Investigation of the influence of technological factors and compositions of binders on the strength characteristics of blast–furnace cement with magnetized ferromagnetic additives

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Abstract. Cement production is a significant source of carbon dioxide emissions. One of the ways to reduce emissions is to reduce the proportion of clinker in cement by introducing active mineral additives into its composition, particularly granulated blast-furnace slag. One of the ways to increase the activity of such cement is the effect of magnetic fields on the spin multiplicity of the substances involved in the hardening reaction. In this case, the maximum effect is ensured by introducing a magnetized finely dispersed ferromagnetic substance into the cement composition. The activation effect depends on the additive’s adding method to the cement’s composition, the components ratio in the cement, and the cement hardening mode. This work aims to identify the influence of the adding method of the additive, the slag and additive proportion in cement, and the steam curing temperature on the activity effect of the binder. Three groups of studies were carried out to determine the strength characteristics of laboratory samples hardened both in natural conditions and during steaming. In the first group, cement samples have tested containing 40% slag obtained by joint grinding and joint mixing of the additive with cement for 0.5, 1, 2, 4 and 8 minutes. In the second group, samples have tested with the additive amount varied from 0 to 2.5%, and slag amount from 0 to 80%. Finally, cement samples were tested in the third group containing 50% slag and from 0 to 2.5% additive. The samples were steam cured at temperatures ranging from 50 to 90°C and tested one day, 28 and 90 days after steam curing. As a result of the research, it has revealed that to obtain the maximum effect, the additive must be introduced into the cement composition by joint grinding. The factors influencing the activity have been determined. At the same time, the time of joint grinding should ensure uniform mixing of the components and the formation of new surfaces of cement grains in the presence of magnetic fields. Too long joint grinding leads to the loss of the magnetic properties of the addition. It was found that the activation effect from the additive addition increases with an increase in the proportion of slag. In cement without slag, an increase in the additive content leads to a drop in strength. It was revealed that the introduction of magnetized ferromagnetic dust additives into the composition of the SPC makes it possible to reduce the steam curing temperature of products by 20-25°C. Studies have shown that using a finely dispersed ferromagnetic substance as an activating additive can save energy resources and reduce emissions.
1. The problem formulation and its relationship with scientific and practical tasks
For the foreseeable future, cement will remain one of the primary materials used in construction. Yearly more than 4,100,000 tons of cement are produced in the world [1]. Cement production is highly energy-intensive and involves the release of significant amounts of carbon dioxide into the atmosphere. The amount of CO₂ emissions associated with cement production is 8% of the global emissions [2] has reached 2.2 gigatons of carbon dioxide per year [3] and continues to grow. About 50% of the emission from cement production occurs during the calcination of limestone during the production of portland cement. Another 40% of emissions occurs when fossil fuels are burned in clinker kilns [4]. Thus, in cement production, 90% of the emissions occur in the production of clinker.

Reducing the energy intensity of cement production and reducing the volume of CO₂ emissions into the atmosphere may be by improving the production technology [5, 6], using alternative fuels [7,8], and capturing and storing carbon [9,10]. However, the most effective way of emissions tumbling in cement production is to reduce the clinker proportion in cement [11,12]. Reducing the balance of clinker in cement can decrease emissions by more than 60% [13].

2. Research and publications analysis
A decrease in the proportion of clinker is possible due to the introduction of active mineral additives into the cement composition. Industrial waste is most often used as such additives. In addition to reducing the consumption of fossil fuels and emissions, the introduction of active mineral additives in cement composition gives them additional favourable properties. For example, the introduction of granulated blast-furnace slag (from now on BFS) into the cement composition increases the corrosion resistance of the cement. National standards limit the share of active mineral additives introduced into cement composition since exceeding the maximum permissible value degrades cement’s strength and other characteristics [14]. However, a further increase in the proportion of active mineral additives in cement without a drop in strength is possible using various activation methods.

We are investigating the mechanism of activating blast–furnace cement (from now on BFC) by introducing magnetized ferromagnetic dust additive (from now on MFDA) into their composition. The magnetic fields effect on chemical reactions has been well studied and, in particular, considered in works [15–18]. Magnetic fields affect spin dynamics and control the spin multiplicity of radical pairs of substances involved in a chemical reaction. Thus, even weak magnetic fields affect the chemical activity of reagents [19, 20]. Furthermore, the influence of magnetic fields on the hardening of binders is considered in works [21–27]. In research results [28, 29], it has been proved that both the magnetization of particles and their dispersed composition affect the strength characteristics of BFC.

The effect of the MFDA on the cement hardening mechanism and the structure of cement stone was studied in [30]. Our research has confirmed that the magnetic fields created by the dust-like additive can positively affect cement activity. Furthermore, during the investigation, nonlinear dependence’s of the activation effect created by the MFDA on the technological parameters of cement hardening were revealed. In particular, studies have shown that heat and moisture treatment of hardening cement reduces the activation effect. We have conducted additional research studies to determine how the method of introduction and the duration of mixing of the MFDA, the proportion of BFS in the cement and the temperature of cement hardening affect the activity of the binder. This article presents the results of these studies.

3. Statement of material and results
3.1. Influence of the mixing method
Previous studies [29,30] have significantly influenced the methods and time of mixing on the resulting activation effect.
The existing technology for cement production allows mixing the binder components either with the creation of tribochemical processes or without them. In the first case, mixing is carried out in ball mills; in the second, using mechanical mixers or aeromixers. Mixing components in a ball mill can be combined with grinding of clinker, gypsum and BFS or carried out after that.

Three series of experiments were comprised in the research to simulate different ways of mixing the MFDA with binders:

– clinker, blast furnace slag and gypsum were mixed into a laboratory ball mill in a ratio of 55.0:40.0:5.0 by weight. The mill was filled with MFDA in the amount of 2% by the weight of the binder components. Next, joint grinding was carried out to obtain cement with $S_{sp} = 3100 \text{cm}^2/g$. Finally, an MFDA-free control binder was prepared similarly;

– a ready-made BFC (CEM III/A) containing 40% BFS and 5% gypsum were placed in a laboratory ball mill. The MFDA and CEM III/A were mixed in 2% of the total mass of cement into the mill. In different experiment series, dust and cement were mixed for 0.5; 1; 2; 4; 8; 16 minutes. Simultaneously, additional mixing of the control samples was carried out without an MFDA;

– the MFDA was introduced into the CEM III/A and mixed in a specially designed device for 0.5; 1; 2; 4; eight; 16 minutes.

This device has been designed to mix the binder with the MFDA without the influence of tribochemical factors. It contains a steel cylinder with 80 mm diameter and 200 mm length and a drive that rotates the cylinder at 250 r/pm. Five cylpebs weighing 100 g each were placed in the cylinder together with the cement to improve the mixing processes. Further tests were carried out following by DSTU EN 196-1:2007. The test results showed in table 1.

Mixing the MFDA with a binder by the first way does not create, and the third way creates a weak activation effect. However, when mixing the components according to the second method, with an increase in the mixing time, the activation effect first increases and then decreases again (figure 1, figure 2).

Figure 1. Influence of the grinding time of the components on the activation effect (A) created by the MFDA.

Figure 2. Influence of the mixing time of the components on the activation effect created by the MFDA.

The results show that the joint grinding time dependence of the activation effect with the MFDA has the form:

– natural hardening
Table 1. Influence of the method and time of mixing the MFDA with the binder on the strength characteristics of cement-sand mortars.

| Mixing method | Type of binder | Mixing time [min] | Strength [MPa] | Steam curing | Natural hardening |
|---------------|----------------|-------------------|----------------|--------------|------------------|
| 1. Joint grinding | clinker+BFS+ +gypsum+MFDA up to specific surface area 310 m²/kg | 3.9 | 18.92 | 5.02 | 23.90 |
| 2. Joint grinding | clinker+BFS+ +gypsum up to specific surface area 310 m²/kg | 3.9 | 18.92 | 5.02 | 23.98 |
| 3. Grinding | BFC + MFDA | 0.5 | 4.07 | 21.87 | 5.09 | 26.88 |
| 4. Grinding | BFC | 0.5 | 4.02 | 19.81 | 5.04 | 24.82 |
| 5. Grinding | BFC + MFDA | 1 | 4.16 | 24.54 | 5.30 | 30.14 |
| 6. Grinding | BFC | 1 | 4.07 | 20.26 | 5.09 | 25.35 |
| 7. Grinding | BFC + MFDA | 2 | 4.29 | 27.16 | 5.48 | 34.94 |
| 8. Grinding | BFC | 2 | 4.13 | 21.07 | 5.19 | 26.50 |
| 9. Grinding | BFC + MFDA | 4 | 4.52 | 31.6 | 5.62 | 42.34 |
| 10. Grinding | BFC | 4 | 4.20 | 22.19 | 5.30 | 28.80 |
| 11. Grinding | BFC + MFDA | 8 | 4.65 | 34.5 | 5.81 | 47.98 |
| 12. Grinding | BFC | 8 | 4.29 | 25.04 | 5.52 | 33.41 |
| 13. Grinding | BFC + MFDA | 16 | 4.62 | 31.52 | 5.81 | 45.04 |
| 14. Grinding | BFC | 16 | 4.48 | 30.98 | 5.73 | 41.97 |
| 15. Mixing | BFC + MFDA | 0.5 | 4.02 | 19.10 | 5.04 | 24.39 |
| 16. Mixing | BFC | 0.5 | 4.01 | 18.98 | 5.04 | 24.24 |
| 17. Mixing | BFC + MFDA | 1 | 4.03 | 19.32 | 5.05 | 24.77 |
| 18. Mixing | BFC | 1 | 4.01 | 19.1 | 5.04 | 24.31 |
| 19. Mixing | BFC + MFDA | 2 | 4.05 | 19.79 | 5.05 | 25.88 |
| 20. Mixing | BFC | 2 | 4.02 | 19.15 | 5.03 | 24.38 |
| 21. Mixing | BFC + MFDA | 4 | 4.05 | 20.65 | 5.03 | 27.06 |
| 22. Mixing | BFC | 4 | 4.02 | 19.13 | 5.01 | 24.27 |
| 23. Mixing | BFC + MFDA | 8 | 4.04 | 21.29 | 5.01 | 27.43 |
| 24. Mixing | BFC | 8 | 4.01 | 19.14 | 4.99 | 24.01 |
| 25. Mixing | BFC + MFDA | 16 | 4.04 | 21.52 | 5.01 | 27.54 |
| 26. Mixing | BFC | 16 | 4.01 | 19.14 | 4.98 | 24.01 |

\[
A_c = 0.672t^2 + 10.97t + 3.602; \\
A_m = -0.103t^2 + 1.404t + 1.617; \\
\]

steam curing
\[
A_c = 0.645t^2 + 11.675t + 2.181; \\
A_m = -0.179t^2 + 3.124t - 0.834; \\
\]

where \( A_c \) - is the activation effect (% increase in strength) for ultimate compressive strength; \( A_m \) - ditto for ultimate strength in bending; 
\( t \) - mixing time in minutes.
The revealed regularities show that a magnetized substance must participate in forming the binder’s surface for the manifestation of activation properties. In this case, magnetic fields can influence the defectiveness of the binder structure and thus its activity. Consequently, the magnetic fields induced by the dust affect the tribochemical phenomena occurring during cement grinding.

The grinding of the MFDA with the binder has a tribochemical effect on cement and the dust itself. The grinding reduces the size of dust particles and causes them to heat up. With an increase in the grinding time, the temperature of the particles passes the Curie point and causes their demagnetization. Another feature is that the magnetic properties of particles manifest themselves only if their size exceeds the critical one [31, 32]. This leads to an activation effect decrease after a specific time of MFDA joint grinding with cement. Thus, long-term grinding completely neutralizes the impact of MFDA. According to the results of the experiments, the optimal time for grinding the MFDA with cement is 5-10 minutes.

3.2. Selection of optimal compositions of BFC with an MFDA

Studies of the strength characteristics of binders with different BFS/MFDA ratios were carried out to identify the optimal amount of MFDA in cement with different BFS content. Investigated binders, having in their composition in various series 0; 1.25; 2.5% MFDA, 0; 20; 40; 60; 80% BFS, 4.5% gypsum. The components of the BFS were ground in a ball mill to obtain $S_{sp} = 310 m^2/kg$, then dust was added, and the grinding was performed for 8 minutes. Further tests were carried out according to the by DSTU EN 196-1:2007 method.

The test results showed in table 2, figure 3 and figure 4.

**Table 2.** Strength characteristics of cement with different BFS and MFDA content.

| Slag fraction in BFC | Natural hardening [MPa] | Steam curing [MPa] |
|---------------------|------------------------|--------------------|
|                     | MFDA amount [%]        |                    |
|                     | 0.00 1.25 2.50 0.00 1.25 2.50 |
| 0                   | $R_m$ $R_c$ $R_m$ $R_c$ $R_m$ $R_c$ $R_m$ $R_c$ $R_m$ $R_c$ |
| 20                  | 6.40 45.55 5.59 35.53 5.59 34.57 4.55 26.34 4.53 26.40 3.78 17.62 |
| 40                  | 6.06 40.58 5.65 42.28 6.10 42.23 4.46 26.77 4.56 30.39 4.51 24.92 |
| 60                  | 5.52 33.24 5.70 50.75 6.20 48.02 4.28 25.05 4.62 29.23 4.78 32.50 |
| 80                  | 5.11 27.16 5.62 49.88 6.15 48.14 4.12 22.27 4.53 26.95 4.78 32.78 |

The results show that the dependence of the strength $R$ on the consumption of the MFDA and the BFS has the form:

- natural hardening
  \[ R_m = 6.47 - 1.21D + 0.35D^2 - 0.03S + 0.04SD - 0.01SD^2 - 0.0002S^2D \]
  \[ R_c = 45.79 - 14.28D + 3.74D^2 - 0.28 \cdot S + 1.34SD - 0.38SD^2 - 0.0004S^2 - 0.01S^2D + 0.003S^2D^2 \]

- steam curing
  \[ R_{m,t} = 4.58 + 0.03D - 0.13D^2 - 0.005S + 0.02SD \]
  \[ R_{c,t} = 26.26 + 4.45D - 3.31D^2 + 0.08S + 0.006SD + 0.09SD^2 - 0.003S^2 - 0.0002S^2D - 0.0006S^2D^2 \]

where: $D$ is the amount of MFDA, %;
Figure 3. Activation effect $A$, achieved with the introduction of MFDA to the BFC of various compositions, (a) - compression, normal hardening, and (b) - bending, normal hardening.

Figure 4. Activation effect $A$, achieved with the introduction of MFDA to the BFC of various compositions, (a) - compression, steam curing, and (b) - bending, steam curing.

$S$ - the amount of BFS, %.

With an increase in the proportion of BFS, the need for MFDA increases, and the activation effect enlarges. At the same time, an increase in the amount of MFDA added to BFS - free cement causes a drop in the activity of the binder.
The described effects confirm the conclusions of [30]. Furthermore, the data obtained show that introducing an MFDA into the composition of the BFC makes it possible to obtain cement of equal strength with an increased BFS content against the usual range. The resulting decrease in the share of clinker makes it possible to significantly reduce the consumption of fossil fuels and emissions associated with cement production.

3.3. Optimization of hardening regimes of blast-furnace cement with MFDA

Earlier studies [33] showed that the introduction of activating additives into cement composition causes a decrease in the optimum steam curing temperature. Studies of the strength characteristics of cement steamed at different temperatures with different amounts of the added MFDA were carried out to clarify the optimal hardening regimes. The study used cement containing 50% BFS. First, the cement mixing with the MFDA was carried out in a ball mill for 8 minutes. Then, steam curing was carried out according to the mode 3 + 6 + 2 hours at temperatures 50°, 70° and 90°. Samples have tested 24 hours, 28 days and three months after steaming. The research results are summarized in table 3 and shown in figure 5, figure 6 and figure 7.

**Table 3.** Influence of the amount of MFDA and steam curing temperature on the strength characteristics of the BFC.

| The amount of MFDA in the BFC, % | Steam curing temperature, °C | Ultimate strength Rc [MPa], mortar samples 7.07×7.07×7.07 cm through 24 hours after steam curing | 28 days after steam curing | 90 days after steam curing |
|---------------------------------|------------------------------|---------------------------------------------------------------------------------|--------------------------|-------------------------|
| 0                               | 50                           | 9.8                                                                              | 19.1                     | 20.3                    |
|                                 | 70                           | 12.8                                                                             | 19.9                     | 20.7                    |
|                                 | 90                           | 17.8                                                                             | 23.6                     | 24.2                    |
| 1.5                             | 50                           | 12.8                                                                             | 21.9                     | 24.8                    |
|                                 | 70                           | 17.1                                                                             | 29.2                     | 30.5                    |
|                                 | 90                           | 21.4                                                                             | 24.9                     | 25.9                    |
| 2.5                             | 50                           | 14.5                                                                             | 28.6                     | 31.8                    |
|                                 | 70                           | 20.9                                                                             | 35.8                     | 36.2                    |
|                                 | 90                           | 23.2                                                                             | 30.4                     | 30.6                    |

With an increase in the amount of MFDA in the binder, a more significant increase in strength is observed at lower temperatures. Thus, the combined action of the MFDA and temperature increases the destructive processes occurring during clinker hydration and, simultaneously, intensifies the process of binding hydrated lime and slag hardening. The processing results showed that the samples strength dependence on the MFDA content and the steam curing temperature is as follows:

\[
R_1 = 13.58 - 12.23D + 0.21D^2 - 0.23T + 0.42TD - 0.003TD^2 + 0.003T^2 - 0.003T^2D
\]

\[
R_{28} = -29.18 - 14.79D + 3.18D^2 + 1.49T + 0.39TD - 0.03TD^2 - 0.01T^2 - 0.002T^2D
\]

\[
R_{90} = -23.87 - 7.94D + 2.83D^2 + 1.36T + 0.247TD - 0.028TD^2 - 0.01T^2 - 0.001T^2D
\]

where \(R_1, R_{28}\) and \(R_{90}\), respectively, the strength of the samples after 1, 28, 90 days after steaming:
Figure 5. Influence of isothermal heating temperature on the MFDA activation effect created one day after steam curing.

Figure 6. Influence of isothermal heating temperature on the MFDA activation effect created 28 days after steam curing.

Figure 7. Influence of isothermal heating temperature on the MFDA activation effect created 90 days after steam curing.

$T$ is the steam curing temperature in degrees Celsius;

$D$ - MFDA consumption in %.

The research results show that the most significant increase in strength has been observed in samples steamed at 70°. The recommended steam curing temperature for ordinary BFC is 90-95° C. The introduction of MFDA into the composition of the BFC makes it possible to reduce the steam curing temperature of products by 20-25° C. Thus, the introduction of MFDA makes it possible to save energy resources on heat treatment prefabricated reinforced concrete significantly.
4. Conclusion
As a result of the research, it has revealed that the optimal method for introducing the MFDA into the binder is a method that ensures the simultaneous passage of tribochemical reactions in cement during the joint grinding of the components. Thus, the optimal time for joint grinding of ingredients is substantiated.

A relationship has been established between the activation effect: the consumption of BFS and MFDA. Furthermore, it has revealed that the MFDA addition activation effect increases with an increase in the proportion of BFS in the binder from 0 to 80%.

It has been determined that the introduction of the MFDA into the binder composition allows one to lower the optimum steam curing temperature by 20–25°C, which significantly reduces energy consumption.

Thus, introducing an MFDA into the composition of BFC allows reducing energy consumption and emission by reducing the proportion of clinker in cement and reducing the temperature of heat and moisture treatment of products on this cement.

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