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Study of the effect of filler from burnt moulding waste on the properties of cement systems

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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) on the properties of a cement system. The main objective of the study was to determine the optimal parameters of BMW (specific surface area and degree of filling) that provide the maximum synergistic effect with cementitious binder. Experiments have shown that the use of a superplasticiser (SP) in an amount of 1.0% of the cement mass is optimal for regulating the rheological and kinetic properties of the mixture. Analysis of the strength characteristics showed that the highest compressive and flexural strength is achieved when using BMW with a specific surface area of 3000 cm²/g at a filling degree of 25%. It was found that exceeding these values leads to a decrease in strength due to the dilution effect of the cement stone and the aggregation of filler particles. Special attention was paid to the influence of BMW storage conditions: it was shown that the deactivation of active centres on the particle surface caused by moisture adsorption leads to a monotonic decrease in strength. The results of the study confirm the high efficiency of BMW as a modifier of cement systems and provide practical recommendations for their use in obtaining materials with improved mechanical properties while simultaneously solving environmental problems.

Keywords: Cement stone, burnt moulding waste, superplasticiser, strength, specific surface area, hydration kinetics, filler, synergistic effect

1. Introduction

In the field of materials science and construction materials technology, it is widely recognised that the processes involved in the formation of cement stone are a complex, multi-factor system. A number of critical factors have a significant influence on its formation. Key among these are the chemical composition and physical properties of the modifiers and fillers added, which can radically change both the kinetics of hydration and the morphology of the hydrate phases formed. A deep understanding of these interrelationships is a prerequisite for the targeted control of the properties of cement systems, including their strength, durability and deformation characteristics [1-4].

Chemical additives and microfillers play an important role in optimising the properties of cement compositions [5-15]. The introduction of these components not only regulates the rheology of the cement paste, but also influences the microstructure of the final material. For example, superplasticisers (SP), thanks to their dispersing action, reduce the water demand of the mixture, which leads to an increase in the density of the cement stone and, as a result, its strength. At the same time, additives such as burnt moulding waste (BMW) act as microfillers and pozzolanic additives. Their high dispersibility allows them to fill the smallest pores between cement particles, and their chemical activity contributes to the formation of additional calcium hydrosilicates, which strengthen the structure of the material. This synergistic interaction between different components opens up wide opportunities for creating cement systems with specified performance characteristics.

As part of this study, a comprehensive analysis was conducted of the impact of superplasticisers (SP) and burnt moulding waste (BMW) on the key properties of cementitious binders. Changes in the normal density and setting times of cement paste were studied, which is crucial for the technological feasibility of construction work and control over the hydration process. Particular attention was paid to the kinetics of cement stone compressive strength gain at different hardening times. Compressive strength is the main criterion for the quality and reliability of the material. The evaluation of this parameter made it possible to determine the optimal dosages of additives and their combinations to ensure maximum efficiency. The results obtained provide an understanding of the mechanisms of interaction between additives and cement clinker, which serves as a basis for further developments in the field of composite binding materials.

2. Research methodology

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

Portlandcement: Portlandcement grade CEM I 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

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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

Determination of normal density and setting time: These parameters were determined on a cement test in accordance with ГОСТ 30744-2001 «Cements. Test methods using standard polyfractional sand».

Determination of compressive strength: Tests were carried out on beam samples measuring 40x40x160 mm, manufactured in accordance with ГОСТ 310.4-81 «Cements. Methods for determining the flexural and compressive strength». The samples were tested at 28 days of age.

3. Results and Discussion

Within the framework of this study, a systematic assessment was conducted on the influence of the superplasticizer (SP) dosage on the rheological and kinetic properties of cement paste. The concentration of SP was varied over a wide range from 0.2 to 1.2% by mass of cement, which allowed for a detailed analysis of how the paste's properties depend on the amount of additive used. The key data on normal consistency (N.C.) and setting times of the cement paste, which are critical indicators of workability and usability, are presented in detail in Table 1.

To ensure high accuracy and reproducibility of the results, the process of preparing the cement paste was strictly standardized. The introduction of water into the mixture was carried out in two stages. In the first stage, half of the total water volume was added by spraying to ensure uniform moistening of the cement. This promoted primary particle dispersion and prevented the formation of lumps. The remaining water, containing the dissolved SP, was then thoroughly mixed with the pre-wetted cement. This two-stage mixing method optimizes the interaction of the modifier with the cement particles, contributing to the effective manifestation of its plasticizing properties and its uniform distribution throughout the entire volume of the mixture.

The analysis of the data presented in Table 3.2 showed that an increase in the SP dosage leads to a systematic decrease in the normal consistency of the cement paste. This is due to the fact that the polycarboxylate superplasticizer, by adsorbing onto the surface of the cement particles, creates electrostatic or steric repulsion forces. This reduces inter-particle friction and lowers the water demand of the mixture. In parallel, a prolongation of the setting times was observed. This phenomenon is related to the formation of adsorption layers that slow down the cement hydration processes, which is a crucial factor for work under high-temperature conditions or during long-distance transportation of the mixture. Thus, precise SP dosing is critically important for controlling the rheological and kinetic characteristics of the cement system.

Table 1

Effect of SP dosage on normal consistency and setting time of cement paste

Additional dosage, %	NC, %	Setting time, hours and minutes	
		beginning	ending
0	27	1-10	6-20
0,2	26	1-40	6-00
0,4	24	2-00	5-40
0,6	22	2-15	5-20
0,8	21	2-25	5-10
1,0	19	2-45	4-40
1,2	19	2-45	4-40

Analysis of the data presented in Table 1 showed that an increase in the superplasticizer (SP) dosage from 0.2% to 1.2% by weight of cement leads to a notable decrease in the normal consistency (N.C.) of the cement paste by 2-8 points. The most pronounced reduction was observed at an SP concentration of 1.0%, while a further increase in the additive's concentration did not have a significant effect on this parameter. This effect is a direct result of the optimized dispersing action of the superplasticizer on the cement particles. The observed decrease in N.C. is explained by the strengthening of electrostatic and/or steric repulsive forces between the cement particles as the amount of adsorbed SP increases. This leads to a more effective fluidization of the cement paste and a reduction in its water demand. Simultaneously, the optimization of the adsorption rate of SP molecules on the surface of the cement particles provides a pronounced stabilizing effect, which is evident in a significant prolongation of the initial setting time of the cement paste—from 1 hour 10 minutes to 2 hours 45 minutes. This phenomenon is critically important for increasing the "pot life" of the cement mixture during transport and placement.

It is also noteworthy that an increase in the SP dosage leads to a reduction in the final setting time of the binder—from 6 hours 20 minutes to 4 hours 40 minutes. This effect can be explained by the accelerating properties of SP, which begin to manifest as the additive penetrates the inter-particle space and becomes uniformly distributed. The improved dispersion of cement particles and a more effective distribution of water contribute to a more complete and intensive course of hydration processes in the subsequent stages. This, in turn, leads to the faster formation of a strong and homogeneous structure in the cement stone.

Considering both the technological and economic aspects, the most optimal values for N.C. and setting times were achieved at an SP dosage of 1.0% of the binder's mass. This concentration ensures maximum plasticizing efficiency without excessive costs or an undesirable over-prolongation of the setting times. Consequently, in all subsequent experimental studies aimed at examining the influence of other additives, the superplasticizer dosage was maintained as a constant at 1.0% of the total binder mass.

Modern scientific research [16-18] has established a direct correlation between the concentration of Brønsted active adsorption centers on the surface of powdered materials and an increase in their fineness (dispersion). These centers play a decisive role in determining the material's reactivity. However, this relationship is only evident up to a certain specific surface area (Sud), after which the rate of increase in reactivity slows down significantly. Consequently, for the effective use of freshly ground fillers, it is crucial to determine the optimal value of Sud. Exceeding this value leads to only a marginal increase in the surface activity of the particles, making further grinding economically and energetically impractical.

Within the scope of this study, we investigated the effect of the specific surface area of burnt foundry waste (BFW), used as a filler, on the strength of cement stone. The control samples were made from cement paste of normal consistency, with the consistency of the binder mass being 27%. The experimental research was aimed at determining the optimal specific surface area of the filler and the optimal degree of filling for the cement binder. To achieve this, the specific surface area of the BFW microfiller was varied in 1000 cm²/g increments, ranging from 1000 to 4000 cm²/g. Various filling levels were also used: 5%, 10%, 15%, 20%, 25%, 30%, 35%, and 40% by mass of cement.

During the study, the strength characteristics of the cement binder, modified with varying degrees of microfilling and a variable specific surface area of the filler, were examined in detail. For this purpose, fine-grained beam specimens measuring 4x4x16 cm were fabricated and tested. The resulting experimental data were subjected to rigorous statistical analysis. This step is critically important for minimizing random errors, identifying statistically significant relationships, and confirming the reliability of the results. Based on this analysis, graphical relationships were plotted and are presented in Figures 1 and 2. These graphs are a key tool for interpreting the relationship between the degree of microfilling of the cement stone, the specific surface area of the material used, and its strength properties.

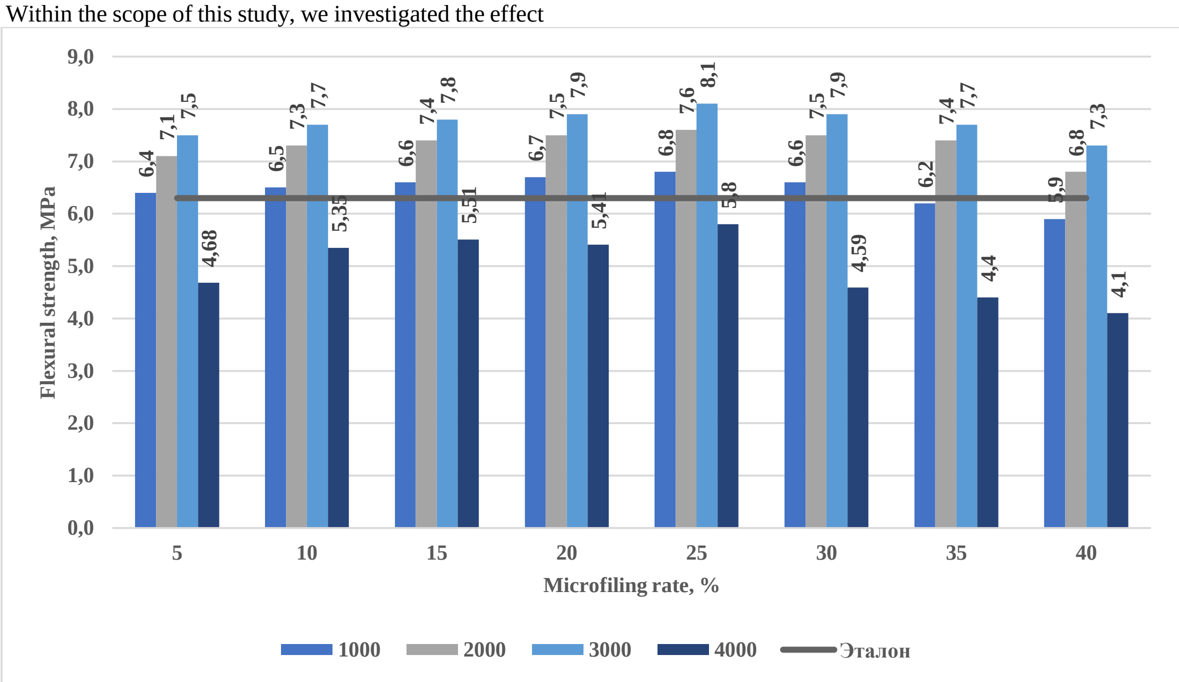

Figure 1. Influence of the relative surface area and the degree of microfiling of BWF on the bending strength of cement stone

Analysis of the data presented in Figures 1 and 2 demonstrates that the influence of both the degree of microfilling and the specific surface area of the filler on the compressive and flexural strength of cement stone increases monotonically. This indicates a direct positive correlation between these parameters and the mechanical properties of the hardened cement paste.

However, this growth isn't limitless. The highest strength values were observed for compositions with burnt foundry waste (BFW) having a specific surface area (Sud) of 3000 cm²/g at a microfilling level of 25% by mass of the binder. This is a critical finding, as it points to the existence of an optimal balance for achieving a maximum synergistic effect between the cement and the filler. At this precise point, the filler particles effectively pack into the interstitial spaces of the cement, densifying the matrix and promoting more complete hydration, which leads to enhanced mechanical properties.

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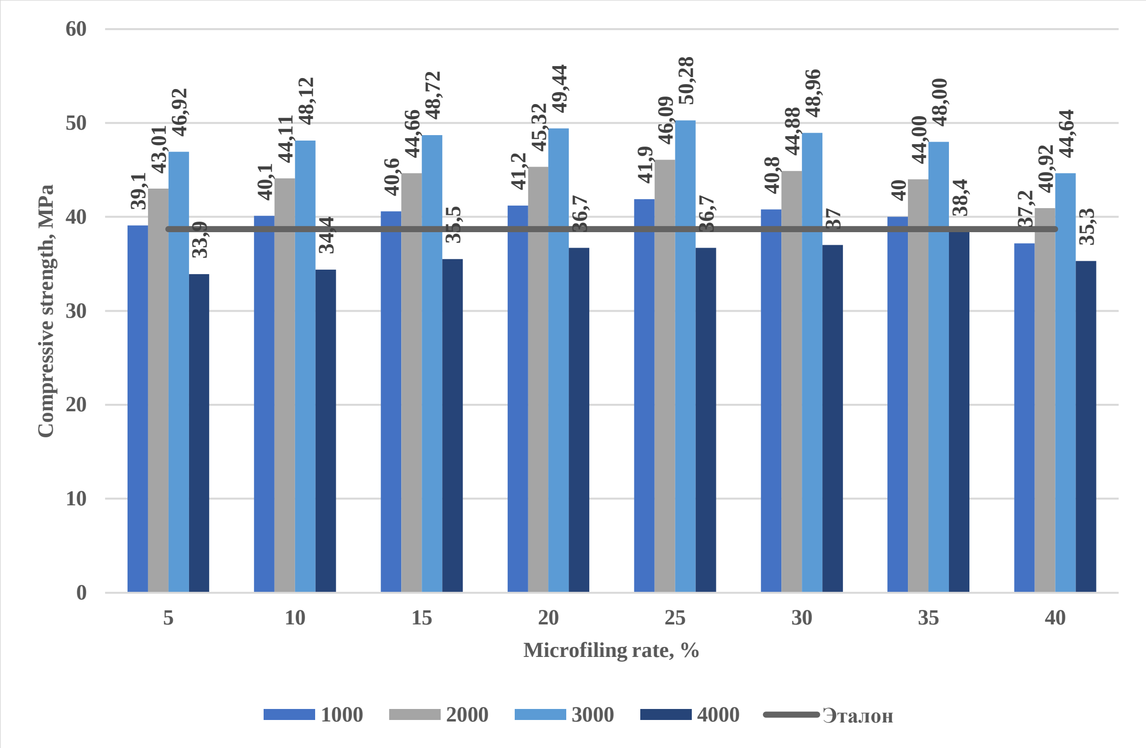

Figure 2. Effect of the relative surface area and degree of microfilling of BWF on the compressive strength of cement stone

It's important to note that exceeding the optimal filling level of 25% leads to a consistent decrease in the strength of the cement stone. This phenomenon is primarily explained by a simple but fundamental factor: the relative proportion of the initial cement in the total binder mass decreases. With the excessive introduction of the filler, the amount of cement gel—the primary component responsible for forming the strong, load-bearing framework of the hardened paste—is reduced. This dilution effect directly and negatively impacts the mechanical properties of the final material.

Furthermore, the study established that an increase in the specific surface area of the filler beyond 3000 cm²/g also adversely affects the mechanical properties of the cement stone. The main reason for this negative effect is the agglomeration of particles, a phenomenon exacerbated by surface polarization during mechanical activation. The high surface energy of excessively ground particles promotes their self-adhesion, leading to the formation of heterogeneous clusters instead of a uniform dispersion. These aggregations reduce the overall density of the composite, disrupt the formation of a continuous and monolithic cement matrix, and create internal stress concentration zones. Consequently, these structural defects collectively lead to a reduction in the overall strength. The results obtained are in strong agreement with the data presented in Fig. 3, which confirms the proposed hypothesis regarding the mechanisms of strength reduction when the filler's dispersion is over-optimized.

Taking this into account, in the subsequent series of experiments, the normal consistency and setting times of the binder with 1.0% superplasticizer (SP) and a microfiller with a specific surface area of ~3000 cm^2/g were determined. The amount of microfiller was 25% of the binder's mass. The results of determining the normal consistency and setting times of the microfilled binder are presented in Table 2.

Analysis of the presented data shows that 25% of the BWF microfiller does not affect the normal consistency indicators at an SP dose of 1.0%. It can be observed that the final stages of the setting period are somewhat shortened.

Table 2

Influence of the BWF microfiller with a specific surface area of ~3000 cm^2/g on the normal consistency and setting times of cement paste with a filling rate of 25% and an SP additive of 1.0%

Additional dosage, %	NC, %	Setting time, hours and minutes	
		beginning	ending
0	27	1-10	6-20
0,2	26	1-40	5-50
0,4	24	2-00	5-20
0,6	22	2-15	5-10
0,8	21	2-25	4-50
1,0	19	2-45	4-00
1,2	19	2-45	4-00

During the storage of freshly ground mineral fillers, a gradual decrease in the concentration of active centers on their surface is observed. This process, known as surface deactivation, is a critical phenomenon that significantly impacts the reactivity and subsequent performance of these materials. The primary driver of this deactivation is the adsorption of water molecules from the surrounding environment, a process whose rate is highly dependent on atmospheric humidity.

The mechanism of deactivation is complex and multi-faceted. On the surface of the filler, water molecules undergo dissociation into protons (H^+) and hydroxyl ions (OH^-). This is facilitated by the strong interaction between the material's proton-donating surface centers and various impurity sites, which form hydrogen bonds with the H_2O molecules. In addition to the direct adsorption of water, this deactivation can also be initiated by the absorption of H^+ and OH^- ions or radical products from the environment. Beyond water, the adsorption of other atmospheric components, such as oxygen (O_2) or nitrogen (N_2), can also affect filler activity. In this case, a "saturation" of the active centers occurs, with O_2 and N_2 molecules acting as electron acceptors or donors, respectively. This alters the electron concentration on the material's surface, leading to its deactivation [19-23].

Given these theoretical considerations on the deactivation of the active surface of mineral fillers, an experimental study was conducted to investigate the influence of storage time on the strength of cement stone. This research, with results presented in Figure 3, was designed to provide a quantitative assessment of how changes in the surface activity of fillers over time affect the final mechanical properties of the composite material. Understanding this relationship is crucial for practical applications, as it provides a basis for determining optimal storage conditions and usage timelines for fillers to ensure consistent and high-quality material performance.

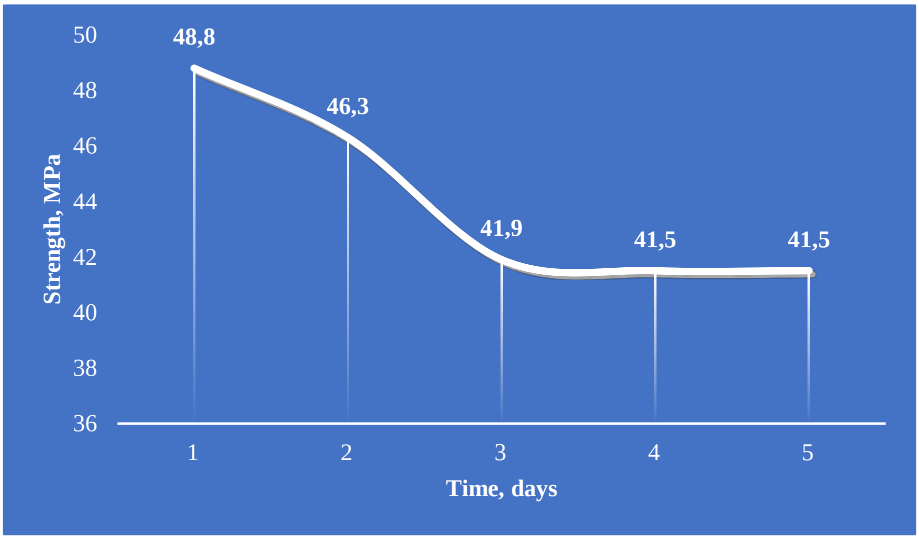

Figure 3. Decrease in micro-filler activity due to moisture adsorption over time following grinding

As demonstrated in Figure 3, a clear correlation exists between the storage time of the burnt foundry waste (BFW) microfiller and the mechanical properties of the resulting cement stone. The most rapid decline in the number of active centers on the surface of the BFW is observed within the initial 24 hours of exposure to air, which directly corresponds to a monotonous decrease in the cement stone's strength. This phenomenon is rooted in the high surface energy of the freshly ground BFW particles. Upon exposure to the atmosphere, these highly reactive surfaces readily adsorb ambient water vapor and other gases. This adsorption process leads to the neutralization of the Brønsted active adsorption centers, which are crucial for the filler's reactivity. The surface becomes saturated with adsorbed molecules, effectively passivating it and significantly reducing its ability to participate in subsequent pozzolanic reactions with calcium hydroxide ($Ca(OH)_2$). The decline in the BFW's reactivity directly compromises its function as both a filler and a pozzolanic additive. Initially, the active centers facilitate the nucleation of C-S-H gel, providing additional sites for hydration product formation and leading to a denser, stronger microstructure. As these centers become deactivated, the filler's ability to act as nucleation sites diminishes. Consequently, the formation of the dense hydration product network is less efficient, and the final hardened cement paste exhibits lower mechanical properties. The monotonous decrease in strength over time, as shown in the figure, is a direct manifestation of this ongoing surface deactivation. The results underscore the critical importance of proper storage and timely use of freshly ground BFW. To maximize its beneficial effects on the strength of cement-based materials, the filler must be used as soon as possible after its production or stored in a controlled, low-humidity environment to prevent surface deactivation. These findings provide a scientific basis for practical guidelines aimed at optimizing the performance of sustainable construction mtaerials that incorporate industrial waste.

4. Conclusion

Studies have conclusively demonstrated that burnt moulding waste (BMW) has significant potential for use as an effective microfiller in cement systems. It has been established that the optimal specific surface area of BMW is 3000 cm²/g, and the optimal filling degree is 25% of the cement mass. Achieving maximum strength at these parameters is due to synergistic interaction, where BWF not only compacts the structure but also participates in pozzolanic reactions. Another important conclusion is the need to control the storage conditions of the filler, since its reactivity critically depends on time and humidity. The use of freshly ground BWF or storage in dry conditions allows its active centres to be preserved and maximises its contribution to strength characteristics. Thus, the use of BWF is not only an economically viable and environmentally sound solution for industrial waste disposal, but also an effective method for improving the properties of building materials, opening up new prospects in the field of sustainable construction.

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