical composition of these raw materials is suitable for ensuring the optimal balance required in the synthesis of sulfate-resistant Portland cement clinker. Barkhan sands are characterized by a very high SiO₂ content (89.22%), which provides a primary source for silicate phases (e.g., C₃S and C₂S) and contributes to improved mechanical strength of clinker. However, due to their relatively low Al₂O₃ (3.15%) and Fe₂O₃ (0.97%) contents, these sands are mainly useful for increasing the silica modulus (SM).

The iron-rich by-product contains a high Fe₂O₃ content (45.24%), which plays a critical role in forming the ferrite phase (C₄AF) and thus contributes to sulfate resistance by reducing three calcium aluminate content. Meanwhile, its SiO₂ (40.40%) and Al₂O₃ (6.00%) levels are moderate, allowing adjustment of the iron modulus (IM) in the raw mix. Low levels of alkaline oxides such as Na₂O and K₂O ensure that adverse effects occur during clinker hydration. Kaolin is high in Al₂O₃ (16.47%) and SiO₂ (54.07%), and acts as the main raw material for the formation of aluminate phases. However, due to the relatively high Al₂O₃ content, kaolin consumption must be carefully controlled, otherwise the tricalcium aluminate content in the clinker may exceed 5%. Kaolin has moderate Fe₂O₃ (9.93%) and Na₂O (1.72%) levels, and it is suitable for adjusting the aluminum modulus (AM). The low content of SO₂ (0%) and TiO₂ (0.17%) does not adversely affect the quality of sulfate-resistant clinker.

Limestone is the main source of CaO (53.14%), which is essential for the formation of alite and belite phases. The almost complete absence of SiO₂ and Al₂O₃ (0% and 0.15%) in it proves that it is a pure calcium source. The low content of MgO (2.56%) and Fe₂O₃ (0.12%) does not cause problems during the clinker hydration process. The loss on heat (LOI, ≈ 42.49%) proves the carbonate nature of the limestone and indicates the release of CO₂ during the calcination process.

In general, the mixture of these raw materials serves to provide the necessary ratio of oxides for sulfate-resistant Portland cement clinker. It is recommended to conduct laboratory tests to determine the main composition. Barkhan sand 15–20%, iron-rich industrial waste 5–10%, kaolin 10–15%, limestone 60–70%. This approach requires the maximum use of local raw materials and allows to increase the resistance of cement in sulfate environments by 20–30%.

The successful balance of the above raw material components is reflected in the mineralogical composition of the synthesized sulfate-resistant Portland cement clinker, since the ratio of oxides directly affects the phases formed (see Table 2).

According to the data from the table, the clinker contains CaO (61.09%). It is mainly derived from limestone and is important in the formation of the main part of the alite (C₃S) and belite (C₂S) phases, which determine the strength of the cement and the hydration process. The SiO₂ content (22.1%) is moderate, which plays an important role in the formation of silicate phases in particular. This oxide is mainly derived from barren sands and kaolin. The amounts of Al₂O₃ (4.98%) and Fe₂O₃ (5.32%) serve to form the aluminate and ferrite phases in the clinker due to kaolin and iron-rich industrial waste. Small amounts of oxides such as MgO (2.07%) and SO₃ (0.80%) improve the stability of the clinker in sulfate environments and at the same time prevent undesirable reactions. The absence of Cl⁻ (0.0%) and the low content of free CaO (0.935%) greatly increase corrosion resistance. The low heat loss (1.14%) confirms the very good quality of the raw materials. The total oxide content of 100.0% indicates that the clinker composition is optimal and fully balanced.

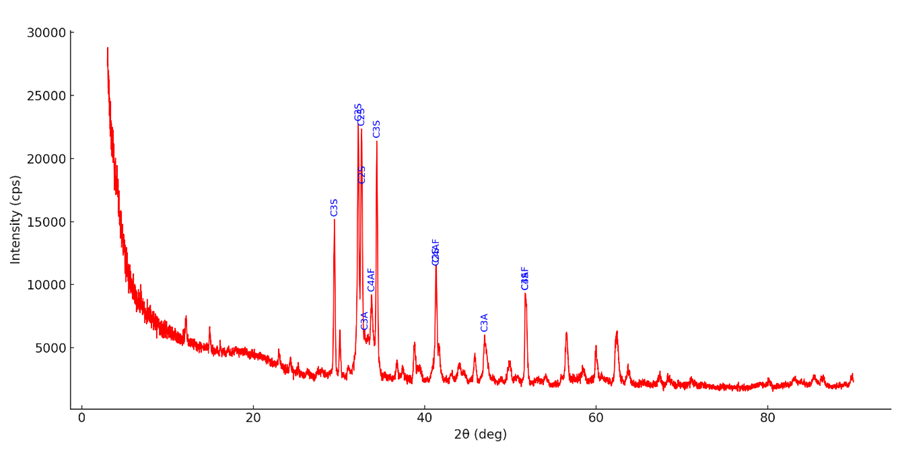
**TABLE 2.** Chemical and mineralogical composition of sulfate-resistant portland cement clinker

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Material name** | **LOI** | **SiO₂** | **Al₂O₃** | **Fe₂O₃** | **CaO** | **MgO** | **SO₃** | **Cl⁻** | **CaO (Free CaO)** | **Σ** |
| Portland cement clinker | 1.14 | 22.1 | 4.98 | 5.32 | 61.09 | 2.07 | 0.8 | 0.0 | 0.935 | 100.0 |
| Mineralogical composition (%) and module indices | | | | | | | | | |
|  | C₃S=38,64; C₂S=41,03; UCH KALSIY ALYUMINAT=4,17; C₄AF=16,16; CaO/SiO₂=2,76; LSF=0,86; SR=2,15; AR=0,94 | | | | | | | | |

In the mineralogical composition, the clinker is dominated by C₃S (38.64%) and C₂S (41.03%) phases, which primarily determine its mechanical properties. The relatively low content of tricalcium aluminate (4.17%, ≤5%) is the main indicator of sulfate resistance, since it greatly reduces the formation of ettringite. This is directly related to the low content of Al₂O₃ in the raw materials. The presence of the C₄AF (16.16%) phase indicated a high ferrite content in the clinker. This phase is formed due to Fe₂O₃ introduced through iron-rich industrial waste and, replacing part of the tricalcium aluminate, increases the strength of sulfate-resistant cement clinker. The calculated modulus values once again prove that the raw material mixture is in equilibrium. The CaO/SiO₂ ratio (2.76) — the silicon modulus — provides high mechanical strength of the cement. The lime saturation factor (LSF = 0.86) indicates that the firing process was carried out under optimal conditions. The aluminum modulus n (2.15) and iron modulus p (0.94) confirm that the oxides have a stable ratio. These results prove that it is possible to effectively produce sulfate-resistant Portland cement clinker based on local raw materials such as barren sand, iron-rich industrial waste, kaolin and limestone.

Reducing the amount of tricalcium aluminate can reduce the expansion and erosion of cement in sulfate environments by 15–25%, resulting in increased long-term durability of structures. However, to maintain optimal modulus values, it is necessary to precisely control the firing temperature in the range of 1350–1450 °C, as well as the time of burning the clinker in the kiln. In future studies, it is proposed to study the mineralogy of the raw material mixture in depth, analyze the hydration kinetics, and conduct mechanical tests in order to improve the efficiency of the synthesized clinker. The chemical composition and phase structure of the clinker were evaluated by X-ray phase analysis along with tabular data. The XRD diffractogram showed clear peaks corresponding to the main crystal phases, the presence of which confirmed the results obtained by the Bogue method (Fig. 1). The strongest diffraction peaks observed between 2θ ≈ 29–34° and 51–52° indicate the dominance of the C₃S and C₂S phases.

The high content of alite and belite phases is an important factor determining the main mechanical properties of clinker. The Alite mineral, which makes up the mineralogical composition of clinker, actively participates in the early days of the hydration process, ensuring rapid hardening of the cement stone from the first days. Belite, on the other hand, exhibits a very slow hydration property compared to allite and serves to increase the long-term strength of the cement stone in the period of 28 days and beyond. The high content of these two phases is the main reason for the rapid and very high-quality mechanical strengthening of the cement.

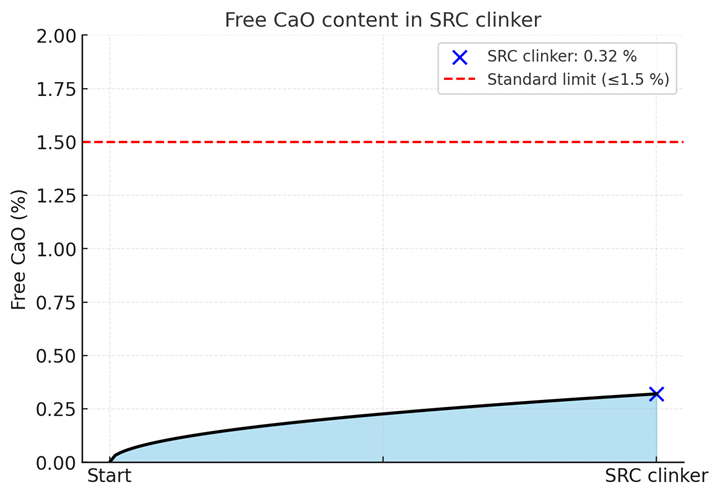


**FIGURE 1.** XRD pattern of sulfate-resistant portland cement clinker showing the main phases.

The results of the above data were found to be in full agreement with the analysis of the X-ray diffractogram. The most intense diffraction peaks were recorded in the range of 2θ = 28–350 and 50–530, which were found to correspond to the alite (C₃S) and belite (C₂S) phases. These indicators confirm the quantitative data obtained from Bogue calculations. The peaks around 2θ = 32–340 and 480 correspond very well to the tricalcium aluminate phase (C₃A = 4.2%) and indicate that its content in the clinker is very low. The low content of tricalcium aluminate clearly indicates that it is an important factor in increasing sulfate resistance. The peaks observed at 2θ = 32–340, 41–420 and 520 also correspond to the tricalcium aluminoferrite phase (C₄AF = 16.2%), which is in full agreement with the results of chemical and mineralogical analyses.

Thus, when the results of the chemical and mineralogical analyses were compared with the X-ray diffractogram, the predominance of alite and belite, the low content of tricalcium aluminate and the high content of C₄AF were scientifically confirmed. These characteristics allow us to consider the synthesized clinker as a sulfate-resistant Portland cement clinker.

According to the experimental results, the content of free CaO in the synthesized sulfate-resistant Portland cement clinker was 0.32%. This value is much lower than the regulatory requirements (≤1.5%), indicating complete reaction of lime in the clinker. On the one hand, this ensured the optimal formation of C₃S and C₂S phases, and on the other hand, it led to the minimal formation of the three calcium aluminate phases (see Fig. 2).

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**FIGURE 2.** Free CaO content in sulfate-resistant Portland cement clinker.

As can be seen from the graph, the measured value (0.32%) is much lower than the normative limit (1.5%). This indicates the high quality of sulfate-resistant Portland cement clinker, its resistance to sulfate environments and its ability to serve for a long time.

The fact that free CaO is lower than the required standard indicates the efficiency of the synthesis process. These results scientifically justify the possibility of using the obtained sulfate-resistant Portland cement clinker in the production of sulfate-resistant cement in accordance with the standards of the Republic of Uzbekistan St 337:2024 and GOST 22266-2013.

Sulfate resistance tests conducted according to the ASTM C1012/C1012M standard showed significant differences in cement samples. The degree of expansion observed in a Na₂SO₄ solution for 28 days was directly related to the composition of the mineral phases of the cement (see Table 3).

**TABLE 3.** ASTM C1012 sulfate resistance test – 28-day results

|  |  |  |
| --- | --- | --- |
| **Sample** | **Medium** | **Length (mm)** |
| **1.1** | Distilled Water | 16.1 |
| **1.2** | Distilled Water | 16.1 |
| **1.3** | 5% Na₂SO₄ | 16.1 |
| **2.1** | Distilled Water | 16.1 |
| **2.2** | Distilled Water | 16.1 |
| **2.3** | 5% Na₂SO₄ | 16.1 |
| **3.1** | Distilled Water | 16.0 |
| **3.2** | Distilled Water | 16.0 |
| **3.3** | 5% Na₂SO₄ | 16.0 |

The 28-day exposure results clearly show that the cement based on sulfate-resistant Portland cement clinker showed significantly higher durability than OPC (ordinary Portland cement) and blended cements (see Figure 3).

**FIGURE 3.** Expansion of cement samples (SULFATE RESISTANT PORTLAND CEMENT, OPC, and blended cement) after 28 days of exposure to 5% Na₂SO₄ solution.

First, the cement based on sulfate-resistant Portland cement clinker had the lowest expansion values. This is explained by the limited content of tricalcium aluminate in the composition (tricalcium aluminate ≤ 5%). Due to the low calcium aluminate, it reduces the reaction with sulfate ions and reduces the formation of excess ettringite or secondary gypsum. As a result, sulfate-resistant Portland cement exhibits long-term durability.

Second, ordinary Portland cement (OPC) showed high expansion. At the end of 28 days, the deformation values approached or exceeded the limit values specified in the ASTM C1012 standard. This is explained by the high content of three calcium aluminates in the OPC composition. Tricalcium aluminate actively reacts with sulfate ions, forming expansive ettringite.

Third, the mixed cements obtained from local raw materials showed lower expansion than OPC, but did not reach the level of sulfate-resistant Portland cement. This indicates that local mineral additives (kaolin, clay derivatives, iron-rich additives) have a partially limiting effect on the reaction with sulfate ions.

In general, experiments have shown that sulfate-resistant Portland cement, which can be produced in Karakalpakstan, has significantly higher resistance to sulfate-rich conditions - in particular, in the conditions of high-sulfate groundwater of the Aral Sea region - than ordinary Portland cement. These scientific results confirm the increased resistance of sulfate-resistant Portland cement to the effects of sulfate ions and its possible use as a promising construction material in aggressive environments.

**CONCLUSION**

The conducted studies comprehensively assessed the possibility of obtaining sulfate-resistant Portland cement clinker from local raw materials of the Karakalpakstan region - limestone, barren sand, kaolin and iron-rich industrial waste.

The analysis of the chemical composition showed that the raw mixture has an optimal balance of oxides CaO, SiO₂, Al₂O₃ and Fe₂O₃. As a result, the modulus values (LSF = 0.82; SR = 2.15; AR = 0.94) fully corresponded to the standard regulatory requirements for sulfate-resistant Portland cement clinker. Mineralogical analyses showed the predominance of alite (C₃S ≈ 38.6%) and belite (C₂S ≈ 41.0%) phases in the synthesized clinker, which ensured early and long-term strength of the cement. The most important indicator — the content of tricalcium aluminate (TRI CALCIUM ALUMINATE ≈ 4.2%) below 5% — ensured the resistance of the cement to sulfate environments. The high content of C₄AF due to the introduction of iron-rich additives further increased the stability of the clinker.

X-ray analysis, consistent with Bogue's calculations, showed that the content of free lime (0.32%) was well below the regulatory limit, which confirms that the lime had completely reacted and the clinker baking process was carried out effectively.

Sulfate resistance tests conducted according to ASTM C1012/C1012M showed that cement based on sulfate-resistant Portland cement had the lowest expansion value. Ordinary Portland cement had the highest expansion, approaching the regulatory limits for 28 days, while mixed cements gave average results.

Finally, it has been scientifically proven that sulfate-resistant Portland cement clinker synthesized from local raw materials has high stability in aggressive sulfate environments. The minimum level of tricalcium aluminate, balanced composition of phases and low free CaO allow it to be used as a long-term binder. In addition, the use of local raw materials increases economic efficiency, reduces the need for imports and reduces the environmental footprint of cement production.

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