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The effect of burnt moulding waste on the hydration and structure formation processes of portland cement

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Abstract: This paper presents the results of a comprehensive study of the effect of a filler based on burnt moulding waste (BMW) and a superplasticiser on the phase composition and structure formation of cement stone at early (1 day) and late (28 days) stages of hardening. X-ray phase analysis (XPA) has established that BFA has a powerful accelerating effect on the hydration of clinker minerals in the first day, which is explained by the adsorption of calcium ions on their surface, while the superplasticiser, on the contrary, slows down this process. A synergistic effect of the combined use of additives has been demonstrated, which negates the retarding effect of the superplasticiser and leads to the intensive formation of hydrate phases at an early stage. By day 28, a significant decrease in the content of portlandite ($Ca(OH)_2$) and an increase in the proportion of amorphous calcium hydrosilicate (C-S-H) by 22% are observed in the modified cement stone, which indicates the occurrence of an active pozzolanic reaction. The data obtained confirm that complex modification allows effective control of the hydration kinetics and the formation of a dense, highly structured cement stone matrix, which is a key factor in improving its strength and performance characteristics.

Keywords: X-ray phase analysis, structure formation, cement stone, burnt moulding waste, superplasticiser, hydration kinetics, pozzolanic reaction, synergistic effect

1. Introduction

The problem of obtaining building materials with improved performance characteristics while reducing their cost and environmental impact is one of the most pressing issues in modern materials science. Traditional cement systems have a number of limitations related to their high water demand and microstructural heterogeneity. In this regard, the search for effective modifiers and fillers capable of optimising structure formation processes and improving the quality of cement stone is becoming particularly important [1-4].

The introduction of superplasticisers (SP), such as polycarboxylates, into the cement mixture significantly reduces the water-cement ratio while ensuring high mobility of the mixture. This effect is due to the adsorption of SP molecules on the surface of cement particles, which creates steric and/or electrostatic repulsive forces that prevent their flocculation. As a result, a denser and more homogeneous structure of the cement stone is formed, which leads to a significant increase in its strength characteristics. In parallel with this, the use of finely dispersed fillers, in particular burnt moulding waste (BMW), is a promising direction. This waste, which is a by-product of foundry production, contains pozzolanic components and can act as microfillers. The introduction of BFM contributes to the compaction of the structure by filling the intergranular space and also initiates additional hydration reactions, forming calcium hydrosilicates.

The aim of this study is to investigate the combined effect of a superplasticiser and a filler based on burnt moulding waste on the structure formation processes of cement stone.

2. Research methodology

In this study, the following materials and methods were used to obtain cement compositions.

Portlandcement: Portlandcement grade CEMI 32.5N was used as the main binder, in accordance with the requirements of ГОСТ 31108-2020 «Cements for general construction. Technical conditions».

Superplasticiser (SP): PRO 500 polycarboxylate superplasticiser was used, which complies with the requirements of ГОСТ 24211-2008 «Additives for concrete and mortar. General technical conditions».

Burnt moulding waste (BMW): The waste obtained after burning the moulding mixtures was ground to a powdery state. The chemical composition of the BWF included silicon (SiO_2), aluminium (Al_2O_3) and iron (Fe_2O_3) oxides, which confirmed their pozzolanic properties (Table 1).

Table 1

Chemical composition of BWF

Compounds	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	ZnO	п.п.п.
Amount (%)	91,56	0,022	4,58	1,12	0,2	0,1	1,30	1,18

An experimental determination of the phase composition of the formed cement stone was conducted using a high-precision AL-27mini diffractometer. The analysis was performed with the powder method to ensure high data

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reliability. A monochromatized CuKα-radiation source with a precise wavelength of λ=1.5406 Å was used, enabling high-precision identification of crystalline phases. The diffraction pattern was recorded in a step-by-step scanning mode with carefully controlled parameters: a 30 kV X-ray tube voltage, a 10 mA current, a scanning angular step of 0.02°, and a scanning speed of 1 degree per minute. This mode provided an optimal balance between resolution and data collection time, allowing for the detection of even minor changes in the phase composition of the samples.

For the analysis, four distinct types of compositions were prepared to investigate the effects of various additives on the cement system's phase development:

1. Composition 1: A normal consistency Portland cement-based composition without any additives (PC), serving as the control.
2. Composition 2: A composition consisting of normal consistency Portland cement and BWF, to study the effect of the pozzolanic additive alone.
3. Composition 3: A composition consisting of normal consistency cement binder and a polycarboxylate superplasticizer (SP), to isolate the effect of the plasticizer.
4. Composition 4: A complexly modified Portland cement-based composition using both BWF and SP, to examine the synergistic effects of both additives.

3. Results and discussions

A detailed description of the compositions of the studied cement stone samples, including the quantitative ratio of the main components and introduced additives, as well as the diffractogram data for interpreting the results of X-ray phase analysis, is presented in Tables 1 and 2. The X-ray phase analysis of the studied cement stone samples, shown in Figures 1 and 2, provides critical insights into the phase evolution at two key hardening periods: 1 and 28 days. These figures display characteristic diffraction spectra that allow us to examine the dynamics of changes in the intensity and position of reflections corresponding to various mineral phases. The diffractograms from the 1-day samples are crucial for understanding the initial hydration kinetics. They primarily reveal the rapid formation of early hydration products, such as ettringite ($C_6A_8H_{32}$), while the main phases of unhydrated cement, alite (C_3S) and belite (C_2S), still show strong peaks. By comparing the samples with and without additives at this stage, we can discern how SP influences the hydration rate and how BWF acts as a nucleation site for initial gel formation. The 28-day diffractograms, on the other hand, provide a comprehensive picture of the long-term microstructure maturity. At this stage, the intense peaks of portlandite ($Ca(OH)_2$) and other hydration products signify the progression of the hydration process. Specifically, the presence of Burnt Foundry Waste (BWF) in the compositions is expected to induce a pozzolanic reaction, which will be evident in the 28-day diffractograms by a notable reduction in the portlandite peak intensity, as $Ca(OH)_2$ is consumed to form additional C-S-H gel. Simultaneously, the inclusion of a Superplasticizer (SP), by facilitating more efficient hydration, might lead to a more advanced hydration degree at both 1 and 28 days, reflected in the relative intensities of the unhydrated and hydrated phases. This X-ray diffraction (XRD) analysis is a crucial aspect for understanding the fundamental mechanisms of hydration and structure formation in cement systems modified with BWF and SP, providing a direct link between the material's composition and its ultimate mechanical properties [5-15].

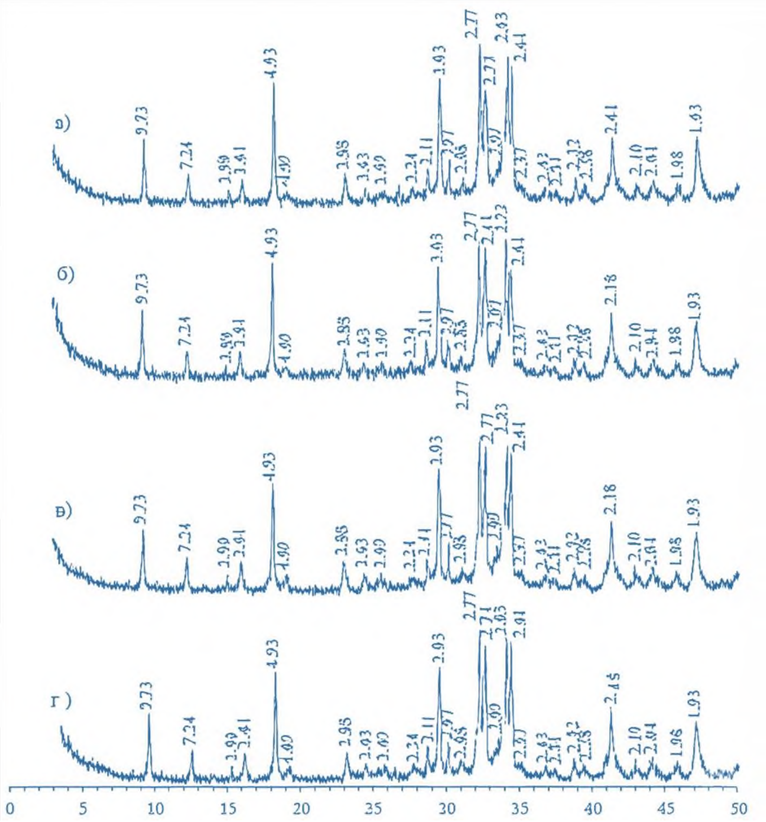

Figure 1. Diffractograms of 1-day cement stone samples:

a) PC, b) PC+BWF, c) SP, d) CM

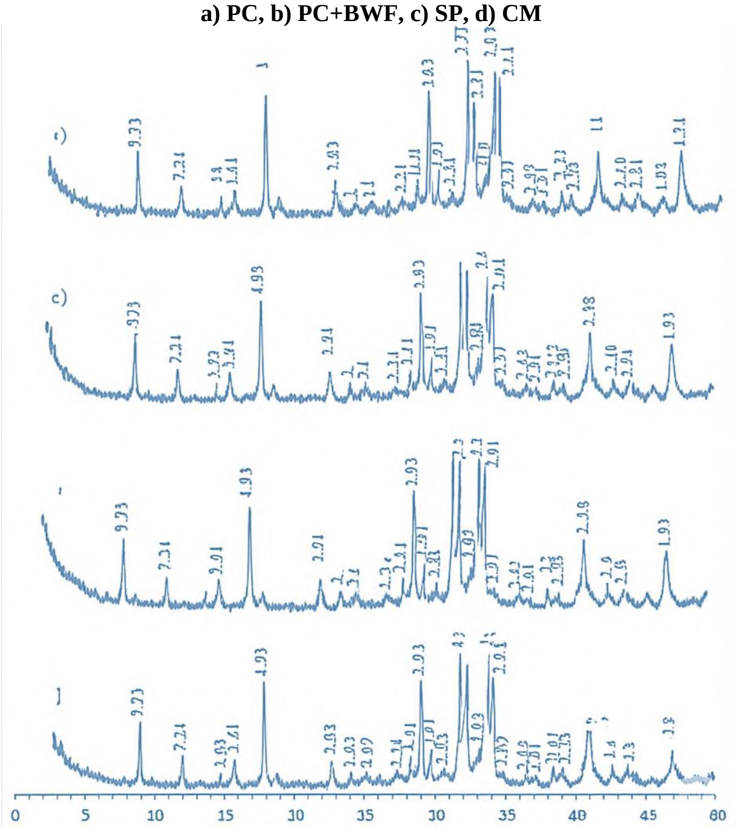

Figure 2. Diffractograms of 28-day cement stone samples:
a) PC, b) PC+BWF, c) SP, d) CM

Analysis of the data presented in Table 1 confirms that the introduction of burnt foundry waste (BWF) into the cement system has a significant influence on the kinetics of early-stage hydration processes. The results clearly demonstrate that the presence of BWF remarkably accelerates the hydration of the main clinker minerals, specifically alite (C_3S) and belite (C_2S), within the first 24 hours of hardening. This observation points to a pronounced accelerating or catalytic effect of the BWF particles. The primary mechanism behind this acceleration is the nucleation effect. Finely ground BWF particles, with their high specific surface area, provide a vast number of heterogeneous sites for the nucleation and growth of early hydration products, such as calcium silicate hydrate (C-S-H) gel and portlandite ($Ca(OH)_2$). By providing these pre-existing templates, the BWF bypasses the initial induction period of hydration, where supersaturation must be reached for crystal formation to begin. The XRD data in Table 1 visually support this, showing a more rapid decrease in the intensity of the unhydrated clinker peaks in the BWF-modified samples compared to the control. Simultaneously, the peaks corresponding to nascent hydration products would show a more rapid increase in intensity. This early-stage acceleration is critical as it sets the foundation for a more mature and dense microstructure at later ages, ultimately contributing to enhanced mechanical properties and the overall strength of the final cement stone.

Table 1

Effect of modifiers on the phase composition of 1-day-old cement stone

№	Mineral composition, %							
	$3CaO·SiO_2$	β-$2CaO·SiO_2$	$4CaO·Al_2O_3·Fe_2O_3$	$3CaO·Al_2O_3$	$Ca(OH)_2$	$3CaO·Al_2O_3$ $3CaSO_4·32H_2O$	C-S-H	Others
1	32,7	15,6	7,3	3,1	10,4	8,2	21,5	1,2
2	29,6	14,2	3,7	2,7	8,9	7,5	28,2	1,2
3	34,5	16,9	8,3	4,0	9,0	6,6	19,2	1,5
4	30,1	15,1	7,5	3,1	9,5	7,5	25,8	1,4

Analysis of the phase composition of cement stone modified with BWF showed a significant decrease in the concentration of unreacted clinker phases at the initial stage of hardening, i.e., within 1 day. Specifically, a decrease in the content of alite by 11%, belite by 10%, shareginite (brownmillerite) by 10%, and tricalcium aluminate by 12% was observed. This phenomenon, indicating the acceleration of hydration processes, is explained by the ability of negatively charged BWF particles to adsorb calcium ions (Ca^{2+}) on their developed surface. This adsorption effectively reduces the local concentration of Ca^{2+} ions in the pore solution, which in turn shifts the dissolution equilibrium of clinker minerals towards their stronger decomposition, thereby accelerating the overall hydration kinetics [16]. Notably, despite the replacement of 25% of the cement with BWF, the amount of portlandite ($Ca(OH)_2$) formed in the BWF cement stone decreased by 1.5% compared to the control sample (without additives). This fact is also a direct consequence of the accelerated hydration of cement clinker phases in the presence of BWF, as the active consumption of Ca^{2+} ions for the formation of calcium hydrosilicates (C-S-H) and other hydrate phases can occur before their release in the form of portlandite.

Contrary to the effect of BWF, the use of polycarboxylate superplasticizer slows down the hydration processes of clinker minerals in the first stage of hardening (1 day). This inhibitory effect is associated with the adsorption of superplasticizer macromolecules on the surface of cement particles. The resulting adsorption layer hinders the dissolution of the surface layers of the clinker phases and slows down both the nucleation and growth of hydration product crystals. Quantitative phase analysis confirmed these observations: the amount of unreacted alite in the superplasticizer composition increased by 6%, belite by 1%, brownmillerite by 11%, and tricalcium aluminate by 26% compared to the control sample. Such a slowdown in hydration leads to a decrease in the content of the main hydrate phases in the cement stone: portlandite decreases by 12%, ettringite by 10%. Additionally, it was noted that the formation of the amorphous phase of calcium hydrosilicates (C-S-H) slows down by 11% compared to the control mixture without additives, indicating a deceleration in the process of basic structure formation [17-19].

The introduction of a complex additive containing BWF and a polycarboxylate superplasticizer provides a synergistic effect, manifested by an acceleration of cement hydration on the 1st day of hardening, which effectively counteracts the inhibitory effect of the plasticizing component alone. In cement stone modified with the complex additive, a significant decrease in the concentration of unreacted initial minerals is observed by day 1: alite by 12%, belite by 10%, brownmillerite by 9%, and tricalcium aluminate by 25%. This intensification of the hydration process leads to a corresponding increase in the amount of hydrate phases in the cement stone at this stage: the amount of portlandite increases by 5% compared to the sample with only polycarboxylate superplasticizer, and ettringite by 14%. Thus, complex modification demonstrates the ability to optimize early hydration kinetics, contributing to the effective formation of the mineralogical framework of cement stone.

Table 2 presents the results of quantitative X-ray phase analysis of 28-day-old cement stone with the addition of complex additives BWF and SP.

Table 2

Effect of modifiers on the phase composition of 28-day-old cement stone

№	Mineral composition, %					
	$3CaO \cdot SiO_2$	$\beta\text{-}2CaO \cdot SiO_2$	$4CaO \cdot Al_2O_3 \cdot Fe_2O_3$	$Ca(OH)_2$	$3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O$	C-S-H
1	17,1	12,9	6,8	13,5	7,4	42,3
2	16,8	12,6	6,1	8,1	7,7	48,2
3	14,1	12,9	7,1	15,2	8,8	42,2
4	13,4	12,1	6,4	8,5	8,4	50,3

A detailed analysis of the phase composition of cement stone modified with BWF during the 28-day hardening period revealed a significant decrease in the content of Portlandite ($Ca(OH)_2$) by 38% compared to the control sample without additives. This indicates an intensification of the pozzolanic reaction of BWF in the final stages of hydration of cement systems. At the same time, an increase in the content of the amorphous phase by 19% was observed. This clearly indicates the formation of a highly dispersed, weakly crystallized structure enriched with low-basic calcium hydrosilicates (C-S-H). Such a change in the phase composition confirms the effective absorption of calcium hydroxide by the pozzolanic particles and the formation of more stable and dense hydrate products. This is important for improving the durability and performance properties of cement stone.

By the 28th day of hardening in cement stone containing a polycarboxylate superplasticizer, the alite content decreased by 18%, and the belite content by 3% compared to the sample without additives. This indicates that the hydration of these clinker minerals continued and even slightly accelerated in subsequent stages. In our opinion, this may be due to the optimization of water distribution and the formation of a stable microstructure under the influence of the superplasticizer. In addition, in the samples with the addition of superplasticizer, an increase in the content of portlandite by 11%, ettringite by 8%, as well as an increase in the proportion of the amorphous phase by 3% compared to the control composition, was observed. These changes indicate a complex effect of the superplasticizer. In addition to its main dispersing properties, it can also influence the kinetics of hydrate phase formation during long-term hardening.

On the 28th day of hardening, when a complex additive was applied, cement stone with a qualitatively altered structure was formed. A characteristic feature is a significant decrease in the content of portlandite by 41% and ettringite by 10%. At the same time, amorphous calcium hydrosilicates (C-S-H) predominate in the composition of the cement stone with complex application of additives. Their quantity increased by 22% compared to the sample without additives. This indicates a high degree of transformation of calcium hydroxide and the active formation of a binding gel. Also, in the composition with a complex additive

By day 28, the subsequent hydration processes of C_3S (alite) and C_2S (belite) gradually slowed down. This phenomenon is explained not only by the compaction of C-S-H gel around the cement grains as a result of the ongoing pozzolanic reaction of BWF, but also by the overall compaction of the cement stone structure. As a result, the porosity and permeability of the matrix decrease, further diffusion of water into unreacted clinker particles becomes difficult, and thus their hydration in subsequent stages slows down.

Experimental data covering the phase composition and microstructural properties of cement stone at different stages of hardening fully confirm the previously stated hypothesis about the complex mechanism of action of the proposed modifying additive. This confirmation applies both to its influence on the formation of mineralogical composition and indirectly to the final strength properties of cement stone. The research results show that the use of this complex additive in the creation of building materials with improved performance properties and long-term durability is promising.

4. Conclusion

Based on the conducted research, it was determined that applying the complex additive before the 28-day hardening period contributes to the formation of a denser, more uniform, and improved microstructure of the cement stone. An important factor determining these structural advantages is the high pozzolanic activity of BWF, which is a component of the complex additive. This activity is manifested by a significant decrease in the amount of portlandite
($Ca\ (OH)_2$), the main product of cement hydration, as well as a proportional increase in the amount of amorphous calcium hydrosilicates (C-S-H). Such a change in phase composition leads to the formation of a chemically more stable and less porous matrix. Calcium hydrosilicates formed as a result of the pozzolanic reaction are characterized by high density and better binding ability, which ultimately ensures the achievement of improved performance properties of the material, such as strength and durability.

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