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Aspects of cement clinker production using various types of industrial waste

ABSTRACT

Silicate systems with technogenic raw materials as starting mixtures for the production of cement clinker have been studied. The features of the chemical and mineralogical composition of high-tonnage waste from the thermal power industry – fly ash and metallurgy – slag from the production of ferroalloys have been shown. Using the computer program "Clinker", calculations and analysis of the possible content of the studied waste in the initial raw material mixture based on the specified clinker characteristics were carried out. New compositions of raw material mixtures based on chalk-fly ash-slag systems with a total content of 24 wt.% of the specified wastes have been determined. The possibility of obtaining normal- and rapid-hardening cement of medium strength from such mixtures has been experimentally confirmed. At the same time, the relationship between the properties of cement and the characteristics of the clinker composition regarding the formation of crystalline phases of silicates and aluminoferritic calcium is shown.

Keywords: cement, clinker, industrial waste, raw material mixture, composition, firing, crystalline phases, properties.

1. INTRODUCTION

Cement is an indispensable silicate material for residential, transport, industrial and military construction [1]. However, cement production is associated with significant costs of natural carbonate and clay raw materials, the deposits of which are exhaustible and non-renewable—thereby failing to meet modern requirements for resource conservation and sustainable development.

The main component of Portland cement is clinker, the phase composition of which is formed during the firing of the raw material mixture, depends on its chemical and mineralogical composition, preparation method, high-temperature treatment regime and determines the quality indicators of the final product of production [2-4]. At the same time, the chemical technology of clinker production is characterized by high material consumption associated with the thermal dissociation of carbonates during firing of initial mixtures based on natural raw materials, and energy consumption associated with high thermal energy consumption during firing with a maximum temperature of over 1400 °C.

The exhaustion and irretrievability of natural mineral deposits, the increasing costs of extraction and value determine the feasibility of replacing them with waste from other industries as technogenic raw materials [5-7].

There are well-known studies and developments regarding the use of waste from other industrial sectors to manufacture construction materials [8-10], including cement [11, 12]. However, with respect to cement technology, these efforts are primarily focused on utilizing technogenic raw materials at the final stage of production. Yes, granulated blast-furnace slag is used as a partial substitute for clinker during the grinding process—much like thermal power plant fly ash is used as an additive to clinker in the production of composite cements. However, natural raw materials remain the primary components of the feed mixtures used to produce cement clinker, with industrial iron-bearing waste serving only as an additive in relatively small quantities (1–5 wt. %). The high material intensity of the initial mixtures makes it advisable to increase the use of industrial waste within them as substitutes for natural carbonate and clay raw materials.

Among such wastes, thermal power plant (TPP) fly ash [13,14] and metallurgical slags [15] are notable due to their accumulation volumes and chemical-mineralogical composition.

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Research and development regarding the use of TPP fly ash and ash-slag mixtures in construction and the production of building materials are well-established [16, 17], with the focus primarily on modifying the composition and properties of cement mortars and concretes [18–20]. Research in this area has seen significant development [21–23]. The feasibility of using fly ash as a component of the raw mix for cement clinker production has been noted [24, 25].

Researchers are devoting considerable attention to the use of slags from ferrous and non-ferrous metallurgy [26–28]. As previously noted, granulated blast-furnace slag is widely used in cement production technology. At the same time, the specific aspects of utilizing various types of metallurgical slags in cement technology are highlighted [29–30].

The aim of this study was to investigate the combined use of various types of thermal power plant (TPP) fly ash and ferroalloy slag as partial substitutes for natural raw materials in the initial mixture for cement clinker production.

Table 1. Particle-size distribution of technogenic raw materials

Samples	Particle fraction content (mm), mass %						
	> 2,5	2,5-1,6	1,6-0,4	0,4-0,2	0,2-0,1	0,1-0,05	<0,05
Fly ashZb	12,1	6,0	10,4	9,45	34,55	21,8	5,3
fly ashZk	-	-	0,15	2,2	12,6	47,3	37,4
slagZs	38,2	27,6	30,4	1,7	0,55	0,45	0,8

Raw material mixtures were prepared according to modern cement technology by proportioning components by mass, mixing and homogenizing them in a ball mill to a uniform degree of fineness, firing, and grinding the final product to a uniform residue on a 008 sieve.

Samples of raw mixtures were fired in a furnace for 15 hours at a maximum temperature of 1400 °C, keeping at a maximum of 1.5 hours. All mixture samples being compared were fired together to rule out any potential differences in the degree of heat treatment.

The methods of physico-chemical analysis of silicate raw materials and testing of binder properties used in this work included:

- analysis of chemical composition using standardized procedures. In this case, along with analytical chemistry methods, X-ray fluorescence analysis on an ElvaX Plus spectrometer was used;
- X-ray diffraction analysis (powder preparations) using a DRON-4-0 diffractometer connected via an interface to a computer;
- computer calculations of the compositions of the initial raw material mixtures based on the

Addressing these practical tasks requires determining the patterns governing the influence of the composition and concentration of these industrial by-products on phase formation and the properties of the cement clinker.

2. EXPERIMENTAL PART

Research objects and methods

The object of the study was raw material mixtures for the production of cement clinker using waste from the thermal power industry and metallurgy—thermal power plant (TPP) fly ash and metallurgical waste—specifically, ferroalloy production slag. For the study, fly ash samples were collected from two TPPs—differing in the types of coal used and the combustion technologies employed—along with a sample of ferrosilicomanganese slag.

Despite sharing the same physical state, the collected samples exhibit differences in particle size distribution (Table 1).

specified values of the binder's modular characteristics;

- testing of binder properties and physical and mechanical characteristics of the material in accordance with current standards. When testing mechanical properties, the arithmetic mean of three parallel determinations was taken as the final result; the relative error was 7.7% at a confidence level of $P = 0.95$.

Culations and analysis of the composition of raw material mixtures for the production of cement clinker were carried out using the created computer program "Clinker" [31]. The program is written in the C# programming language. It can run on any PC running the Windows operating system, version NT or later. The program enables the rapid determination of optimal component ratios in the initial raw material mixture based on specified values of the saturation coefficient (SF) and the silica modulus (n).

The principle of rapidly solving the problem on a computer using specialized software is as follows:

1. A table containing the chemical composition of an unlimited number of potential raw material components (≥ 4) is entered.

2. Values are specified for SF (for calculating a 2-component mixture), SF and n (for a 3-component mixture), or SF, n, and p (for a 4-component mixture).

3. Using the adopted calculation formulas, all combinations of two, three, or four components that yield the specified clinker characteristics are determined. Thus, given a sufficiently extensive raw material base, the optimal component ratios for the initial raw material mixture can be rapidly determined.

In this work, the following types of natural and man-made raw materials were used:

- chalk from the Zdolbunivka deposit;
- fly ash - waste from the Burshtynskaya TPP (sample Zb);

- fly ash - waste from the Kurakhivskaya TPP (sample Zk);
- slag - waste from the production of ferroalloys (sample Zsh).

Samples of the starting raw materials differ significantly in genesis and composition.

According to the chemical composition (Table 2), the Kurakhiv fly ash sample differs from the Burshtyn fly ash sample in a higher quantitative ratio of $\text{SiO}_2:\text{Al}_2\text{O}_3$ – 2.6 versus 2.2, which indicates lower refractory. The slag sample differs from the fly ash samples in a higher quantitative ratio of $\text{SiO}_2:\text{Al}_2\text{O}_3$ – 3.3, and an increased amount of calcium and manganese oxides. The chalk sample is characterized by a predominant content of CaO (55.0 wt. %),

Table 2. Chemical composition of raw material

Samples	Content of oxides, wt. %											
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	MnO	SO_3	Na ₂ O	K ₂ O	SrO	P ₂ O ₅
Zb	53,92	24,66	13,13	1,31	1,14	0,92	0,12	0,52	0,24	3,35	0,07	0,37
Zk	56,71	21,77	11,58	1,22	2,01	0,76	0,06	1,81	0,20	3,51	0,04	0,13
Zsh	46,85	8,84	3,80	0,36	11,46	2,57	17,13	3,30	0,46	2,86	1,46	--

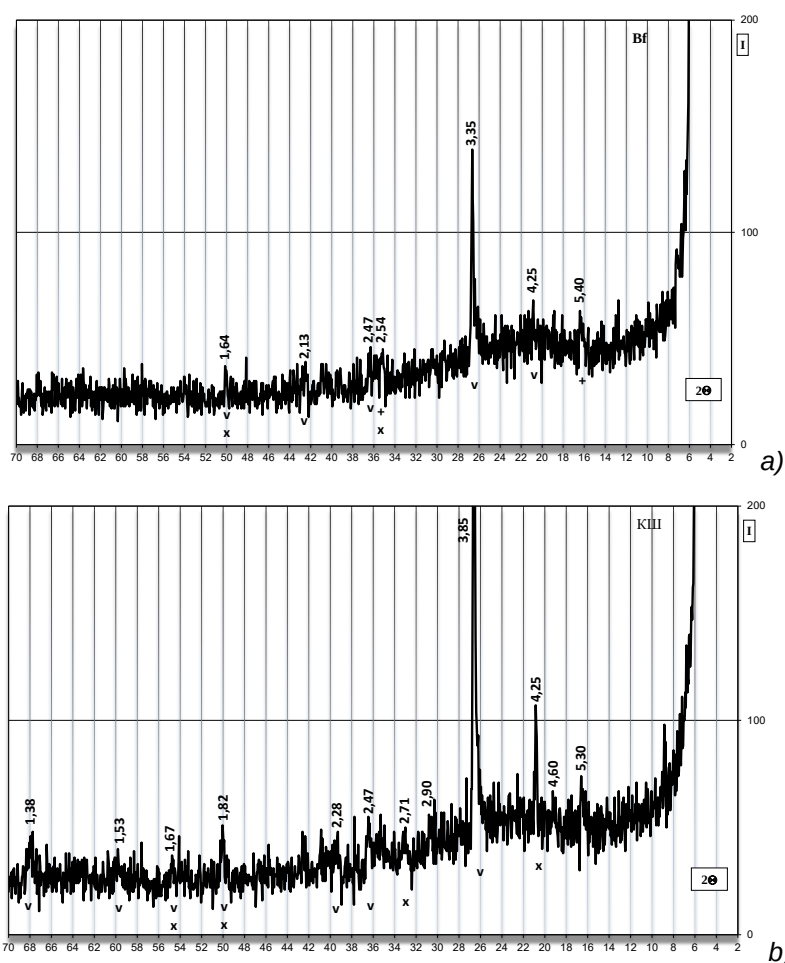


Figure 1. X-ray diffraction of fly ash Zb (a) and Zk (b): v-quartz, +mullite, x-calcite

According to the mineralogical composition, chalk is characterized by a predominant content of calcite, TPP fly ash and slag are characterized by the development of a glass phase and varieties of crystalline phases (Fig. 1, 2).

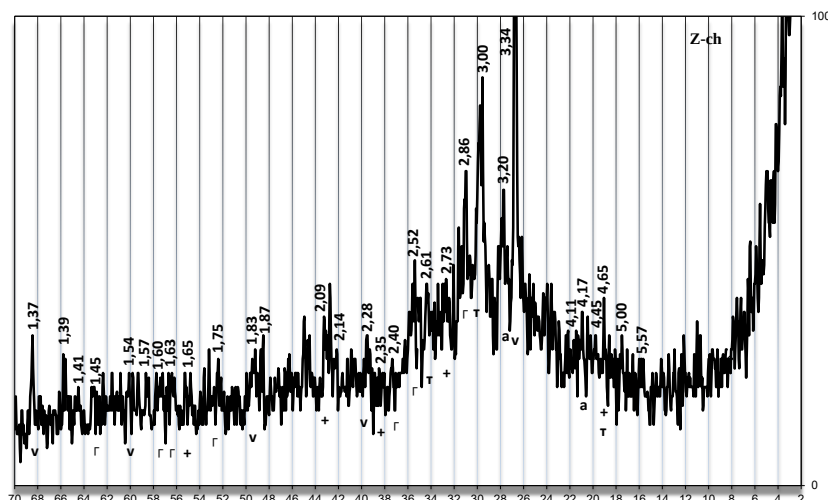


Figure 2. X-ray diffraction of slag Zsh: V quartz, + brownit, e hematite, a and anorthite, m belite

Calculations and analysis of the composition of raw material mixtures for the production of cement clinker were carried out using the created computer program "Clinker" [21]. This allowed us to quickly determine the rational ratios of components in the initial raw material mixture based on the given values of the saturation coefficient SF and the silica n modulus.

Analysis of the composition of raw material mixtures

Analysis of the obtained results of computer calculations showed that in binary raw material mixtures for the production of cement clinker in the interval of the specified values of the saturation coefficient $SF=0.80-0.95$, the content of fly ash from thermal power plants can be from 23-27 wt. %, the content of slag - from 23-26 wt. %. However, the indicators of the silica and alumina modules of clinker from binary mixtures do not correspond to the recommended ones (Table 3).

Table 3. Characteristics of clinker from binary mixtures at $SF=0.80-0.95$

Modular characteristics	Recommended value	Indicators for different types of waste		
		chalk -Zb	chalk -Zk	chalk -Zsh
n	1,9-3,0	1,17	1,71	3,60
p	0,9-2,0	0,83	1,17	2,30

According to the results of computer calculations, at the recommended value of the

saturation coefficient $SF = 0.90$ in 3-component mixtures based on the chalk - fly ash - slag system, the possible content of technogenic raw materials is 24 wt. % in the range of values of the silica modulus $n = 2.0-3.0$ and the alumina modulus $p = 1.2-1.8$ (Fig. 3). At the same time, significant differences are determined in the quantitative ratio of slag and types of fly ash Csh / Cz, which is from 1.3 to 7.5 for Zb ash and from 0.3 to 4.0 for Zk ash (Fig. 4).

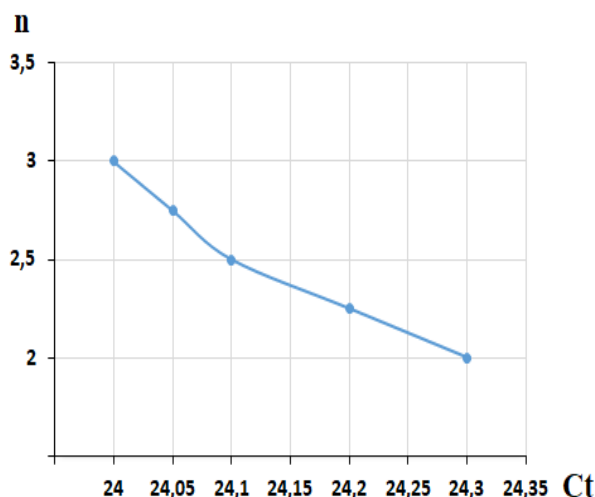


Figure 3. Dependence of silica modulus (n) on the content of technogenic raw materials (Ct) in the chalk-fly ash-slag mixture at $SF=0.90$

Based on the analysis of the calculation results, 3-component mixtures were selected for further

research, characterized by the use of compositions of TPP fly ash and ferrosilicomanganese slag as the aluminosilicate and ferro-containing component (Table 4). Unlike known compositions, however, natural clay is not used.

Table 4. Composition of raw mixtures

Code of mixture	Quantity of components, mass %			
	chalk	Fly ash		Slag Zsh
		Zb	Zk	
11D	75,9	6,1	-	18,0
09D	76,0	-	10,3	13,7

Thus, through the use of the waste composition, the consumption of natural raw materials is reduced by 24 wt.%—or 432,000 tonnes—given a total raw material consumption of 1.8 million tonnes for the production of 1 tonne of cement clinker.

With the specified material composition and the same chalk content, the differences in the chemical composition of the mixtures are determined by the compositions of technogenic raw materials (Table

5). At the same time, the composition of amber fly ash with slag, with a slightly lower content of silicon, aluminum and iron oxides, differs in a larger amount of calcium and manganese oxides.

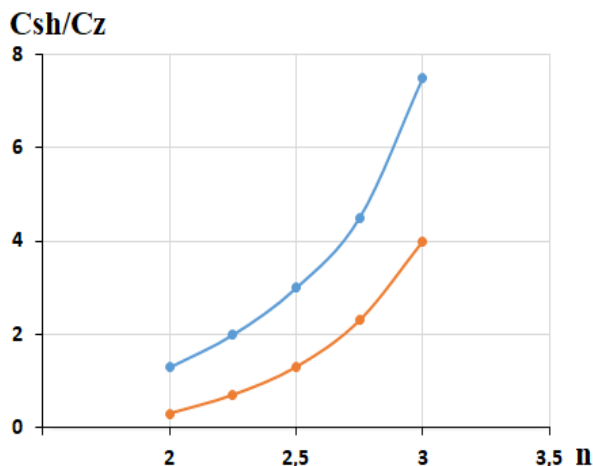
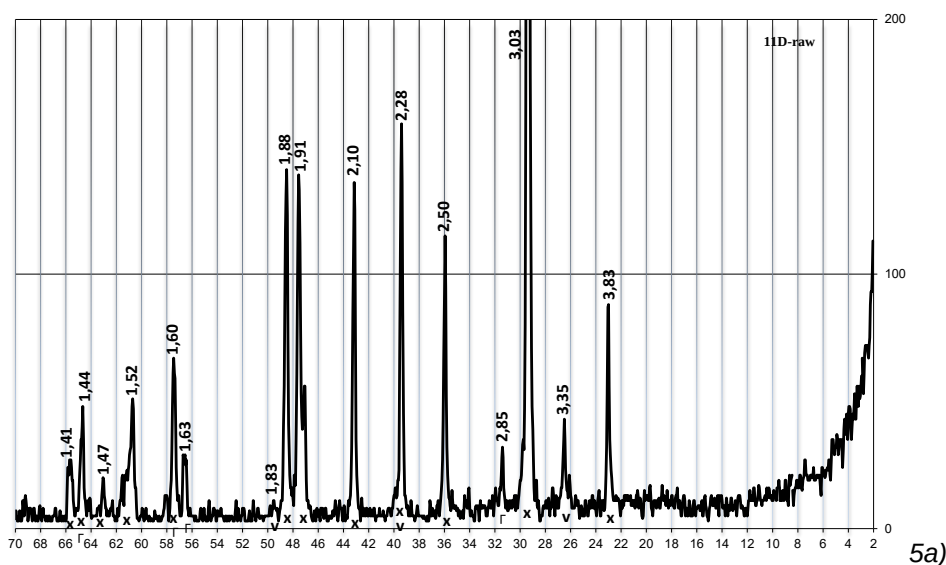


Figure 4. Dependence of the quantitative ratio (C_{sh}/C_z) of slag with Zb (a) and Zk (b) fly ash on the silica module (n) in a mixture with chalk at $SF=0.90$

Table 5. Chemical composition of waste compositions

Sample code	Content of oxides, wt. %									
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	MnO	SO ₃	Na ₂ O	K ₂ O
Zb+Zsh	11,72	3,09	1,48	0,14	2,13	0,52	3,09	0,92	0,09	0,71
Zk+Zsh	12,26	3,44	1,71	0,18	1,78	0,43	2,36	0,64	0,08	0,75

The mineralogical composition of the studied mixtures corresponds to the physical and chemical (Fig. 5). At the same time, mixture 09D differs from 11D in a larger amount of crystalline quartz.



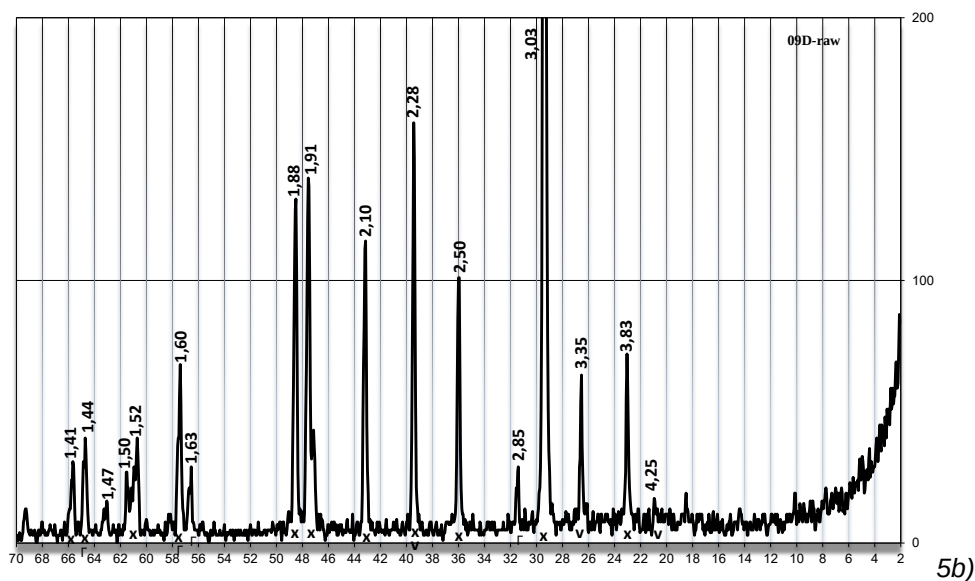


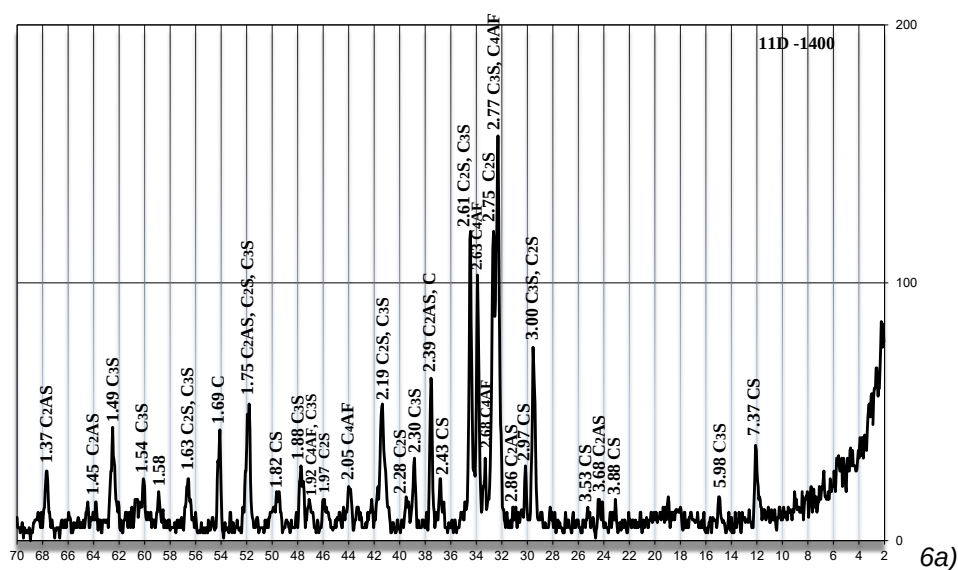
Figure 5. X-ray diffraction of raw material mixtures 11D (a) and 09D (b): x calcite, v quartz, + kaolinite, • hydromica, e hematite

Phase composition and properties of the binder material

After firing at a maximum temperature of 1400 °C, differences in the chemical composition and, accordingly, the modulus characteristics of the binder are determined according to X-ray fluorescence analysis (Table 6). In this case, the values of the silica modulus n are from 2.0 (09D) to 2.2 (11D), and the values of the alumina modulus $p = 2.1$ –2.2, which correspond to those recommended for cement.

Table 6. Chemical composition of the material after firing (1400 °C)

Sample code	Content of oxides, wt. %								
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	SO ₃	K ₂ O	P ₂ O ₅
11D	26,48	8,20	3,77	0,30	49,70	1,49	1,74	0,62	0,17
09D	27,80	9,59	4,48	0,34	48,14	1,30	1,65	0,86	0,16



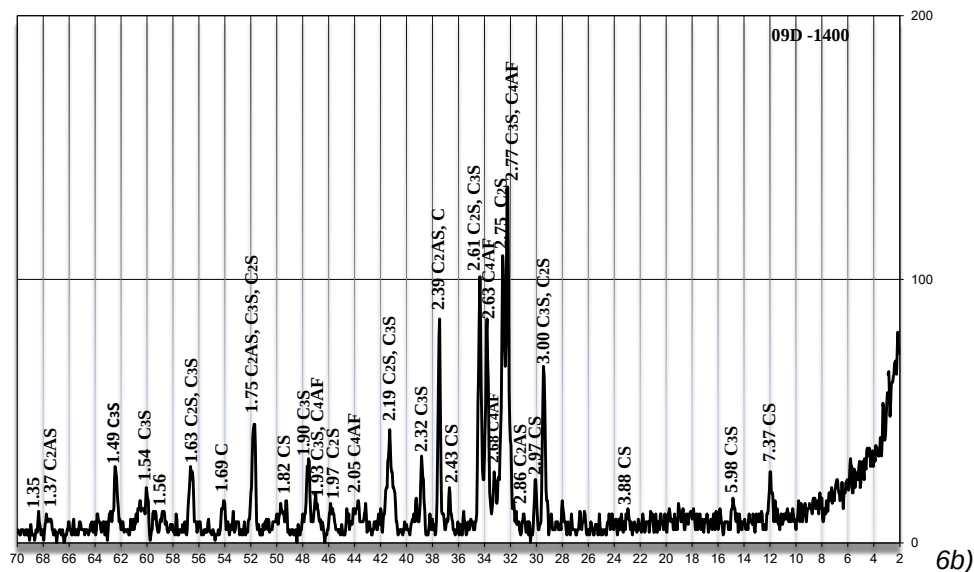


Figure 6. X-ray diffraction of samples 11D (a) and 09D (b) after firing (1400 °C)

The obtained results of X-ray phase analysis allowed us to identify certain features of the crystallization structure of the material after firing the studied mixtures with a maximum temperature of 1400 °C (Fig. 6). It was established that when using the composition of amber fly ash with slag in the initial mixture at a quantitative ratio of 1 : 3, sample 11D differs from 09D in a greater development of crystalline phases:

- calcium silicates C_3S (3.00, 2.77, 2.61 Å), C_2S (2.75, 2.61, 2.19 Å), CS (2.97 Å);
- calcium aluminoferrite - C_4AF (2.77, 2.68, 2.63 Å).

The observed features of the material's crystallization structure following the co-firing of samples—that is, under identical high-temperature treatment conditions—are evidently linked to the previously noted differences in the material composition of the initial mixtures (Table 3), the chemical composition of the fly ash and slag blends (Table 4), and the phase composition of the slag (Fig. 2). According to established concepts [32, 33], clinker minerals are the product of the combination of calcium, silicon, aluminum, and iron oxides during the firing process—a result primarily of the breakdown of the crystal lattices of the raw mix's rock-forming minerals during heat treatment. In the silicate systems under study, the primary source of CaO is a carbonate rock—chalk—while fly ash and slag serve as the main sources of SiO_2 , Al_2O_3 , and Fe_2O_3 . At the same time, the composition of the technogenic components $Zb+Zs$ (sample 11D) differs from $Zk+Zs$ (sample 09D) by having a lower Al_2O_3 content and a higher $SiO_2:Al_2O_3$ ratio, indicating lower refractoriness; furthermore, the thermal decomposition of the slag's rock-forming

mineral lattice—brucite ($Mn^{2+}(Mn^{3+})_6SiO_{12}$)—leads to the formation of manganese oxides, which act as mineralizers to intensify the sintering process. Naturally, this effect is more pronounced in sample 11D due to its higher slag content.

Table 7. Properties of mineral asringent material

Characteristics		Sample code	
		11D	09D
Fineness of grinding, sieve residue no. 008, mass. %		10	10
Consistency, %		41	40
Setting time, min	initial time	55	35
	final time	105	95
Compressive strength, MPa			
2 days		3,9	4,0
7 days		23,1	21,8
28 days		42,6	39,3

Technological testing showed differences in the properties of the obtained materials (Table 7). According to the classification of DSTU B V.27-91-99 [22], according to the hardening rate after firing at a maximum temperature of 1400 °C, sample 11D belongs to the group of normal-hardening (starting time from 45 min. to 2 h), the typical representatives of which are Portland cement, slag Portland cement, pozzolanic cement. Sample 09D belongs to the group of fast-setting (starting time from 15 to 45 minutes), typical representatives of which are anhydrite and alumina cement.

3. DISCUSSION

The research findings regarding the compositional characteristics of various types of technogenic raw materials and the influence of

their blends on the phase composition and properties of cement clinker are consistent with the principles of modern materials science concerning the relationship between material composition, structure, and properties.

Unlike known studies regarding the use of industrial waste at the final stage of cement production—either during clinker grinding or as an iron-bearing corrective additive (1–5 wt.%) in the raw mix for clinker production—this work addresses the comprehensive use of thermal power and metallurgical wastes as substitutes for natural raw materials in the initial raw mix.

The solutions developed for new raw material mix compositions—utilizing a blend of 24 wt.% thermal power plant fly ash and ferroalloy slag for cement clinker production—enable the replacement of natural clay with industrial waste materials, thereby addressing the integrated challenges of resource conservation and the chemical technology of mineral binders.

At the same time, the practical application of the research results requires addressing organizational issues regarding the optimization of logistics for supplying various types of waste to cement manufacturing companies.

Nevertheless, future research could focus on utilizing other industrial waste compositions in clinker production mixtures to replace natural raw materials, the deposits of which are finite and non-renewable.

4. CONCLUSIONS

The study establishes the specific characteristics of using compositions of TPP fly ash and ferrosilicomanganese slag—serving as aluminosilicate and iron-bearing components in raw material mixtures for cement clinker production—as a means of regulating the properties of the mineral binder and promoting resource conservation.

Based on computer calculations, raw mix compositions for the production of cement clinker without the use of natural clay have been determined, incorporating up to 24 wt.% of industrial waste as technogenic raw material.

The feasibility of producing normal-setting and rapid-setting medium-strength cement from the investigated mixtures at a maximum clinker firing temperature of 1400°C (compared to 1450°C at existing plants) has been experimentally confirmed.

The features of the chemical and phase composition of clinker, which are manifested in the formation of crystalline phases of silicates and calcium aluminoferrite as a factor influencing the binding properties and strength of the resulting material, have been established.

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IZVOD

ASPEKTI PROIZVODNJE CEMENTNOG KLINKERA KORIŠĆENJEM RAZLIČITIH VRSTA INDUSTRIJSKOG OTPADA

Proučavani su silikatni sistemi sa tehnogenim sirovinama kao polaznim smešama za proizvodnju cementnog klinkera. Prikazane su karakteristike hemijskog i mineraloškog sastava velikotonačnog otpada iz termoelektrične industrije – pepela i metalurgije – zguze iz proizvodnje ferolegura. Korišćenjem računarskog programa „Klinker“ izvršeni su proračuni i analiza mogućeg sadržaja proučavanog otpada u početnoj smeši sirovina na osnovu zadatih karakteristika klinkera. Utvrđeni su novi sastavi smeša sirovina na bazi sistema kreda-pepeo-zgura sa ukupnim sadržajem od 24 tež.% zadatog otpada. Eksperimentalno je potvrđena mogućnost dobijanja normalno i brzo očvršćavajućeg cementa srednje čvrstoće iz takvih smeša. Istovremeno, prikazana je veza između svojstava cementa i karakteristika sastava klinkera u pogledu formiranja kristalnih faza silikata i aluminoforitukalcijuma.

Ključne reči: Cement, klinker, industrijski otpad, mešavina sirovina, sastav, pečenje, kristalne faze, svojstva.

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