PROPERTIES OF PORTLAND CEMENT PRODUCED USING ARGILLACEOUS SHALE AND METALLURGICAL SLAG

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ABSTRACT

This study presents the results of investigations into the chemical and mineralogical properties of clay shales and metallurgical slags with the aim of producing Portland cement based on these industrial materials. Three-component raw mixtures for Portland cement were designed, and the optimal composition was identified with a lime saturation factor (LSF) of 0.91 and a silica modulus (SM) of 2.3. Following thermal treatment, Portland cement clinker was successfully synthesized, exhibiting the following phase composition: C_3S - 58.89 %, C_2S - 17.71 %, C_3A - 7.14 %, and C_4AF - 12.46 %. The physical and mechanical properties of the resulting cement were evaluated. After 28 days of curing under standard conditions, the cement demonstrated a flexural strength of 6.5 MPa and a compressive strength of 41.8 MPa. The results confirm the feasibility of producing Portland cement with a standard phase composition and satisfactory mechanical performance using metallurgical slags and clay shale as alternative raw materials.

Keywords: Portland cement, metallurgical slags, industrial waste, argillaceous shale, X-ray phase analysis, mineralogical composition, phase.

INTRODUCTION

The rapid growth of the construction industry worldwide has led to a sharp increase in the demand for building materials, among which various types of Portland cement occupy a dominant position. The technological performance of Portland cement is largely governed by the properties of the raw material compositions, which are typically based on natural limestone, clay, and iron-containing components. It should be noted that the reserves of these natural raw materials are limited and continue to decline over time, highlighting the need for alternative resources and sustainable production approaches [1 - 5].

Therefore, there is an urgent need to ensure a stable and uninterrupted supply of diverse raw material components for the construction industry. At present, expanding the raw material base of the construction sector through the utilization of non-traditional resources and industrial wastes remains a highly relevant and pressing challenge. Addressing this issue requires a fundamental shift in the approach to raw material selection, with an emphasis on sustainability and resource efficiency [6 - 9].

In this context, the utilization of clay shales from areas unsuitable for agricultural use, along with metallurgical slag wastes, as promising alternative raw materials for Portland cement production represents a highly relevant and timely research direction.

Clay shales are sedimentary rocks composed predominantly of clay minerals, including hydromicas (illite group minerals), chlorites, kaolinite, and others, whose particles are typically oriented in a highly parallel arrangement. As a result of this structure, clay shales

exhibit pronounced fissility, i.e., the ability to split into thin laminae. Unlike many clay-rich sediments, clay shales do not disintegrate in water, which is characteristic of deposits formed under geosynclinal conditions [10, 11]. They are formed through the compaction of clay sediments accompanied by partial recrystallization during burial at significant depths under the influence of dynamometamorphism. With further metamorphic transformation, clay shales can evolve into phyllites or chlorite schists. Major deposits of clay shale are found in the Caucasus and the Ural regions, and they also occur in Central Asia.

Clay shales are composed of very fine clay particles, primarily kaolinite or montmorillonite, along with significant amounts of micaceous minerals, as well as fine fractions of feldspar dust, quartz grains, and other mineral constituents.

In terms of chemical and mineralogical composition, clay shales are similar to clay materials traditionally used in the production of Portland cement clinker. Therefore, investigating their potential use as a substitute for natural loess and kaolinitic clays in clinker production is of considerable interest, particularly for expanding the raw material base of the cement industry.

At present, extensive fundamental and applied research is being actively conducted worldwide on the utilization of industrial wastes and secondary resources through the development of innovative compositions of construction materials with enhanced technological performance [12 - 17]. Since Portland cement production is highly resource- and energy-intensive, requiring substantial amounts of mineral raw materials and energy, contemporary research efforts are increasingly focused on incorporating alternative raw materials, secondary resources, and industrial wastes into cement manufacturing processes. Attention has been devoted to oil and clay shales, basaltic and effusive rocks, volcanic ashes and thermal power plant ash-slag wastes, as well as flotation tailings and metallurgical slags, among other solid industrial by-products.

EXPERIMENTAL

Materials

Clay shale from the Devanasay occurrence, located in the Samarkand region, Uzbekistan, was investigated in this study. Limestone from the Jizzakh region, Uzbekistan was used as the carbonate component, while processed metallurgical slags from JSC "Uzmetkombinat" (Bekabad, Uzbekistan) served as the iron-bearing component.

Methods

The chemical composition of the raw materials was determined by comprehensive silicate analysis in accordance with GOST 5382 - 2019 [18].

The phase composition of the minerals in both the initial components and the synthesized clinker samples was analysed using X-ray diffraction (XRD) on a LABX XRD-6100 diffractometer (SHIMADZU, Japan) with CuKα radiation (Ni -filter, wavelength λ = 1.5418 Å). The operating conditions included a tube current of 30 mA and a voltage of 30 kV.

Data acquisition was performed at a constant detector scanning rate of 4°/min with a step size of 0.02° (ω/2θ configuration), over a 2θ range of 4 - 80°. Phase identification and quantitative analysis were carried out using the International Centre for Diffraction Data (ICDD) Powder Diffraction File (PDF-2, 2007 release) database, along with established standard methods for evaluating the properties of Portland cement.

For the experimental study, a representative composite sample was prepared from ten clay shale specimens collected from different locations across the Devanasay occurrence. This averaged sample was used for determining the mineralogical composition and conducting physicochemical analyses.

Chemical analysis was performed on the averaged clay shale samples, as well as on the limestone used as the carbonate component in Portland cement production, and the processed iron-containing slags.

The results of the chemical analysis (Table 1) of the averaged samples of the investigated components (limestone, clay shales, and metallurgical slags) indicate that their compositions are relatively stable. In addition to the major oxides SiO_2, Al_2O_3, CaO, and MgO, the materials also contain appreciable amounts of iron oxides (FeO and Fe_2O_3). According to the literature, these oxides constitute the essential components required for the formation of Portland cement clinker.

The X-ray diffraction patterns of the Devanasay clay shale (Fig. 1) exhibit reflections at d = 0.425, 0.334, 0.245, 0.228, 0.223, 0.212, 0.199, 0.181, 0.166, 0.154, 0.138, and 0.137 nm, corresponding to quartz.

Table 1. Chemical composition of the investigated raw materials.

Raw materials	Content of oxides, wt. %								LOI, wt. %
	SiO_2	Al_2O_3	Fe_2O_3	FeO	MgO	CaO	Na_2O	K_2O	
Limestone 1	1.55	0.60	0.14	-	0.60	53.4	0.04	0.06	42.25
Limestone 2	1.35	0.55	0.11	-	0.30	55.37	0.02	0.05	41.05
Limestone 3	1.47	0.67	0.18	-	0.45	55.14	0.03	0.06	41.93
Shale 1	64.90	15.10	3.89	2.66	2.82	2.90	1.56	2.03	3.20
Shale 2	65.23	15.89	4.25	2.34	2.74	2.55	1.89	2.23	2.80
Shale 3	65.14	16.12	3.92	2.48	2.69	2.63	1.61	2.11	3.00
Slag 1	20.11	7.25	16.12	4.30	14.20	36.07	0.10	0.20	1.25
Slag 2	21.54	6.95	17.80	4.15	13.24	33.26	0.06	0.24	1.11
Slag 3	19.48	7.32	17.05	3.97	13.96	35.28	0.03	0.14	1.18

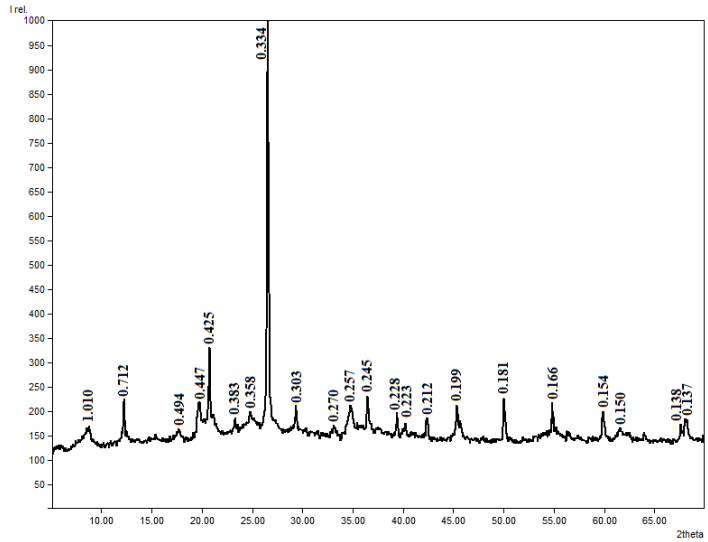


Fig. 1. X-ray diffraction pattern of Devanasay clay shale.

Additional reflections at d = 0.712, 0.447, 0.383, 0.358, 0.228, and 0.199 nm are attributed to kaolinite, while illite is identified by characteristic reflections at d = 1.010, 0.494, 0.447, and 0.257 nm. Minor diffraction peaks corresponding to calcite are also observed at d = 0.383, 0.303, and 0.245 nm.

The X-ray diffraction patterns of the Jizzakh limestone and metallurgical slag were previously reported in our earlier study [5].

Thus, based on the results of chemical and X-ray phase analyses of the Devanasay clay shale, it can be concluded that its chemical composition and mineralogical characteristics provide a solid basis for designing raw mixes suitable for Portland cement clinker production. To evaluate this potential, a series of three-component experimental raw mixtures were prepared with varying LSF and SM. The compositions of the Portland cement raw mixes were calculated using standard and widely accepted methodologies [19 - 21]. As a result of the laboratory investigations, an optimal composition was selected from the prepared series, characterized by an LSF of 0.91 and an SM of 2.3 (Table 2).

The study of the melting behaviour of the raw mixture (Table 3) showed that both the onset and completion temperatures of melting are significantly lower compared to conventional industrial compositions, where the melting temperature typically ranges from 1380 to 1450°C. This reduction in melting temperature can be attributed to the presence of FeO in the processed

Table 2. Calculated composition of the experimental clinker.

Raw materials	Composition of the raw mix, wt. %	Content of oxides, wt. %							LOI, wt. %
		SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	R_2O	
Limestone	78.73	1.23	0.48	0.11	42.445	0.48	0.08	0.04	33.58
Argillaceous shale	18.68	12.30	2.86	1.24	0.53	0.27	0.02	0.52	0.61
Slag	2.58	0.73	0.16	1.36	0.09	0.08	0.01	0.05	0.00
Raw mix composition	-	14.26	3.50	2.70	43.06	0.83	0.11	0.61	34.19
Clinker composition	-	21.67	5.32	4.10	65.43	1.26	0.17	0.93	0.00

Table 3. Composition and melting temperature of the raw mix.

LSF	SM	Mix composition, wt. %			Melting temperature, °C	
		Limestone	Argillaceous shale	Metallurgical slag	Initial	Final
0.91	2.3	79.10	18.73	2.17	1320	1390

slags, as well as low-melting compounds contained in the clay shale. These components promote fluxing effects, thereby lowering the firing temperature required for the formation of Portland cement clinker.

The results of the melting temperature determination for the cement raw mixtures confirm our assumption that the incorporation of Devanasay clay shales and metallurgical slags into three-component raw mixes enables the production of Portland cement clinker at reduced firing temperatures.

To obtain reliable information on the mineral formation process and to validate the results, the clinker samples produced from the investigated compositions were subjected to X-ray diffraction (XRD) analysis.

The XRD analysis of the experimental clinker (Fig. 2) revealed that the formation of clinker minerals begins at temperatures of 1320 - 1390°C. With increasing temperature, only minor amounts of free CaO were detected. The diffraction pattern shows low-intensity reflections at d = 0.240, 0.170, 0.145, and 0.138 nm, corresponding to uncombined CaO, which indicates a high degree of completion of clinker mineral formation.

At the same time, well-defined reflections corresponding to C_3S were observed at d = 0.302, 0.277, 0.273, 0.260, 0.217, and 0.162 nm, while reflections corresponding to C_2S appeared at d = 0.278, 0.273, 0.260, 0.240, and 0.218 nm. In some cases, the reflections of C_3S overlap with those of C_2S.

The results of the chemical analysis (Table 2) indicate that the experimental Portland cement clinker meets the requirements for industrial clinker in terms of its major oxide composition. The calculated mineralogical composition further confirms that the contents of the principal clinker phases fall within standard ranges, with C_3S = 58.89 %, C_2S = 17.71 %, C_3A = 7.14 %, and C_4AF = 12.46 %, in accordance with established standards [22].

Since the physical and mechanical properties of Portland cement produced from the experimental clinker are of critical importance, laboratory tests were conducted to determine the setting times of the cement paste and its strength characteristics under flexural and compressive loading (Tables 4 and 5), in accordance with the requirements of GOST 310.1 - 76 to 310.4 - 81 [23].

The setting times of the prepared cement paste (initial setting: 2 h 30 min; final setting: 3 h 50 min) fall within the permissible limits specified by current standards. The flow diameter of the cone (115 mm) also indicates that the selected water-to-cement ratio corresponds to the standard consistency of the cement paste.

The results of the physical and mechanical testing of Portland cement specimens based on Devanasay clay shale (Table 5), conducted in accordance with GOST

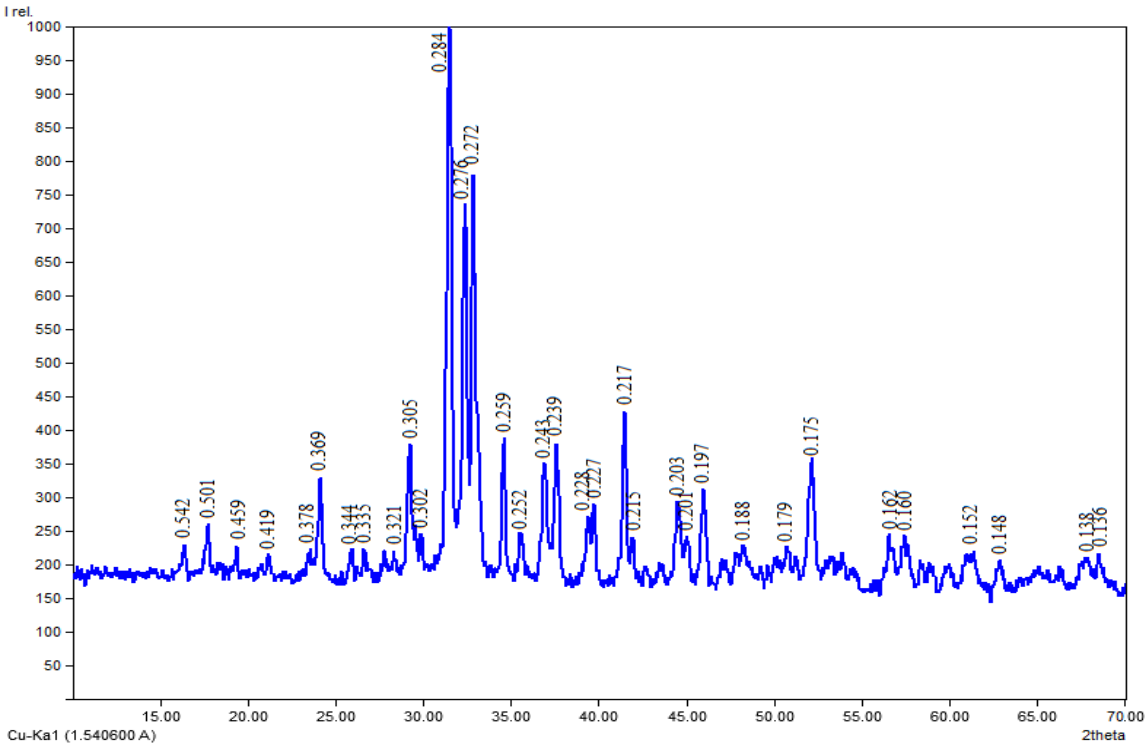


Fig. 2. X-ray diffraction patterns of the experimental clinker with LSF = 0.91 and SM = 2.3 fired at 1400°C.

Table 4. Setting times and standard consistency of the experimental cement.

LSF	SM	Initial set, h : min	Final set, h : min	Residue on 80 μm sieve, %	Water / cement	Cone spread, mm
0.91	2.3	2:30	3:50	8.0	0.42	115

Table 5. Physical and mechanical properties of the experimental cement.

LSF	SM	Hydrothermal treatment at 95 ± 5°C		Normal curing			
				3 days		28 days	
		Flexural strength, MPa	Compressive strength, MPa	Flexural strength, MPa	Compressive strength, MPa	Flexural strength, MPa	Compressive strength, MPa
0.89	2.5	4.6	31.0	4.7	29.8	6.5	41.8

requirements, demonstrate that the samples exhibit satisfactory strength properties.

After 28 days of curing under standard conditions, the flexural strength reached 6.5 MPa, while the compressive strength attained 41.8 MPa. It should also be noted that under hydrothermal curing at 95 ± 5 °C, the samples achieve 50 - 60 % of their design strength.

CONCLUSIONS

The results of this study demonstrate that Devanasay argillaceous shale and metallurgical slag can be effectively utilized as alternative raw materials for the production of Portland cement clinker. Chemical and X-ray phase analyses confirmed that the investigated

materials contain the essential oxides required for clinker formation, enabling the design of balanced three-component raw mixes. The optimal composition with a lime saturation factor (LSF) of 0.91 and a silica modulus (SM) of 2.3 was successfully identified.

The incorporation of clay shale and metallurgical slag led to a noticeable reduction in the melting and firing temperatures (1320 - 1390°C) compared to conventional clinker production. This effect is attributed to the presence of fluxing components, such as FeO and low-melting phases, which accelerate solid-state reactions and promote the formation of clinker minerals. XRD analysis confirmed the formation of well-crystallized C_3S and C_2S phases with minimal free CaO, indicating a high degree of reaction completion.

The synthesized cement exhibited satisfactory physical and mechanical properties, including standard setting times and strength values of 6.5 MPa (flexural) and 41.8 MPa (compressive) after 28 days of curing. The results confirm that the proposed approach not only ensures compliance with industrial requirements but also contributes to reducing energy consumption and expanding the raw material base through the efficient utilization of industrial waste.

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Authors' contributions

Kh.U.: writing original draft preparation, data curation; Z.K.: validation; investigation; F.Kh.: review and editing, formal analysis, data analysis and interpretation; F.U.: methodology.

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