BASED ON THE LIMESTONE FROM THE “UCH O‘CHOQ” DEPOSIT AND THE MARL CLAY FROM THE “MESHIKLI” DEPOSIT, ENERGY-EFFICIENT PORTLAND CEMENT CLINKER

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Abstract. *This article studies the processes of obtaining Portland cement clinker using raw materials: limestone from the “Uch O‘choq” deposit and marl clay from the “Meshikli” deposit (Lower Amudarya region), as well as pyrite cinder from JSC “Almalyk mining and metallurgical combine”. The chemical and mineralogical composition of the raw mixtures was calculated, and the influence of the saturation coefficient and silica modulus on the formation of clinker phases was investigated. Under laboratory conditions, the quality of the clinker was assessed by the content of free calcium oxide (f-CaO), as well as by X-ray phase and electron microscopic analyses. The obtained results demonstrate the possibility of obtaining high-quality clinker at lower burning temperatures using limestone from the “Uch O‘choq” deposit, marl clay from the “Meshikli” deposit, and pyrite cinder from JSC “Almalyk mining and metallurgical combine”.*

Keywords: *portland cement clinker, Lower Amudarya, limestone from the “Uch O‘choq” deposit, marl clay from the “Meshikli” deposit, pyrite cinder from JSC “Almalyk mining and metallurgical combine”, burning process, f-CaO, mineralogical composition.*

Introduction

Globally, Portland cement consumption increased 3.5-fold between 1990 and 2019, and nearly 7-fold between 1970 and 2020. The majority of cement (73%) was used in concrete production. Portland cement reserves, which represent the amount of cement embodied in various construction structures and buildings, reach 12–30 tons per capita in some developed countries. Per capita Portland cement consumption typically ranges from 0.4 to 0.8 tons. After reaching a saturation point, cement consumption stabilizes and may subsequently decline [1].

The mineralogical composition of Portland cement clinker depends on the properties of the raw materials used. In multi-component raw mixtures, creating optimal conditions for mineral formation is one of the most challenging tasks, as the resulting crystalline structure determines the quality of the clinker and cement [2, 3]. Even with identical chemical composition and the same modulus values, a raw mix can yield clinker with different reactivity and mineralogical composition depending on the type of mill used for mechanical activation [4].

The dependence of Portland cement clinker synthesis on heat-engineering and technological parameters, as well as issues related to reducing material and energy consumption and increasing clinker reactivity, have been studied in detail by N.Kh. Babaev [5, 6, 7, 8, 9, 10]. In his published scientific works, new theoretical foundations have been proposed for increasing clinker reactivity. These include reducing fuel consumption by optimizing combustion conditions for high-performance clinker production, partially replacing limestone and clay components in the raw mix with industrial wastes from the chemical and metallurgical industries, and accelerating clinker burning processes without compromising clinker quality. The results of these studies have been implemented in optimizing the technological performance of Portland cement plants in Uzbekistan and Kazakhstan.

Materials and Methods

In the research, the method accepted for three-component compositions was used to calculate the composition of the raw mixture for Portland cement clinker. As raw materials, limestone from the “Uch O‘choq” deposit, marl clay from the “Meshikli” deposit (both located in the Lower Amudarya region), and pyrite cinder (a byproduct from Almalyk MMC JSC) were used.

The chemical composition of the raw materials was determined based on laboratory analyses. According to the calculations, the saturation coefficient (SC) was taken in the range of 0,84–0,95, and the silica modulus (n) was taken in the range of 1,8–2,3. The composition of the mixtures was calculated with an accuracy of 0,01%.

The burning (sintering) processes were carried out in a laboratory electric furnace at temperatures between 1300 and 1420 °C. During the burning process, the content of free calcium oxide (free lime, f-CaO) in the samples was determined using the ethyl-glycerate method in accordance with GOST 5382. The phase composition of the clinker was studied using X-ray phase analysis (XRD), and the microstructure was studied and analyzed using electron microscopy methods.

Results. In the process of calculating the composition of the raw mix for Portland cement clinker, the method adopted for calculating three-component mixtures was used [11].

Several calculations of the composition of different three-component raw mixes were carried out, in which limestone from the “Uch O‘choq” deposit and marl clay from the “Meshikli” deposit in the Lower Amudarya region were taken as the carbonate and aluminosilicate components. Pyrite cinder from “Olmalyk MMC” JSC was used as the iron-rich component [12, 13, 14].

The raw mix used to obtain the clinker consisted of limestone, clayey marl, and pyrite cinder. The chemical composition of the materials used is presented in Table 1.

In the calculations, the saturation coefficient (SC) was taken in the range of 0,84–0,95, and the silicate modulus (n) was taken in the range of 1,8–2,3. The variation of the mix composition within the specified range can be observed in the data presented below. The calculations were carried out with an accuracy of 0,01%.

Table 1. Chemical composition of raw materials

Raw material name	**Oxide content, wt. %**							
	SiO_2	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**SO_3**	**LOI**	**Total**
Limestone from the “Uch O'choq” deposit	4,96	0,90	0,97	52,47	0,68	0,76	39,26	100,00
Marl clay from the “Meshikli” deposit	35,49	9,11	2,97	24,58	1,57	0,40	25,88	100,00

Pyrite cinder from “Olmalyk MMC” JSC	14,14	1,46	79,53	1,23	0,22	1,93	1,49	100,00

According to calculations, the computed composition of the raw material mixture consists of: limestone from the “Uch O‘choq” deposit – 65,46–69,21%; marl clay from the “Meshikli” deposit – 27,25–31,58%; and pyrite cinder – 2,73–3,79%. The calculated mineralogical composition of the raw mixtures shows: C_3S = 45,52–69,86%; C_2S = 9,20–31,83%; C_3A = 1,35–6,39%; C_4AF = 17,46–21,34%. With an increase in the lime saturation factor (LSF), the C_3S content increases. An increase in the silicate modulus from 1,8 to 2,0 leads to an increase in the C_3A content from 1,35% to 6,39%. At the minimum LSF (LSF = 0,84), the belite content in the mixture reaches its maximum (C_2S = 31,83%). It was observed that the C_4AF content in the mixture is high (21,34%) at low values of the silicate modulus and low (17,46%) at high values of the silicate modulus.

The calculation results indicate that with an increase in the LSF of the mixture, the C_3S content in the clinker increases, making it possible to obtain high-strength clinker. An increase in the silicate modulus reduces the iron-rich component in the raw feed, leading to a decrease in the liquid phase. Consequently, the burnability conditions of the raw mixture become more difficult.

Taking the above into account, for further research, a raw mixture with LSF = 0,93 and silicate modulus (n) = 2,3 was adopted as the optimal composition (Table 2).

Table 2. Chemical composition of the raw mixture and clinker

№	**Raw material name**	**Oxide content, wt. %**							
		SiO_2	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**SO_3**	**LOI**	**Total**
1	Limestone from the “Uch O'choq” deposit	3,41	0,62	0,67	36,07	0,47	0,52	26,99	68,74
2	Marl clay from the “Meshikli” deposit	10,53	2,70	0,88	7,29	0,47	0,12	7,68	29,66
3	Pyrite cinder from “Olmalyk MMC” JSC	0,26	0,02	1,27	0,02	0,01	0,03	0,02	1,60
4	Raw mixture	14,16	3,34	2,82	43,38	0,94	0,67	34,69	100,00
5	Clinker	21,69	5,12	4,31	66,43	1,43	1,02	0	100,00

Calculation coefficient K = 1,531237

Component quantities: Limestone from the “Uch O‘choq” deposit – 68,74%; Marl clay from the “Meshikli” deposit – 29,66%; Pyrite cinder from JSC “Olmalyk MMC” – 1,60%.

Mineralogical composition of the clinker (%): C_3S = 66,66; C_2S = 13,47; C_3A = 6,39; C_4AF = 13,43.

The above-selected optimal composition mixture was ground in a laboratory mill. The residue of the mixture on a №008 sieve was 12%. A small amount of water was added to the prepared mixture until its moisture content reached 6–8%, and it was passed through a № 1 sieve several times to ensure uniform distribution of moisture in the mixture.

From the mixture with a moisture content of 6–8%, raw material samples with a diameter of 50 mm and a height of 0,8–1,2 mm were prepared using a mechanical hand press. The shaped samples were labeled and first dried at ambient temperature, and then in a laboratory drying oven at 105–110 °C for 3 hours. For comparison of the test results, samples were also prepared from the raw mixture produced at JSC "BEKOBODSEMENT" following the same procedure.

The samples prepared for testing and comparison were fired in a programmable laboratory electric furnace designed for 1500 °C at temperatures ranging from 1300 °C to 1420 °C, and the content of unbound free calcium oxide (f-CaO) was determined. The holding time at the maximum temperature was 30 minutes, which was intended to ensure uniform temperature distribution and the formation of clinker minerals.

The determination of the unbound free calcium oxide (f-CaO) content was carried out using the ethylene glycol method of chemical analysis in accordance with GOST 5382. The results of the determination of the unbound calcium oxide (f-CaO) content in the firing products are presented in Table 3.

Table 3. Content of unbound f-CaO in fired samples

№	**Name of the raw material mixture**	**f-CaO content, mass %**			
		1300 ºC	**1350 ºC**	**1380 ºC**	**1420 ºC**
1	**Experimental clinker**	8,8	7,1	1,9	1,1
2	**Clinker of JSC "Bekobodsement"**	8,8	5,0	2,3	1,8

The kinetics of f-CaO assimilation are presented in Figure 1. From the calcium oxide assimilation kinetics, it can be seen that although the f-CaO content in the experimental mixture and in the mixture from JSC "Bekobodsement" is the same at a temperature of 1300 °C, by 1420 °C the difference in f-CaO assimilation becomes significant. It should also be noted that the f-CaO content in the experimental mixture at a temperature of 1380 °C meets the requirements of GOST 34850. Thus, when the experimental mixture is fired at 1380 °C, a clinker that meets the requirements was obtained. The clinker firing temperature is 40 °C lower than that of the plant clinker.

Thus, in the mixtures obtained from the Lower Amu Darya limestone-based raw materials under study, clinker formation reactions were completed at temperatures 30–70 °C lower than those for clinker produced by conventional technology. When the constituents of the chemical composition of limestone from the "Uch O'choq" deposit and marl clay from the "Meshikli" deposit in the Lower Amu Darya region – together with an iron additive (pyrite cinder) – are introduced into the clinker raw mixture, earlier formation of alite and belite occurs, creating favorable conditions for lowering the firing temperature and accelerating clinker burning. It was determined that in the experimental clinker fired at 1380 °C, the f-CaO content is 1,9%.

During clinker formation, only a part of the solid materials forms a liquid phase. The formation of the liquid phase depends on the chemical composition of the mixture, the properties of the raw materials, and the firing temperature and duration.

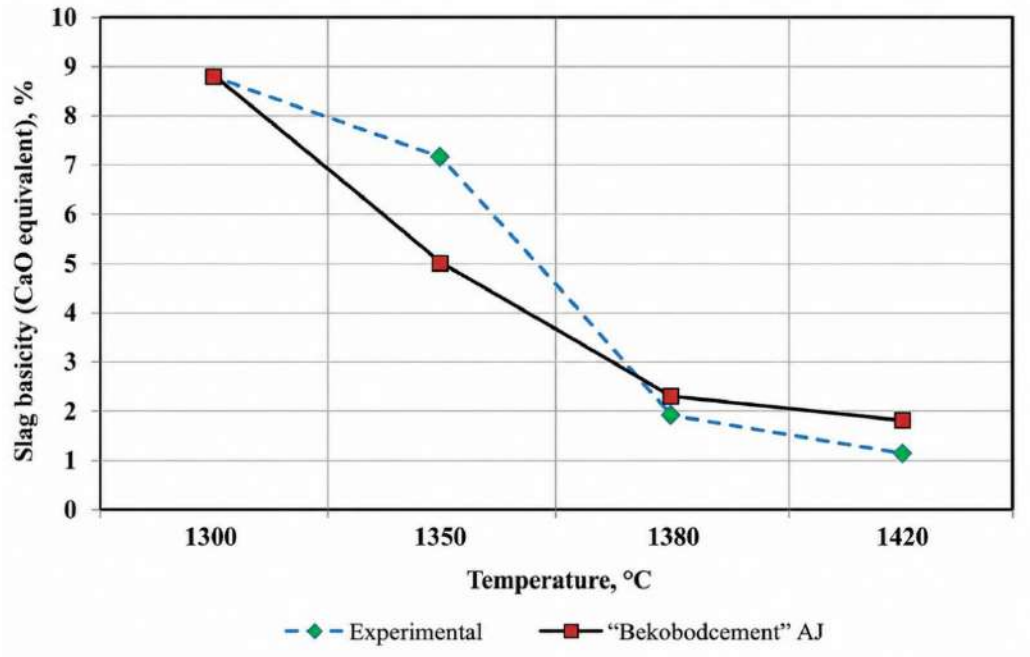

Figure 1. Kinetics of calcium oxide assimilation

Analyzing the processes occurring in clinker kilns in general terms: at 100 °C, free physically bound water is removed from the raw mixture. In the temperature range of 500–900 °C, dehydration of clay minerals occurs. In the range of 900–1200 °C, $CaCO_3$ decomposes, and the resulting CaO combines with aluminosilicates. In the range of 1250–1280 °C, a liquid phase forms. Above 1280 °C, the formation of clinker minerals is completed with the participation of the liquid phase. The maximum high temperature inside the kiln is observed in the burning zone, approximately 5–12 m from the hot end of the kiln. The maximum temperature in the kiln is selected so that 20–30% of the material forms a liquid phase, which, during burning, ensures the appearance of small, rounded particles. After the burning zone, the clinker temperature decreases and enters the cooler at approximately 1000 °C. The raw material passes through the burning zone of the kiln in about 20 minutes.

At the clinker formation temperature, the material consists mainly of C_3S and C_2S crystals. Simultaneously with the crystals, a liquid phase containing CaO, Al_2O_3, Fe_2O_3, and MgO is also present in the material. Small amounts of SiO_2 may be present in the liquid phase. Therefore, the aluminate and aluminoferrite phases of the clinker are formed only during the cooling process. The oxides Al_2O_3 and Fe_2O_3 are the primary liquid-phase formers during clinker burning. At 1400 °C, Al_2O_3 plays a major role, while at temperatures below 1400 °C, Fe_2O_3 is more significant.

The cooling rate of the clinker affects the mineral content of the clinker. Portland cements produced from slowly cooled clinkers may exhibit "false set". False set reduces the strength of Portland cement. When the same clinker is rapidly cooled, false set does not occur, and the Portland cement hardens normally.

The reactivity of the raw mixture depends not only on its chemical composition but also on the mineralogical composition of its components and their particle size. The degree of crystallinity of certain minerals is important here; the presence of various defects in crystals increases reactivity.

The particle size of limestone and quartz greatly influences the reactivity of the raw mixture. Quartz is the component with the lowest reactivity. Quartz present in the clay is more favorable than that in the limestone. If the raw mixture is not well mixed, the reactions of clinker mineral formation do not proceed sufficiently, resulting in reduced alite formation. This, in turn, leads to the formation of belite instead of alite and the presence of unbound f-CaO.

Taking the above into account, we programmed the laboratory furnace as follows: time to reach the maximum temperature – 2 hours; holding time at the maximum temperature – 30 minutes; cooling to 1000 °C – with the power turned off; and after 1000 °C – rapid cooling.

The appearance of clinkers fired at 1380 °C under laboratory conditions is shown in Figures 2 and 3.

Figure 2. Clinker fired at 1380 °C from the raw material mixture of JSC “Bekobodsement”

Figure 3. Clinker fired at 1380 °C from the experimental raw material mixture

Figure 2 shows the appearance of clinker obtained from the raw mixture of JSC “Bekobodsement” at a temperature of 1380 °C. It can be seen that due to the low temperature being insufficient for the formation of clinker minerals, there are particles that did not participate in the process. In the image, they appear as white particles. The f-CaO content is 2,3%, as presented in Table 3.

The high f-CaO content does not meet GOST requirements, and a further increase in temperature is required to complete the formation of clinker minerals.

Figure 3 shows the appearance of clinker obtained from the experimental raw mixture at a temperature of 1380 °C. It can be seen that the temperature is sufficient for the formation of clinker minerals. The image shows that the clinker is well burned. The f-CaO content is 1,9%, as presented in Table 3. The f-CaO content meets GOST requirements.

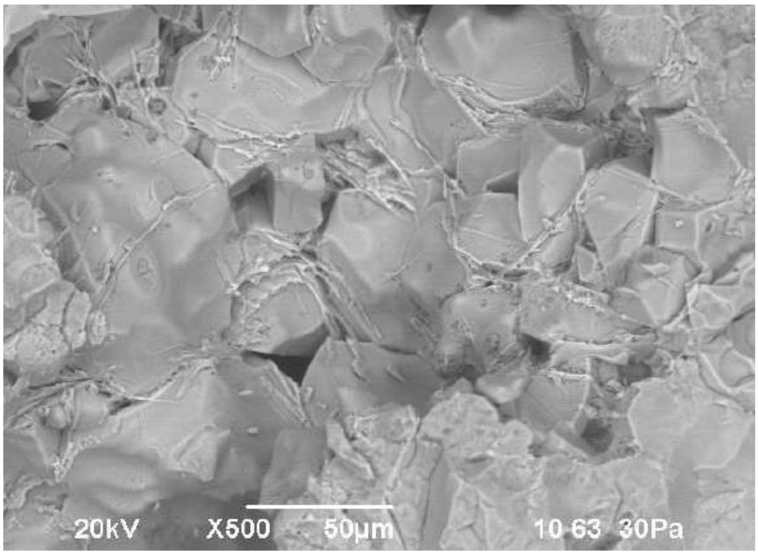

Figure 4. Microstructure of the experimental clinker (1380 °C), x500

Figure 4 shows the microstructure of the experimental clinker fired at 1380 °C. The obtained clinker microstructure is distinguished by its unique characteristics. The clinker contains

1,9% f-CaO. The crystals are sufficiently well formed. Alongside large alite crystals, one can also see alite intergrown with belite. In some cases, poorly crystallized alite is observed. Fine-crystalline belite is secondary, formed from the decomposition of alite into belite and free CaO. The interstitial phase is observed in large quantities and consists mainly of calcium aluminoferrite (C_4AF). Our conclusions based on the conducted experiments are consistent with [15].

Thus, from the electron-microscopic analysis of the experimental clinker, it can be concluded that the Lower Amu Darya limestone raw materials rank among high-quality raw materials for clinker production, enabling the completion of clinker mineral formation at 1380–1420 °C. The possibility of obtaining high-quality clinker by firing raw material mixtures is confirmed by chemical (f-CaO content), X-ray, and electron-microscopic analyses**.**

Discussion

The research results showed that the limestone from the “Uch O‘choq” deposit, the marl clay from the “Meshikli” deposit, and the pyrite cinder from JSC “Almalyk MMC” available in the Lower Amu Darya region are technologically suitable raw materials for Portland cement clinker production. The determined chemical and mineralogical composition serves to accelerate the clinker mineral formation process in mixtures prepared from these raw materials.

The reduction in clinker burning temperature is primarily related to the optimal content of aluminum and iron oxides in the raw materials, which facilitate the formation of the liquid phase. The earlier appearance of the liquid phase ensures active interaction of calcium oxide with the aluminosilicate phases, accelerating the formation of the alite phase. Furthermore, the highly reactive aluminosilicate minerals present in the marl clay from the “Meshikli” deposit also positively affect the clinker formation kinetics.

Comparison of the experimental raw mixture and that of the industrial enterprise (JSC “Bekobodsement”) showed that although the f-CaO content at 1300 °C is almost the same in both cases, at higher temperatures the calcium oxide assimilation process proceeds more actively in the experimental mixture. This indicates the higher reactivity of the mixture prepared from the limestone of the “Uch O‘choq” deposit and the marl clay of the “Meshikli” deposit in the Lower Amu Darya region.

The results of electron microscopy analysis showed that the clinker microstructure is well-formed. The presence of large and medium-sized alite crystals in the clinker, their balanced distribution with the belite phase, and the sufficient amount of interstitial phase confirm that the clinker has high quality indicators. The relatively low content of f-CaO indicates that the burning process has been fully completed.

Overall, the above results demonstrate that Portland cement clinker can be produced in energy-efficient modes using the specified raw materials. This is important not only for reducing fuel consumption but also for increasing the economic efficiency of cement production.

Conclusion

As a result of the conducted research, it was determined that the limestone from the “Uch O'choq” deposit, the marl clay from the “Meshikli” deposit in the Lower Amu Darya region, and the pyrite cinder additive from JSC “Almalyk MMC” are technologically suitable raw materials for the production of Portland cement clinker. The selection of the raw mixture at optimal values of the lime saturation factor and silicate modulus facilitates the effective formation of clinker mineral phases.

Laboratory experiments have shown that in clinker fired at a temperature of 1380 °C, the unbound f-CaO content is within the regulatory requirements, and the complete formation of alite and belite phases is ensured. This confirms the possibility of firing clinker at lower temperatures compared to conventional technology.

The results of X-ray phase and electron microscopic analyses showed that the mineralogical composition and microstructure of the obtained clinker exhibit high quality indicators. In particular, the adequate formation of alite crystals and the stable distribution of interstitial phases serve to increase the technological activity of the clinker.

Thus, it has been scientifically substantiated that by utilizing the raw material resources of limestone from the “Uch O'choq” deposit, marl clay from the “Meshikli” deposit in the Lower Amu Darya region, and pyrite cinder from JSC “Almalyk MMC”, it is possible to produce energy-efficient, economically viable, and high-quality Portland cement clinker. The obtained results reveal the prospects for industrial-scale application of these raw materials

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