



Increase of Wear Resistance and Strength of Cementing Mortars Due to Stabilizing Fine-Dispersed Additives of Metallurgical Sludge

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Abstract

The main aim of this study is to determine the efficiency of enhancement of the strength and heat-resistant characteristics of cementing mortars by addition of metallurgical sludge in the form of iron oxide powder, which is a waste product in steel production, to the composition of a finely dispersed fraction. The use of stabilizing strengthening additives is due to the need to increase the strength properties of cementing solutions, especially those that are used in extreme conditions, at high temperatures and high loads, creating pressure on the formations. At the same time, the use of expensive strengthening stabilizers increases the cost of manufacturing cementing solutions, which in turn leads to an increase in the cost of the entire process of creating oil wells. According to the data presented, the use of finely dispersed and ultra-finely dispersed fractions of metallurgical sludge in the form of hematite powder leads to an increase in the strength properties of cementing solutions, and also increases resistance to long-term thermal heating due to an increase in resistance to high-temperature degradation associated with cracking and embrittlement. It was also determined that the transition from a finely dispersed fraction and an ultrafinely dispersed fraction leads to an increase in strength properties, which makes it possible to reduce the consumption of the stabilizing strengthening additive powder while maintaining strength characteristics.

Keywords: Cementing solutions; Strengthening; Fine powders; Strengthening additives; Heat resistance.

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1. Introduction

One of the key factors in the production of cementing solutions used to secure oil wells is their resistance to high pressures that occur at great depths, as well as thermal stability and heat resistance, the main parameters that determine the stability of solutions to high-temperature operating conditions.^[1-3] The parameters of strength and wear resistance of cementing solutions are the most critical parameters, as they determine the longevity of well operation, as well as their operating modes, which are often accompanied by exposure to high formation pressures, various vibrations during well operation, as well as temperature heating and temperature changes caused by both climatic features and operating

processes.^[4,5] An important role in determination of the stability of cementing solutions is played by the processes of high-temperature corrosion and thermal stability, which can lead to embrittlement of hardened cementing solutions, which can lead to emergency situations when external restraining pressure can damage steel pipes by deformation and fracture.^[6-8] In most cases, both factors (strength and heat resistance) play an important role in determining the type of cementing solution, as well as the area of its application in well stabilization, especially in hot climates, as well as at great depths of oil and gas bearing formations.^[9-11]

The methods proposed today for enhancement of the strength characteristics of cementing solutions by adding mineral additives to them, such as microsilica, ash, finely dispersed metal powders obtained from waste from metallurgical production, are among the most promising methods for increasing the resistance of cementing solutions to external influences, as well as improving physical and mechanical properties.^[12-15]

As a rule, adding small amounts of mineral additives to the composition of cementing solutions improves the adhesion of

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cement particles to each other, increasing the density and reducing the amount of voids, increasing wear resistance and crack resistance to various vibration modes that occur during long-term dynamic loads during operation at drilling sites, increasing resistance to mechanical impacts, as well as increasing adhesion and moisture resistance, which reduces the rate of corrosion during long-term exposure to salts and chemicals.^[16,17] At the same time, the choice of stabilizing strengthening additives is usually based on such factors as the cost of their production, which has a great influence on the pricing of oil production, the availability of materials in large volumes, as well as a number of structural factors that determine the effectiveness of strengthening due to structural and dimensional features.^[18-20]

It should be noted that in recent years, much attention in this area has been paid to considering the possibilities of using metallurgical production waste, which is a powder fraction consisting of iron oxides or iron-containing particles, the cost of which is minimal due to the fact that they are production waste.^[21,22] Thus, the use of secondary raw materials, including metallurgical waste in the form of iron-containing sludge, can not only reduce the costs associated with the use of various stabilizing additives and plasticizers, the addition of which is necessary to improve strength properties, but also increase the stability of materials due to size effects, the achievement of which is achieved by chemical reduction and crushing of sludge during preparation. The use of metal oxides, including nanostructured powders in the form of iron oxide, has been studied quite intensively recently, in view of the possibility of increasing the stability of cementing solutions, as well as the formation of a reinforcement effect due to the size and structural features of oxide powders used as modification materials. For example, in Refs. [23,24], the prospects of using oxide powders as modifiers for increasing the strength properties of cement stone were shown. The innovation and novelty of this research lies in the possibility of using metallurgical production products in the form of sludge consisting of iron oxide to increase the resistance to external influences and wear resistance of cementitious solutions.

In this case, adding finely dispersed additives to the composition of cementing solutions allows for increasing resistance to wear and increasing the strength of cement stone during its hardening, as well as expanding the potential for using metallurgical waste, which has fairly large reserves in the form of powders. In turn, the introduction of technologies for modifying cementing solutions due to the use of cheap materials in small quantities that improve mechanical properties and resistance to degradation, including temperature effects, will increase the efficiency of fixing oil wells, as well as reduce the risks of emergency situations that can lead to environmental disasters. The use of these iron-containing powders, subjected to minimal impact associated with fractionation, in order to sift out the coarse-grained fraction with subsequent separation of the finely dispersed or ultra-dispersed fraction, is one of the promising methods for

increasing resistance to mechanical impacts due to strengthening.^[25,26] It should be considered that a high content of stabilizing additives in the form of iron oxides can lead to a decrease in heat resistance, due to the oxidizing ability of iron oxide, which leads to a decrease in the stability of materials during long-term operation under high temperature conditions. In this regard, the search for optimal concentrations of adding stabilizing strengthening additives is one of the key factors requiring detailed study in the field of increasing the strength parameters of cementing solutions.

2. Materials and methods

The objects of the study were cementing mortar prepared from Portland cement PCT-I-50, iron ore concentrate (IOC-1) and acetal-alcohol stabilizer. This composition is used for cementing casing columns, which can be used under high temperatures and high reservoir pressures, which requires high strength (bending, compression), as well as heat resistance and resistance to thermal effects. A finely dispersed fraction of metallurgical sludge in the form of iron oxide powder - hematite, obtained by mechanical dispersion in order to separate the fine fraction from large particles, with subsequent separation of the finely dispersed fraction by size using an Analysette 3 disperser (Fritsch, Berlin, Germany) was used as a stabilizing strengthening additive. A detailed description of the production of finely dispersed hematite particles is presented in Ref. [27]. The concentration of the stabilizing strengthening additive was 0.5, 1.0 and 1.5 wt. %. The introduction of stabilizing additives in the form of finely dispersed iron oxide was carried out at the stage of mixing and preparing the cementing solution, by adding powders to the Portland cement PCT-I-50, followed by mixing it in order to uniformly distribute the stabilizing additive. The choice of iron oxide obtained from metallurgical waste as a component for modification is primarily based on the possibility of expanding promising areas of waste recycling, allowing not only to reduce existing waste stocks in production, which are increasing every year, but also to expand the possibilities of improving the mechanical properties of cementing solutions. At the same time, the proposed technologies for extracting iron oxide from metallurgical sludge, which are described in detail in Ref. [25], are not expensive, which in turn will not increase the cost of production of cementing solutions, and the effect of strengthening and increasing resistance to external influences will fully cover all costs. It should be noted that the choice of low concentrations of stabilizing additives is due to the fact that at high concentrations of iron oxide, as shown in Refs. [28,29], the degradation processes become more intense due to the oxidation of iron oxide, leading to the destabilization of the cement stone.

Figs. 1(a-b) show the results of morphological studies of the used stabilizing additives in the initial state, after they were extracted from metallurgical waste according to the procedure and after mechanochemical grinding,^[27] reflecting the influence of grinding on the size effects. Fig. 1c shows the

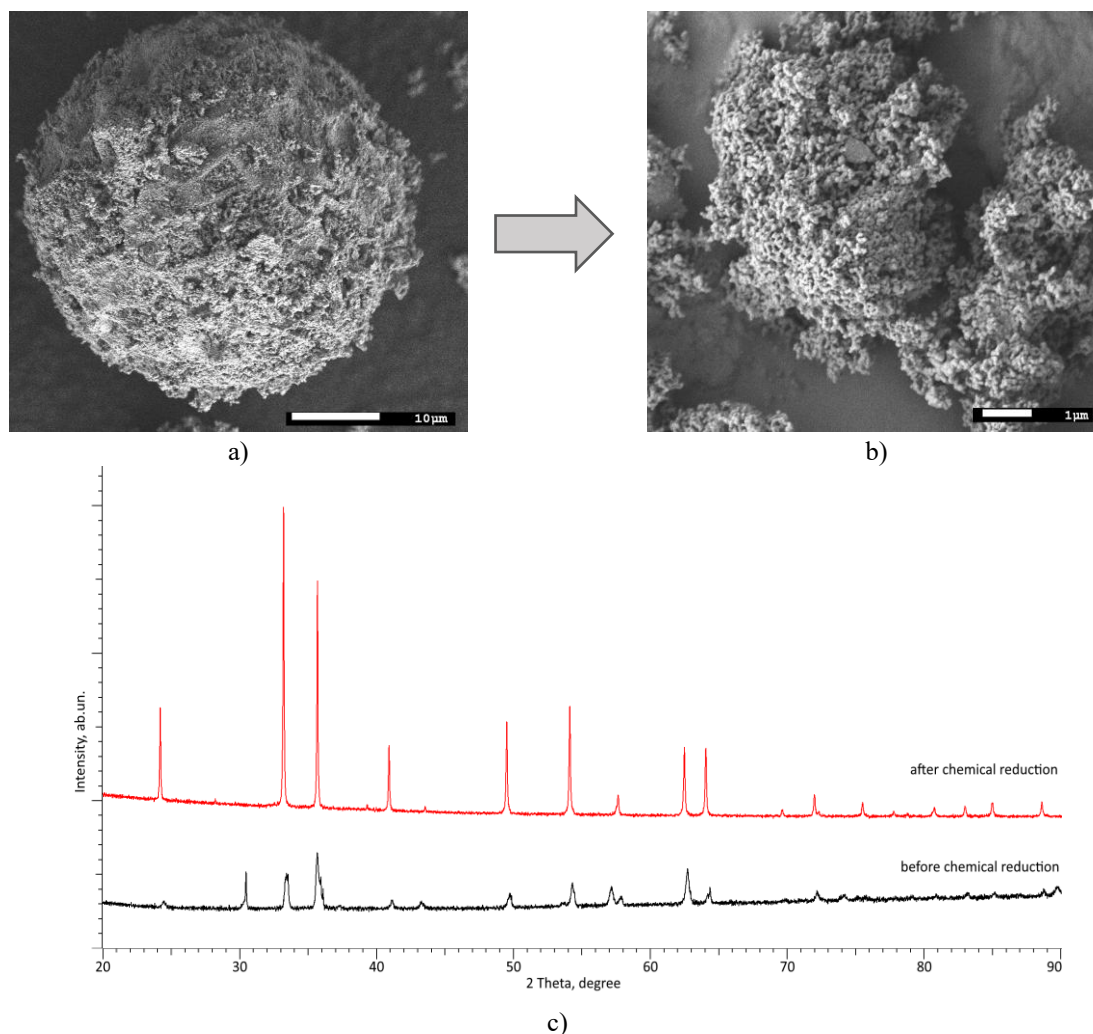


Fig. 1: a) SEM image of powders after chemical reduction; b) SEM image of powders after mechanochemical milling; c) Results of X-ray diffraction of the powders under study.

results of X-ray diffraction of the samples before and after treatment, reflecting the change in the phase composition of the used powders. In the case of powder samples without chemical treatment in the initial state, the iron oxide extracted from the metallurgical sludge is a mixture of two phases: maghemite (Fe_2O_3) (PDF-01-089-5894) and magnetite (Fe_3O_4) (PDF-01-071-6336). The established effect of phase transformations from $\text{Fe}_2\text{O}_3/\text{Fe}_3\text{O}_4 \rightarrow$ hematite Fe_2O_3 as a result of chemical reduction and heat treatment is a fairly well-known fact and has been studied in detail in a number of studies.^[30,31]

According to the data obtained, after chemical reduction, the studied samples are spherical particles of a fairly large size (about 30 - 50 μm) of hematite Fe_2O_3 (PDF-00-033-0664), the formation of which occurs as a result of chemical reduction and heat treatment (see the data of the method given in the work,^[27] as well as the results of the X-ray phase analysis shown in Fig. 1c). At the same time, as can be seen from the presented data, the mechanochemical effect leads to the crushing of large particles and the formation of a finely dispersed fraction, agglomerating into larger grains due to magnetic properties.

The effect of temperature on the strength of cementitious mortars during hardening at different temperatures was assessed by measuring the compressive strength at different holding times at different temperatures, which made it possible to determine the optimal hardening conditions, as well as the effect of varying the concentration of the stabilizing strengthening additive on the hardening rate, as well as permissible temperature conditions that allow increasing both the hardening rate and increasing the strength characteristics. The strength tests of the obtained cementitious mortars were assessed using standard methods for determining flexural strength and compressive strength. The measurements were carried out using a universal single-column electromechanical testing machine LFM-L 10kH (Walter + Bai AG, Lönigen, Switzerland). The tests were carried out using GOST 10180-2012 and GOST 18105-2018.

The determination of the dispersion of the stabilizing strengthening additive on the strength characteristics was assessed by measuring the values of the bending and compression strength with variations in the grain sizes of the stabilizing additive, the change in which was obtained by mechanical activation with the aim of crushing the finely

dispersed fraction to obtain an ultrafine fraction with grain sizes of about 300, 500 and 1000 nm. Mechanical activation was carried out using a PULVERISSETTE 6 planetary mill (Fritsch, Berlin, Germany), which contained a grinding cup filled one-third with 10 mm diameter grinding balls and one-third with ground powder of dispersed metallurgical sludge. Variation of the grinding rate in the range from 200 to 500 rpm with a grinding time of 30 minutes made it possible to obtain ultrafine hematite fractions with different grain sizes. The homogeneity of grain sizes after grinding was about 90 %.

Experiments on heat resistance and resistance to long-term thermal effects were conducted by long-term holding of cementing solutions at different temperatures in muffle furnaces. Holding was carried out for 1000 hours at temperatures of 100, 150, 200, 250 °C, after each 100 hours, measurements of strength characteristics were taken, based on the changes in which a conclusion was made about the resistance of cementing solutions to temperature effects and the possibility of using them in conditions of elevated temperatures. The choice of these temperature conditions for testing samples for thermal resistance was made taking into account the possibility of using cementing solutions at these temperatures. At the same time, this range of measurements, as well as the time intervals of tests, were chosen in order to simulate operating conditions as close as possible to real ones. The measurements were carried out on samples measuring

5×5×5 cm obtained by pouring cementing solutions into special press forms, which made it possible to produce samples of the same size and then test them for resistance to external influences. All experiments were carried out on serial samples, which made it possible to determine measurement errors and standard deviation values, as well as to establish the convergence and repeatability of the results of the experiments.

3. Results and discussion

Fig. 2 reveals the assessment results of changes in the compressive strength of the studied cementitious mortars depending on the heating temperature during the hardening process. The data are presented as dependencies of the change in the value of compressive strength on the test time for each temperature mode. In the case of the original cementitious mortar without adding a stabilizing strengthening additive, an increase in compressive strength is observed with an increase in the hardening time, which stabilizes in the range from 24 to 48 hours, with subsequent stable strength indicators. However, it should be noted that at temperatures above 110–120 °C, a slight decrease in hardness is observed for the initial cementing solutions, compared with the results obtained at temperatures of 100–110 °C, at which the strength change curve after 48 hours stabilizes and maintains its values for a fairly long time. Such clearly expressed changes in the decrease in strength at higher temperatures can be explained

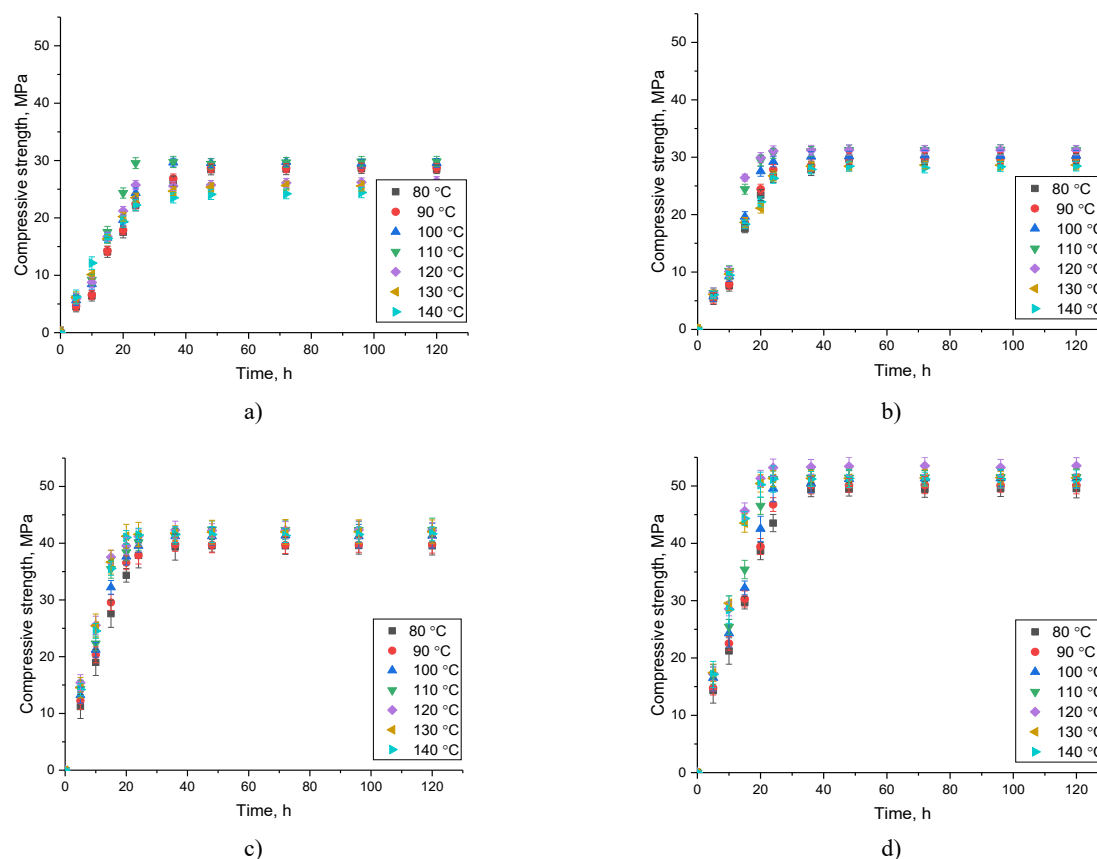


Fig. 2: Results of changes in the value of compressive strength of cementitious mortars depending on the exposure temperature, characterizing the hardening process: a) without the addition of a stabilizing strengthening additive, b) with the addition of a concentration of 0.5 w.%, c) with the addition of a concentration of 1.0 w.%, and d) with the addition of a concentration of 1.5 w.%.

by the structural transformations of hydrosilicates during the hydration process, with the formation of larger particles in the form of plates, the formation of which leads to a weakening of strength due to the low degree of bonding between themselves, as well as the formation of micropores in the structure.

According to the data obtained, the addition of a stabilizing strengthening additive to the cementing solution at concentrations of 0.5 wt.% leads to an increase in the value of compressive strength with a change in temperature during the hardening process (see Fig. 2b), while an increase in the temperature of the external effect leads to a more pronounced increase in compressive strength, as well as a shift in the change curves, indicating earlier hardening and strengthening of the cementing solutions. At the same time, the analysis of the strength characteristics at temperatures of external influences above 120 °C indicates a positive effect of adding a stabilizing additive on the hydration processes, as well as the formation of large particles restrained by the finely dispersed fraction of oxide particles of the stabilizing additive. An increase in the concentration of the stabilizing strengthening additive in the composition of the cementing mortar has a positive effect on the strengthening effect, which consists of an increase in the values of compressive strength from 28–29 MPa for the original non-stabilized cementing mortars to 42 – 51 MPa for cases where the concentration of the stabilizing strengthening additive is 1.0 w.% and 1.5 w.% (see Fig. 2c and Fig. 2d), respectively. Moreover, changes are observed not only in strength indicators but also in trends of strength stabilization over time with an increase in exposure temperature, which indicates positive dynamics of strengthening due to stabilizing additives.

Fig. 3 shows the results of a comparative analysis of changes in the value of compressive strength after 24, 48 and 120 hours of hardening, reflecting the kinetics of changes in strength, as well as the hardening rate with the acceleration of hydration processes, which are accompanied by changes in the shape and size of grains, which have a positive and negative effect on hardening. As can be seen from the data presented, the optimal conditions for hardening of cementing solutions

without stabilizing additives are temperature conditions of 100–110 °C, the use of which allows obtaining high-strength compositions, however, an increase in the exposure temperature leads to a decrease in strength parameters due to the structural features of the grains during hardening and subsequent hydration. Addition of stabilizing additives to the composition of cementing solutions makes it possible to increase the strength, as well as stabilize them at higher temperatures, as well as the possibility of operation at elevated temperatures. The change in strength characteristics, in turn, indicates a positive effect of stabilizing strengthening additives on the increase in strength, caused by more pronounced processes of hydration and compaction of the cementing solution, which in turn restrains the processes of softening at higher temperatures of exposure. Fig. 4 shows the evaluation results of the tests of the studied cementing solutions for resistance to thermal effects over a long period of time, typical for operating modes at high temperatures, as well as modeling the processes of thermal aging. Simulation of the thermal aging processes was carried out using muffle furnaces at various temperatures, the range of which was determined on the basis of operating modes in oil production. The general appearance of the data presented indicates the degradation of cement mortars with an increase in test time and temperature, from which it follows that the processes of degradation and embrittlement have clearly expressed temperature and time dependencies, and the degradation rate is most pronounced at high temperatures of 200–250 °C. At the same time, in the case of using stabilizing strengthening additives in the form of finely dispersed hematite particles at concentrations of 0.5 w.% and 1.0 w.% (see Fig. 4b and Fig. 4c), a positive dynamic of increasing the stability of strength characteristics to thermally induced corrosion processes is observed, while at a concentration of stabilizing strengthening additive of 1.5 w.% in the case of temperatures of 200 – 250 °C (see Fig. 2d), a sharp deterioration in the stability of strength properties is observed, which can be explained by the processes of corrosion of finely dispersed hematite particles in the structure of the cementing solution, as a result of which a

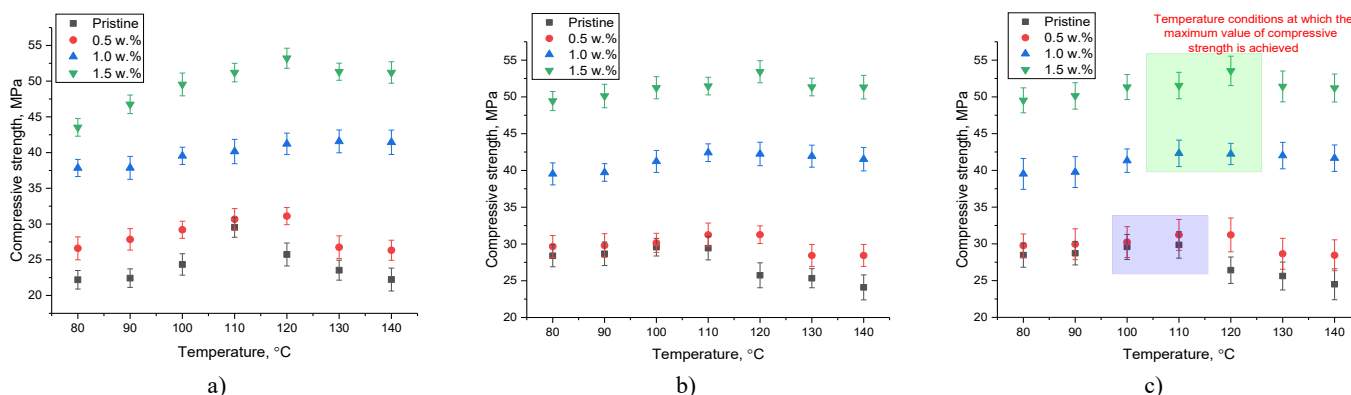


Fig. 3: Results of changes in the compressive strength of cementitious mortars after (a) 24, (b) 48 and (c) 120 hours of hardening (the highlighted areas indicate the observed changes in solidification temperature, the change of which indicates the influence of the addition of stabilizing additives).

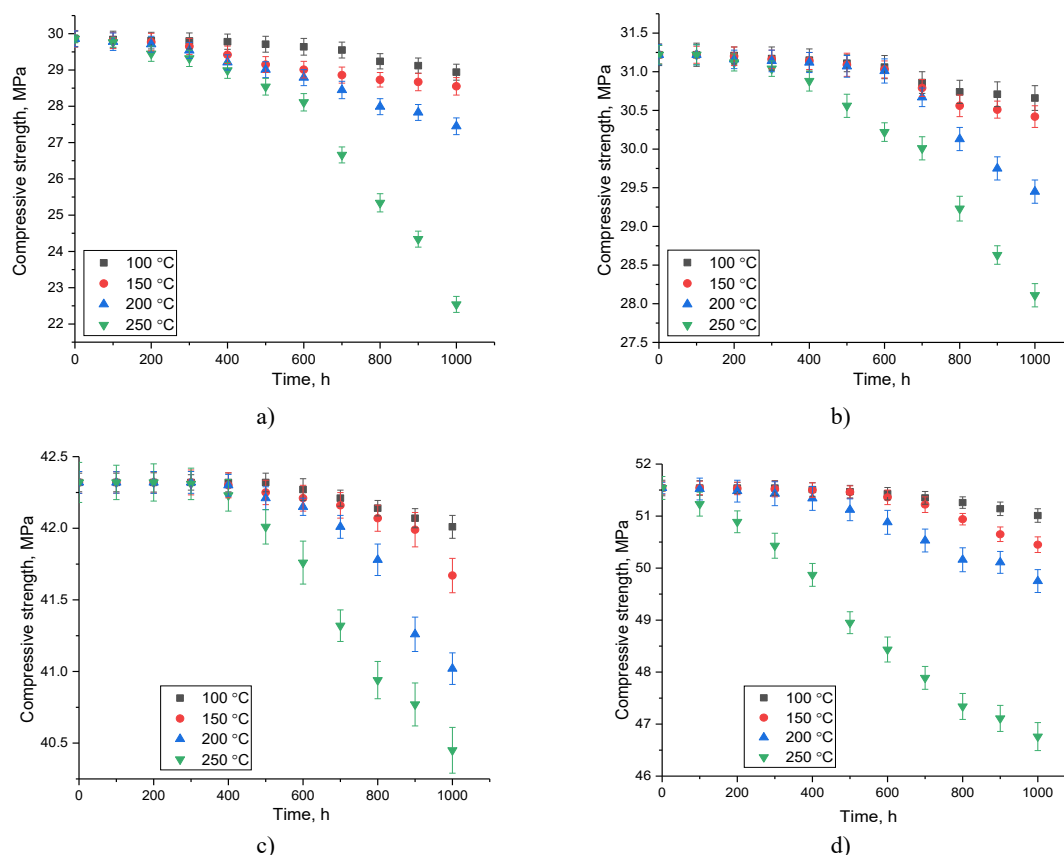


Fig. 4: Assessment results of the stability of cementing mortars during determination of their wear resistance in the case of thermal effects at different temperatures over long periods of time, typical for operating modes: a) without the addition of a stabilizing strengthening additive, b) with the addition of a concentration of 0.5 w.%, c) with the addition of a concentration of 1.0 w.%, and d) with the addition of a concentration of 1.5 w.%.

destructive decrease in strength occurs due to oxides.

Changing the grain sizes of the stabilizing strengthening additive through the use of mechanical activation processes allows increasing the strength characteristics of cementing mortars, as evidenced by the results shown in Fig. 5. Moreover, the dispersion strengthening effect observed in the obtained dependences has a clearly expressed exponential dependence on the grain

sizes, for which a decrease in grain sizes less than 300 nm can lead to significant strengthening effects. The most pronounced changes in strength characteristics are observed with a decrease in grain size to less than 1 μm , which allows us to judge the formation of a dispersion strengthening effect in the structure, associated with the fact that small particle sizes allow filling voids, thereby preventing deformation effects on the structure under mechanical action, as well as increasing the

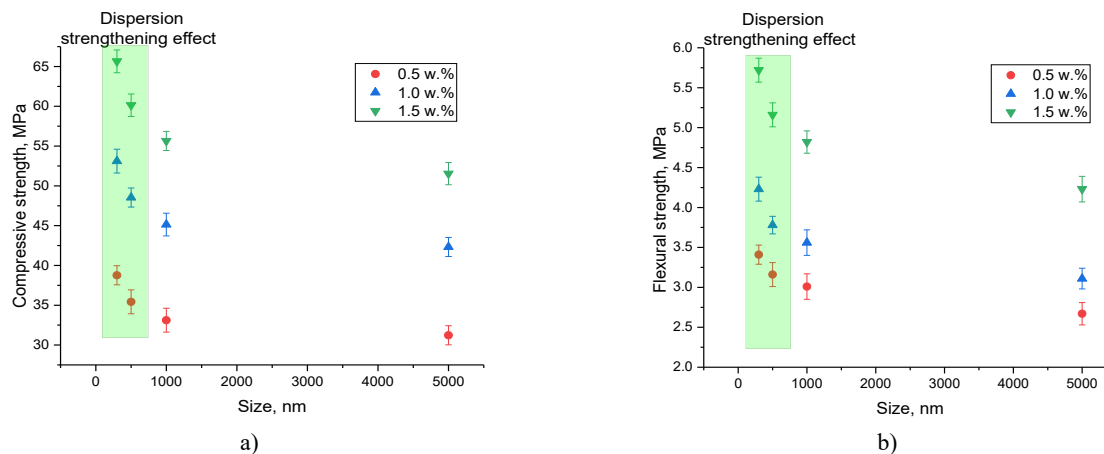


Fig. 5: Assessment results of the change in strength characteristics – compressive strength (a) and bending strength (b) in the case of variation in the grain size of the stabilizing strengthening additive used to modify cementitious mortars (the highlighted areas indicate the greatest strengthening effect associated with the dimensional factors of the stabilizing additives used).

crack resistance of cementing solutions. It should be noted that a decrease in grain size allows for a strengthening effect comparable to the effect of increasing the concentration of a stabilizing strengthening additive in the composition of cementing solutions, which in turn allows for a decrease in its concentration, an increase in which, as was shown in the analysis of the thermal stability of ceramics, plays a negative role in reducing strength characteristics at high temperatures, at which corrosion processes are initiated.

Analyzing the data obtained, it can be concluded that the stabilization of cementing solutions by adding finely dispersed hematite particles to them, the addition of which leads to an increase in strength characteristics by more than 30-70 % compared to the original values, as well as an increase in wear resistance at temperatures of 100-150 °C by more than 1.5-2.5 times compared to unstabilized cementing solutions. Enhancement of the strength characteristics, as well as resistance to thermal aging and wear resistance during long-term thermal exposure of cementing solutions due to the use of stabilizing strengthening additives in the form of finely dispersed particles of hematite, which are waste from metallurgical production, makes it possible to increase the service life of cementing solutions, as well as increase their stability to external influences that arise during operation.

It should be noted that the use of a low concentration of a stabilizing additive in the form of hematite particles makes it possible to increase the wear resistance of cementing mortars by more than 2 times compared to unstabilized mortars, which together makes it possible to increase the service life, and also to consider these cementing mortars with the possibility of their operation at higher temperatures without the risk of strength loss. The obtained results of changing the strengthening efficiency of cementing mortars due to the addition of finely dispersed hematite particles to them are in good agreement with the results of Refs. [32,33]. It should be noted that the proposed technological solution for modifying and strengthening the cementitious mortar by adding a recycled metallurgical product to it shows higher efficiency in strengthening than magnetite particles.[34] The results of the influence of size factors on the change in resistance to external mechanical influences and temperature heating are in good agreement with the results of Agnieszka *et al.*[35]

4. Conclusion

In conclusion, the following results of the conducted studies can be summarized, reflecting the prospects of using finely dispersed hematite particles as stabilizing strengthening additives to increase the strength and wear resistance of cementing solutions. It has been established that the addition of a stabilizing strengthening additive in the form of iron oxide leads to a decrease in the hardening time and stabilization of strength characteristics at higher temperature conditions, while for non-stabilized cementitious mortars, an increase in the heating temperature during hardening above 100 °C leads to an acceleration of the hardening processes, but at the same

time the compressive strength decreases, which indicates destabilization of the structure of the mortar due to the acceleration of hydration processes at high temperatures. At the same time, an increase in the concentration of finely dispersed hematite particles from 0.5 to 1.0-1.5 wt.% in the composition of the cementing solution leads to an increase in strength by more than 1.5 times compared to unstabilized cementing solutions, which in turn makes it possible to increase their resistance to external loads under mechanical impacts or dynamic vibration effects characteristic of operating modes. At the same time, an increase in thermal stability under long-term temperature exposure allows us to conclude that it is possible to increase wear resistance and resistance to corrosion processes associated with the use of cementing solutions.

Summarizing the conducted research, it can be concluded that the use of ultrafine particles of hematite (iron oxide obtained from metallurgical waste) allows to significantly increase the resistance of cementing solutions to wear and increase the solidification temperature, which allows using them in extreme conditions, as well as in the case of large external mechanical loads. Further plans in this direction will be aimed at developing the scalability of the proposed technology, taking into account industrial capacities, as well as determining not only wear resistance, but also resistance to the processes of interaction of cementing solutions with aggressive environments during their operation.

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Conflict of Interest

There is no conflict of interest.

Supporting Information

Not applicable.

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