

SULFATE-RESISTANT CEMENTS BASED ON TECHNOGENIC WASTE FROM NON-FERROUS METALLURGY

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Abstract - Due to the possibility of producing new construction materials with increased resistance to aggressive environments based on secondary raw materials and industrial wastes, there is a growing need for systematic research and their use in the production of Portland cement. In this regard, the tailings from the beneficiation of ferrous and non-ferrous metal ores are considered promising in terms of their content and reserves of valuable components compared to natural deposits and industrial dumps. At the concentrating plants of the Almalyk Mining and Metallurgical Combine (AMMC), tens of millions of tons of tailings rich in Al_2O_3 , Fe_2O_3 , and other oxides are annually discarded, which are valuable components for cement production, formed during the beneficiation of lead- and copper-containing ores. This article presents the results of research on the topical issue of utilizing tailings from lead-enrichment and copper-smelting plants of the Almalyk Mining and Metallurgical Combine in the production of sulfate-resistant Portland cement as an active mineral additive. The influence of the mineralogical compositions of clinkers from cement plants in Uzbekistan on the phase composition and properties of the obtained sulfate-resistant cement in the presence of lead-copper active mineral additives is analyzed. It is shown that the additives play an important role in strength development and the formation of the cement stone structure. Acceleration of the hydration and hardening processes of sulfate-resistant cement is observed due to changes in the mineralogical composition, namely the reduction of tricalcium aluminate content and the decrease in clinker basicity resulting from the introduction of high-silica wastes, which meets the requirements for sulfate-resistant cements.

Keywords: Sulfate-resistant cement, Technogenic waste, Mechanical strength, Hydration activity, Sulfate resistance, Hydration, Bound water.

1. Introduction

At present, scientific research at the global level is focused on developing ingredients and compositions that improve the construction and technical properties of cement, creating highly efficient energy- and resource-saving technologies, and enhancing their performance characteristics. Existing scientific foundations for obtaining high-performance sulfate-resistant cements using secondary resources from various industrial sectors have been further developed. The influence of the mineralogical composition of clinker on the phase composition and properties of cement products in the presence of technogenic additives is analyzed.

The effect of the mineralogical composition of Portland cement clinker on the corrosion resistance of cement stone in aggressive environments has been investigated by many researchers [1-10]. The high mineralization of surface and groundwater in several regions of our country has always necessitated the use of corrosion-resistant Portland cements for the construction of hydraulic and irrigation structures [1]. Particularly high mineralization of groundwater is observed in the Aral Sea region, where construction structures are exposed to combined sulfate-magnesium-chloride aggression.

Previously, the Laboratory of Silicate Chemistry at the Institute of Chemistry of the Academy of Sciences of the Republic of Uzbekistan, under the

supervision of Doctor of Chemical Sciences I.S. Kantsepolsky, developed sulfate-resistant Portland cements using local raw material resources [2].

In the study by L.I. Sychev and Bakeev [9], a sulfate-containing cement was obtained from low-aluminate and technogenic raw materials and was identified as a type of sulfoaluminate cement. Moreover, many researchers investigated sulfate-resistant (SR) cement, focusing on chemical modifications, the use of industrial waste, and energy-saving production techniques. Among them, Jing Dai et al. investigated the mechanism of Nano-Silica (NS) in High Sulfate Resistance Portland Cement (HSRPC) blended with Mineral Powder (MP) or Fly Ash (FA). Small doses of NS (0.1–0.5%) improved 3d and 28d compressive strength. NS reacts with Calcium Hydroxide (CH), filling “harmless pores” and densifying the matrix. For MP-based mixes, higher NS content actually increased cumulative porosity by 9.8% and decreased total hydration heat, suggesting that excessive NS can inhibit hydration in specific mineral powder systems due to its high specific surface area [3]. Amir Mohammad evaluated the effectiveness of slag in preventing traditional sulfate attack (23 °C) and the more destructive Thaumasite Sulfate Attack (TSA) at low temperatures (5 °C) by replacing 30–50% of high C_3A cement with slag successfully rendered all mixes sulfate-resistant. It was revealed that 30% slag replacement was insufficient for Portland-limestone cements at low temperatures, indicating a high risk of structural failure in cold-climate sulfate environments if replacement levels are too low [4]. Moreover, Islem Labidi conducted study focused on the “burnability” and chemical optimization of SR cement using natural Tunisian raw materials (limestone, marl, and fluorapatite). The results showed that Traditional SR cement is hard to make because low C_3A (<3%) requires extremely high kiln temperatures. Integrating 0–15% natural fluorapatite lowered the required energy by maintaining a stable liquid phase at lower temperatures [5]. In our previous research, we explored the using lead and copper concentration tailings from the Almalyk Mining and Metallurgical Complex (AMMC) in Uzbekistan as active mineral additives for SR cement. She reported that Tailings rich in Al_2O_3 and Fe_2O_3 act as active modifiers. They accelerate hydration and hardening while reducing the overall basicity of the clinker. However, the effectiveness of these tailings is highly dependent on the specific mineralogical composition of the base clinker, suggesting that a “one-size-fits-all” waste additive may not work across different cement plants [6].

As a result of the conducted literature review and research, we have developed the physicochemical basis of a new progressive energy-saving technology for the production of high-efficiency sulfate-resistant cements using metallurgical waste. The features of the formation of hydration phases in silicate material, such as low-basic calcium hydrosilicates, tobermorite, hydrocarbon silicates, as well as gel-like C–S–H structures, have been studied. The regularities of the reactions occurring when introducing mineral additives MOF and SOF into the composition of Portland cements have been established. Amorphous silica separates the released $Ca(OH)_2$ and other impurities contained in the additives, fixing them on the surface of silica particles. As a result, the activating ability of the additives increases, and consequently, due to the accelerated removal of Ca^{2+} ions from the liquid phase, the formation of hydrated minerals and the development of the cement stone structure are intensified [7–10].

2. Research Methodology

The objects of the study were the wastes from the beneficiation of lead- and copper-containing ores of AGMK and clinkers from cement plants. Modern methods of analysis of raw material mixtures and reaction products were used for the research. During the research, chemical and gravimetric methods of analysis were used, as well as the method of determining the ultimate strength in bending and compression, the fineness of grinding was determined by the sieve method, the setting time was determined using a Vicat device, chemical resistance was studied using the method of V.V. Kind and other standard methods of analysis. The chemical composition of raw materials was determined according to GOST 5382–91. The dependence of cement properties on individual factors was studied in accordance with GOST 310–89. As aggressive environments, the most common and practically significant 3% $MgSO_4$ solution and 5% Na_2SO_4 solution were used, corresponding to subsoil and groundwater. The samples were tested after 3, 7, 14, 28, 90, 180, and 360 days. The evaluation of sulfate resistance of cements was carried out according to the Resistance coefficient, which after 6 months remains not less than 0.8. The hydraulic activity of SOF and MOF wastes was determined by the standard method—measuring the amount of lime absorbed from a saturated solution per gram of additive. The additives were studied using physicochemical methods of analysis: determination of qualitative and quantitative composition—X-ray phase analysis (XRD) (diffractometer ARL X'TRA), processing of diffractometric data was performed automatically using the computer program PDWin 4.0; X-ray fluorescence analysis (VRA-30, Carl Zeiss Jena).

3. Research Results and Discussion

Sulfate-resistant cement is obtained on the basis of clinker, the composition of which is characterized by low values of KN and alumina modulus with a relatively high silicate modulus. They are produced brands 400, 500. It is possible to introduce plasticizing and hydrophobic additives that increase the frost resistance of concrete and its total density. This cement shall contain C_3S not > 50%, KH - 0.83-0.87; S_3A not > 5%. The increase in KH causes a decrease in sulfate resistance, it is compensated by the introduction of hydraulic additives (10-15%). Plants of Uzbekistan produce three varieties of sulfate-resistant cements: sulfate-resistant Portland cement, sulfate-resistant Portland cement with the addition of electrothermophosphorus slags, sulfate-resistant Portland cement with the addition of clay. Gliège and electrothermophosphorus slags are used as additives in the Republic of Uzbekistan. Currently, the reserves of Angren gliés are running out, electrothermophosphorus slag is imported from Kazakhstan. In Uzbekistan, there are practically no effective additives to cement, at the same time there are potential raw materials on the basis of which, it

is possible to organize the production of effective additives [11].

In the practice of using cements in construction, the initial strength of cement plays an important role. The main disadvantage of Portland cement with pozzolanic additives is that its initial strength is reduced compared to Portland cement. The initial strength of Portland cement depends on the mineralogical composition of Portland cement clinker, the amount of additive added and on the physical structure of the additives, dispersion and uniformity of distribution in the powder [12-14]. In this regard, it would be promising to use the flotation waste of SOF and MOF of the Almalyk Mining and Metallurgical Plant as hardening activators and an active mineral additive to Portland cement of different mineralogical composition.

For the study, additive-free Portland cements CEM 0 of strength class 32.5H according to GOST 31108-2020 were used, produced by "Akhangarancement" LLC, "Bekabadcement" LLC, and "Kuvasaycement", as well as tailings from lead beneficiation and flotation waste from copper beneficiation plants, the chemical composition of which is given in Table 1.

Table 1. Chemical composition of initial materials, wt. %

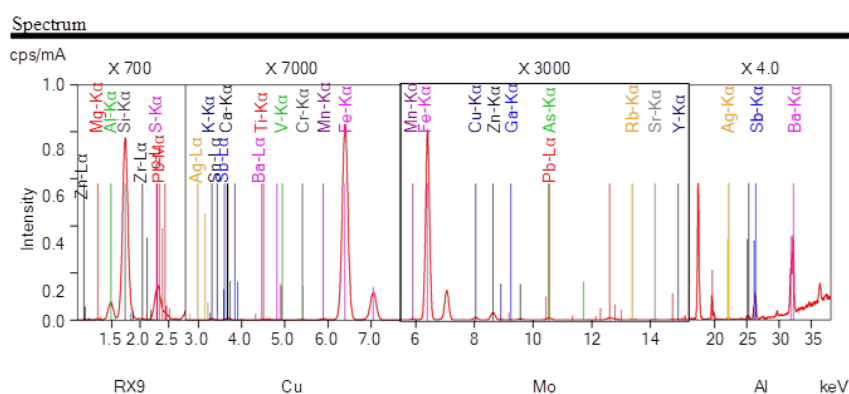
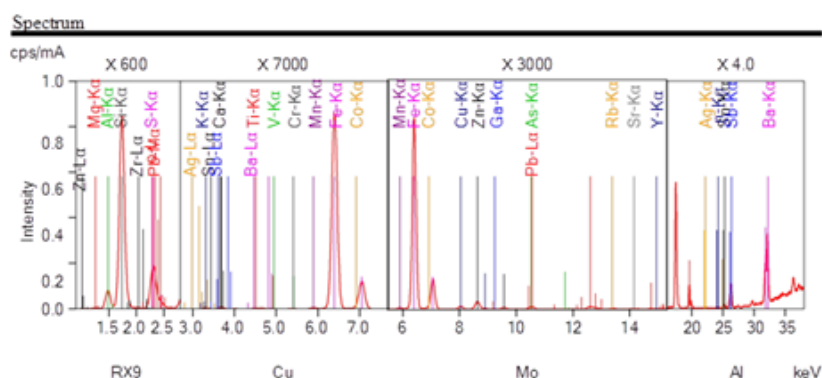
Initial materials	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	CaO _f	R ₂ O	loss. mas.	Other	Σ
Akhangaran Portland cement	22.68	4.55	3.65	65.48	2.45	0.30	0.12	–	–	0.87	100
Bekabad Portland cement	20.63	4.52	4.05	65.92	1.73	2.15	0.43	–	–	0.70	100
Kuvasay Portland cement	21.78	4.81	4.08	64.14	1.89	0.64	0.37	–	–	0.60	98.31
SOF tailings	45.75	8.72	7.19	14.59	7.10	2.98	–	2.98	8.83	–	98.14
MOF flotation waste	60.68	14.02	9.54	1.37	0.11	5.69	–	4.13	4.11	–	99.65

According to GOST 24211-2003, additives for cements, concretes, and mortars are various products introduced into cement and concrete mortar mixtures to improve their technological properties, enhance the construction and technological characteristics of concrete and mortars, and impart new properties [4, 15-17]. Additives are chemical substances of both organic and inorganic structure, of complex or simple composition. The need to search for new additives is determined by the selective nature of their modifying effect, which depends not only on the chemical composition of the additives but also on the chemical and mineralogical composition of the cement, the fineness of its grinding, etc. The magnitude of the modifying effect of many additives

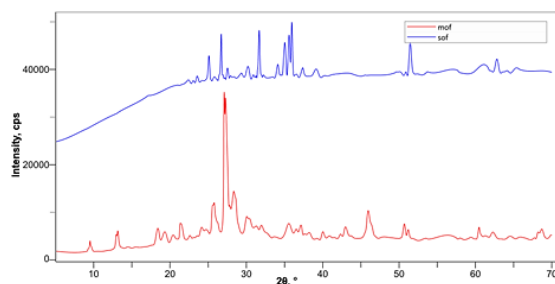
also depends on the specific consumption of cement in the concrete mix, the content and type of mineral additives, and the water-cement ratio. At the same time, the process of formation of the solid-phase state of the cement system should be taken into account [18-21].

To identify the possibility of using technogenic waste in the production of sulfate-resistant cements, their properties were studied using modern physicochemical methods of analysis.

The wavelength-dispersive X-ray fluorescence spectrometric elemental analysis of SOF and MOF (Fig. 1, Fig. 2) showed an insignificant difference in the content of the main oxides contained in MOF and SOF (Table 1).



According to X-ray phase analysis data, the additives studied are X-ray amorphous. At the same time, on the X-ray diffraction pattern of the SOF flotation waste (Fig. 3), interplanar spacings corresponding to quartz are recorded at $d = 0.334, 0.246, 0.181, 0.153$ nm; to CaCO_3 at $d = 0.303, 0.191, 0.187$ nm; to feldspar at $d = 0.320, 0.252, 0.212$ nm; and to hydromica at $d = 0.334, 0.256, 0.190$ nm. For more accurate mineral identification, the most intense and characteristic lines for each mineral corresponding to the obtained interplanar distances are provided.



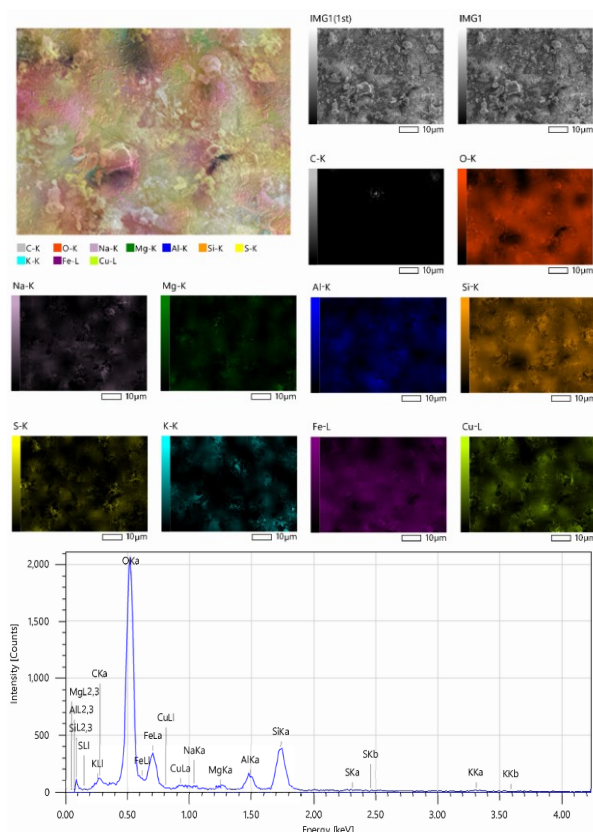


Figure 5: Electron microscopic images of cement samples with SOF additive

According to the X-ray phase and electron microscopic data, the additives are multiphase materials containing the main phases in the form of quartz, feldspars, hydromicas, plagioclases, gypsum, and limestone.

The processes of agglomeration were studied by the electron microscopic method: the smallest particle size is about 5 μm , followed by the formation of large agglomerates up to 10 μm . Further growth of the particles was observed. For SOF, tabular or prismatic crystals of feldspars are typical. Iron compounds are present in the form of magnetite, as well as yellow-brown aggregates of needle-like or plate-shaped crystals. At a temperature of 590–650°C, a transformation into hematite is observed. Crystals of anhydrite are found, having a blue, violet, pale yellow, or pinkish color. Alumina hydrates appear in the form of basalt-like scales, while kaolinite crystals have hexagonal scaly shapes. In metallurgical slags, plagioclases are also found as lamellar crystals with grayish, yellowish, or greenish shades.

For MOF, the presence of magnetite and hydromica compounds is also typical. Hydromicas are hydroaluminates containing iron, magnesium, alkalis, and other elements. Their thermograms are close to those of kaolinite. The crystals have a lamellar structure [22–23].

Using X-ray phase analysis, the phase compositions of the matrix material for obtaining cement mortars based on clinkers from Akhangaran, Bekabad, and Kuvasay cement plants and additives SOF and MOF were identified. The clinkers of the cement plants are mainly represented by:

C_3S – $d = 0.303; 0.296; 0.260; 0.218; 0.192; 0.176$ nm; C_2S – $d = 0.385; 0.277; 0.272; 0.260; 0.208$ nm; C_3A – $d = 0.273; 0.269; 0.216; 0.202; 0.192; 0.154$ nm; C_4AF – $d = 0.269; 0.264; 0.218; 0.204; 0.192; 0.182$ nm.

Table 2. Strength of sulfate-resistant cements with the addition of metallurgical wastes MOF and SOF

№	Additive Introduced	Aggressive Medium	Compressive Strength of Samples, MPa (Days)						
			3	7	14	28	90	180	360
			Akhangaran Portland Cement						
1	MOF	3% MgSO ₄	30	38	40	46	66	80	94
2	SOF	"	38	42	48	59	71	87	103
3	–	"	14	19	20	29	33	36	50
4	MOF	5% Na ₂ SO ₄	32	38	41	50	73	75	80
5	SOF	"	38	41	47	58	64	77	89
6	–	"	15	19	25	33	39	38	38
			Kuvasay Portland Cement						
1	MOF	3% MgSO ₄	38	42	43	49	77	82	96
2	SOF	"	30	48	75	87	88	90	90
3	–	"	24	26	27	30	33	35	37
4	MOF	5% Na ₂ SO ₄	35	38	40	47	75	81	97
5	SOF	"	30	40	60	74	75	91	95
6	–	"	18	18	20	23	25	37	45
			Bekabad Portland Cement						
1	MOF	3% MgSO ₄	43	48	54	63	79	98	106
2	SOF	"	33	36	40	54	78	96	102
3	–	"	33	41	50	53	50	51	48
4	MOF	5% Na ₂ SO ₄	41	50	54	62	85	106	110
5	SOF	"	35	40	48	66	84	102	109
6	–	"	26	28	35	41	45	46	47

The obtained data are consistent with the results of determining the Resistance coefficients of the samples in aggressive environments depending on the curing time. The dependence of the Resistance coefficient (RC) of samples in aggressive media on curing time is shown in Figures 6 and 7.

Based on the above data, it can be concluded that the addition of SOF and MOF significantly increases the sulfate resistance of Portland cements with different mineralogical compositions.

The increase in sulfate resistance of cements due to the introduction of SOF and MOF additives can be explained by changes in the structure of the cement stone [24-25].

The introduction of SOF and MOF additives leads to a reduction in the proportion of C_3A in the cement and to the binding of calcium hydroxide released during hydration, which results in a significant increase in the resistance of cements to sulfate corrosion [26-29].

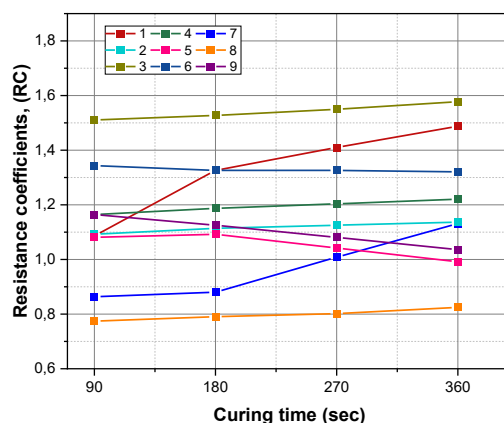


Figure 6: Resistance coefficients of samples of Akhangaran, Kuvasay, and Bekabad Portland cements in a 3% $MgSO_4$ solution. 1 – Akhangaran PC with MOF additive; 2 – Akhangaran PC with SOF additive; 3 – Akhangaran PC without additive; 4 – Kuvasay PC with MOF additive; 5 – Kuvasay PC with SOF additive; 6 – Kuvasay PC without additive; 7 – Bekabad PC with MOF additive; 8 – Bekabad PC with SOF additive; 9 – Bekabad PC without additive.

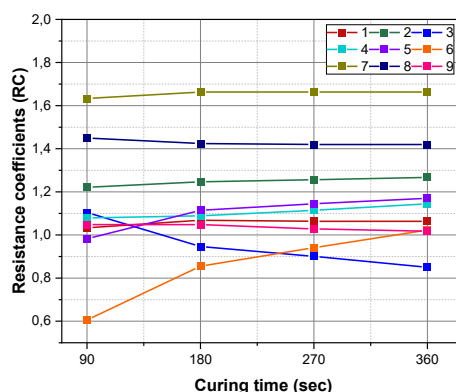


Figure 7: Resistance coefficients of samples of Akhangaran, Kuvasay, and Bekabad Portland cements in a 5% Na_2SO_4 solution. 1 – Akhangaran PC with MOF additive; 2 – Akhangaran PC with SOF additive; 3 – Akhangaran PC without additive; 4 – Kuvasay PC with MOF additive; 5 – Kuvasay PC with SOF additive; 6 – Kuvasay PC without additive; 7 – Bekabad PC with MOF additive; 8 – Bekabad PC with SOF additive; 9 – Bekabad PC without additive.

The improvement in salt resistance is associated with changes in the mineralogical composition of cements toward a decrease in C_3A content and a reduction in clinker basicity. The introduction of MOF and SOF additives reduces the C_3A content from 9–10% to 2–3% and the cement basicity to 0.84–0.86, which meets the requirements for sulfate-resistant cements.

It is well known that the interaction of cement with water begins almost instantly, during which hydration particles gradually form on the surface of the cement grains.

Water penetrates inward, exposing new surfaces of unhydrated cement particles, which leads to accelerated dissolution of minerals and crystallization of new formations. As long as the exposure of unhydrated surfaces prevails over the crystallization of new formations, a decrease in the strength of the cement stone is not observed.

Over time, within the setting period, the number of new formations increases, while the exposure of new unhydrated surfaces becomes minimal, resulting in continuous strength growth without reduction.

4. Conclusions

A comprehensive study of lead and copper tailings was carried out, and the results indicate the consistency of the content of main components in them and the possibility of using tailings as mineral additives in cement production. The physicomaterial properties of the studied cements fully meet GOST requirements, and the optimal amount of additive introduced is 5–10%. Based on the experiments conducted, the important role of the mineralogical composition and the activity of the matrix Portland cement clinker in obtaining cements with SOF and MOF additives is confirmed. The obtained results indicate that for the production of cements with SOF and MOF additives, it is advisable to use alumina-rich clinkers as the matrix clinker. A comparative assessment of the $\text{Ca}(\text{OH})_2$ content in the composition of cement stone containing SOF and MOF allows the conclusion that the introduction of 5% SOF and 3% MOF favorably affects the process of binding $\text{Ca}(\text{OH})_2$ into calcium hydrosilicates and hydroaluminates, as well as into carbonate-containing hydrated products.

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