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Peculiarities of hydration and frost resistance of cement with natural zeolite additive

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Abstract. The work is directed to the researches of composite binders, especially their strength properties in conditions of negative temperatures. Since the slowing down of concrete hardening with the addition of pozzolanic additives limits the possibility of using composite binders in regions with a short period of positive temperatures, a whole area of scientific works fails to find its application. The article presents the results on the determination of strength properties and hydration processes in cement stone with pozzolanic additive from natural zeolite during freezing and thawing. In determining the strength of the cement stone we chose the method of ultrasonic penetration. Graduation dependence of strength on the time of ultrasound propagation was revealed for its correct application. It allowed us to obtain dependences of cement stone strength with different amounts of additives on cycles of alternate freezing and thawing. The obtained results made it possible to get information on frost resistance of different compositions of the binders. It was shown that the addition of 30 wt. % and more of ground natural zeolite into Portland cement slows down the destructive effect of freezing-thawing due to unfinished hydration processes, which leads to an increase in the critical number of freezing-thawing cycles and the Portland cement frost-resistance grade from F300 to F400, respectively.

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1. *Introduction*

All over the world, there is a lot of experience in the use of composite binders, including cements with various mineral additives. However, their effective use in construction in cold climates is severely limited by temperature parameters, forcing to increase the cost of construction works by resorting to various additional technologies to control the curing conditions [1–3], while there are no attempts to increase the hydration capacity of binders in cyclic freezing and thawing. Thus, research is needed to improve the frost resistance of concrete binders and to expand the temperature conditions of their hardening.

It is a known fact that a low positive temperature slows down the process of setting and gaining strength of concrete, and then the negative temperature stops it completely. At negative temperatures, it is impossible to perform concrete work, because glaciated water at 0 °C is not able to bind with the cement. Thus, the hydration processes in concrete responsible for strength gain are stopped [4–7]. For example, at an ambient temperature of –15 °C, even taking into account the preheating of the concrete mixture to 60 °C, in less than 60 hours the temperature of 200 mm thick concrete drops below zero throughout its entire thickness [8].

Because of this fact, the organization of construction work is carried out mainly in the period of positive temperatures, which, depending on the region of construction can be quite short. In this case, a well-known fact that Portland cement reaches natural strength within 28 days after mixing with water, which introduces its own limitations on the duration of concrete work, which should be carried out before the onset of sub-zero temperatures. In this regard, it becomes undesirable to use such new developments as mixed cements, which due to their pozzolanic properties slow down the process of setting the strength of concrete [9–12].

Thus, the retardation of hydration processes, typical for some composite binders, limits the possibility of their use in conditions of rapid onset of negative temperatures. Because of this fact in regions with a short period of positive temperatures the whole area of scientific developments dedicated to composite binders is not practically applied [13–20]. On this basis, studies that study the behavior of composite binders, namely their pozzolanic and hydration properties in conditions of negative temperatures are relevant.

The aim of the research is to establish the features of hardening processes and frost resistance of Portland cement with natural zeolite additive. As the first step to achieve this goal, the task of studying the process of changing the strength of cement stone during freezing-thawing cycles has been set. As a method of determining the strength is used ultrasonic screening method.

2. Materials and Methods

Portland cement of CEM I 32.5H grade produced by Yakutcement JSC (Mokhsogolokh, Russia) was used for the tests (Table 1). Ground natural zeolite produced by Suntarzeolite LLC (Suntar, Russia), an enterprise producing and enriching natural zeolite, was used as a pozzolanic additive. Properties of the additive are given in Table 2.

Table 1. Properties of Portland cement CEM I 32.5H (JSC "Yakutcement").

Indicator	Value
Normal density, %	25
Residue on sieve 008, %	7.0
Uniformity of volume change, mm	0.11
Timing of setting:	
– start, hr-min	2–35
– end, hour-minutes	3–55
Average activity of cement at the age of 28 days, MPa	40.95

Table 2. Properties of pozzolanic additive – ground natural zeolite.

Indicator	Value
ulk density, kg/m^3	1420
True density, kg/m^3	1780
Specific surface, m^2/kg	3000
Activity towards CaO, mg/g	76.6
Quality factor as a component of the composite binder*	1.16

Samples of Portland cement with pozzolanic additive in the amount of 10, 30 and 50 % (instead of cement by mass) were made in the form of cubes 70×70×70 mm without using large and small aggregates according to interstate standard GOST 10180-2012 Concretes. Methods for strength determination using reference specimens. The number of samples in the series was 3 pieces. All samples in the series were made from the same sample of the cement mixture, with the same molding and hardening conditions. After the end of hardening, all samples are tested at the same age.

The molding and compaction of cement mixtures into molds was carried out manually using baying. Forms are filled with cement mix in 3 layers no more than 30 mm. Each layer is sealed by baying with a steel rod 16 mm in diameter with a rounded end.

Samples intended for hardening under normal conditions, after manufacturing before they are stripped, are stored in molds covered with a damp cloth, which excludes the evaporation of moisture from them, in a room with an air temperature of (20±5) °C. After stripping, the samples are placed in a chamber with normal hardening conditions: temperature (20±2) °C and relative humidity (95±5) %. A schematic representation of the temperature regime of the medium of one cycle-freeze-thaw is shown in Fig. 1.

Before testing, the samples were kept for 4 hours at a temperature of (20±5) °C and a relative air humidity of at least 55 %.

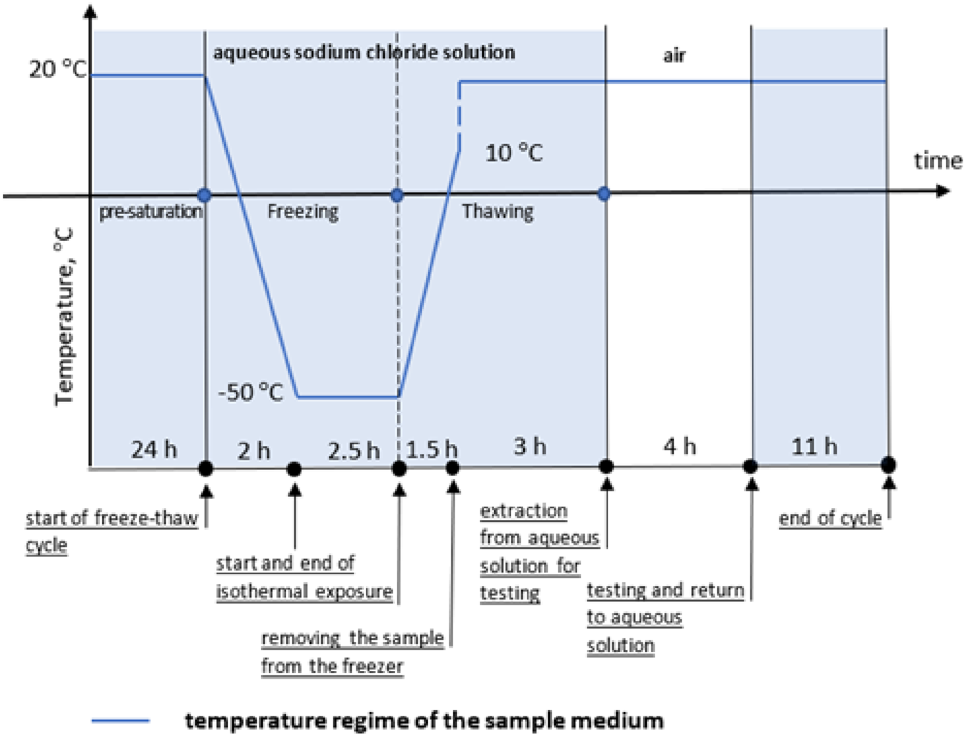

Figure 1. Schematic representation of the temperature regime of the medium of one cycle-freeze-thaw.

Ultrasonic measurement of the strength of the obtained cement stones were carried out with the device "Pulsar" according to the interstate standard GOST 17624-2012 Concrete. Ultrasonic method of strength determination.

Before testing specimens, calibration dependence "time of ultrasound spreading – strength" on the basis of specimen series of each binder composition was established. Graduation dependence was established according to formula 1 for specimens on day 28 of hardening.

$$R = a_1 \times t + a_0, \tag{1}$$

where, R is the strength of the sample according to the ultrasonic measurement, MPa; t is the time of ultrasound propagation according to the ultrasonic measurement, μs; a_1 and a_0 is the calibration coefficients determined according to formulas 2 and 3.

$$a_1 = \frac{\sum_{j=1}^{N}\left(\overline{R}_f - R_{jf}\right)\left(\overline{t} - t_j\right)}{\sum_{j=1}^{N}\left(\overline{t} - t_j\right)^2}; \tag{2}$$

$$a_0 = \overline{R}_f - a_1 \times \overline{t}, \tag{3}$$

where, N is the number of samples in the series; $\overline{R}_f$ is the average actual compressive strength of samples in N series of samples, MPa; $\overline{t}$ is the average time of ultrasound propagation in N series of samples, μs; R_{jf} and t_j is the single value of strength and ultrasound propagation time for j sample in the series.

Determination of frost resistance of cement stone samples was carried out in accordance with the interstate standard GOST 26134-2016 Concretes – Ultrasonic method of frost resistance determination.

The method for determining frost resistance is to determine the critical number of cycles of freezing and thawing (M) using the breakpoint (K) corresponding to a sharp increase in the spread of ultrasound after a certain number of cycles of freeze-thaw, determined on the logarithmic graph $(N-N_m)-(t-t_m)$, where t is the total value of ultrasound propagation time through 4 channels of one sample, t_m is the lowest value of the total ultrasound propagation time, $N,\ N_m$ is the corresponding freeze-thaw cycles. The breaking point is determined by the point of intersection of the linear dependence graphs of two groups of corresponding points – before and after a sharp increase in the ultrasonic propagation time. In this case, the very definition of the breaking point is carried out through a system of equations and substitution of one equation into another. Thus, critical number of freezing and thawing cycles will be determined according to formula (4).

$$M = N_m + K. \tag{4}$$

One cycle of freezing-thawing of samples corresponded to freezing in closed containers filled with 5 % aqueous sodium chloride solution at –50 °C for 2.5 hours and subsequent thawing for 2.5 hours at +20 °C, after which the ultrasound propagation time was measured.

3. Results and Discussion

Tests were carried out to determine physical and mechanical characteristics of cement stone samples based on Portland cement with pozzolanic additive in the amount of 10, 30 and 50 % (instead of cement by mass) in the form of 70×70×70 mm cubes (Table 3). The dosages were chosen based on the results of earlier studies on the rational content of mineral components in concretes and a review of the scientific literature [21–23]. Increase in addition of ground natural zeolite leads to reduction of average density of samples and water absorption index. Also, a decrease in the specific amount of Portland cement in the samples leads to a decrease in the strength of the samples at 28 days, which may be due to the fact that natural zeolite minerals show their pozzolanic activity in the late stages of hardening. This fact was also shown in the works of researchers from the Bulgarian Academy of Sciences, who found a decrease in the specific pore volume between samples with the addition of natural zeolite aged 28 days and 180 days, due to new formations that have arisen as a result of pozzolanic reactions [24]. In addition, Turkish researchers found, using EDX analysis, unreacted zeolite minerals in their mixed samples on the 28th day of hardening, and it is also said that a significant contribution of the pozzolanic effect to the strength of cement stones is achieved with large dosages of additives from natural zeolite [25].

Table 3. Physical and mechanical characteristics of cement stone samples of different compositions.

Binder composition	Average mass, kg	Average density, kg/m^3	Natural humidity, %	Water absorption,%	Average compressive strength at 28 days of age, MPa
Pure Portland cement	0.718	2093	2.7	4.4	29.1
Portland cement with the addition of 10% ground natural zeolite	0.692	2017	2.9	5.8	21.1
Portland cement with the addition of 30% ground natural zeolite	0.646	1883	3.6	7.7	26.8
Portland cement with the addition of 50% ground natural zeolite	0.643	1875	3.6	8.1	24.1

To establish the calibration dependence "ultrasound propagation time – strength" the average values of actual compressive strength and ultrasound propagation time of a preliminary series of samples from each composition were determined. Then calibration coefficients a1 and a0 were determined according to formulas 2 and 3. Because of small sample quantity and its description by linear dependence the value of approximation reliability is not high and makes 0.87.

Table 4. Establishment of the graduation dependence "time of ultrasound propagation – strength".

Binder composition	R_{jf}, MPa	t_j, µs	$\overline{R}_f$, MPa	$\bar{t}$, µs	a_1	a_0
Pure Portland cement	32.30	19.85	25.29	21.31	-2.99066109	89.01907188
	25.95	20.96				
Portland cement with the addition of 10% ground natural zeolite	21.99	22.32				
	26.27	22.10				
Portland cement with the addition of 30% ground natural zeolite	31.18	19.53				
	22.37	19.99				
Portland cement with the addition of 50% ground natural zeolite	21.49	22.67				
	20.79	23.04				

After the graduation dependence has been established, 13 cycles of freezing and thawing of samples of each composition have been performed, after each of which the mean values of parallel tests of ultrasound propagation time have been determined (Table 5, Fig. 2) and strength characteristics have been calculated by formula 1 (Table 6, Fig. 3). In spite of the low reliability of the certain dependence aproximation, the strength of the cured pure Portland cement stone before freezing is quite consistent with the declared strength of CEM I 32.5H. Also, immediately after the first cycle of freezing-thawing the strength of Portland cement without the addition of ground natural zeolite begins to decrease, indicating that the images have reached their maximum strength and undergo destruction in the process of freezing-thawing. When ground natural zeolite is added to Portland cement the strength of obtained cement stone keeps growing during freeze-thaw cycles. Thus, after addition of 10 % of ground natural zeolite after the 1st cycle the strength of obtained cement stone increases by 4 %, and after the 3rd cycle it decreases by 1 % after every 2nd cycle of freezing-thawing. When ground natural zeolite is added 30 % and 50 % to Portland cement, the peak strength of cement stone samples is achieved after the 3rd cycle of freeze-thawing and amounts to 113 % of 28 day strength, and the decrease of strength after the 5th cycle occurs more intensively – about 4 % every 2 cycles. Thus, delayed strength growth of binders with the addition of natural zeolite makes it possible to increase the strength of cement stone damaged by freezing and thawing cycles. Not the last role in the continuation of the strength gain of the samples is assigned to the conditions of freezing-thawing of the samples. Freezing and thawing does not occur instantly in containers with an aqueous solution of sodium chloride, and during positive temperatures of the set mode (Fig. 1), incomplete pozzolanic reactions continue, provided that ground natural zeolite is added. Based on the above, the conclusions of the work of the authors from Southeast University [26] are supplemented, on the possibility of standard curing of concretes damaged by freeze-thaw cycles at an early age, in our case, standard curing continues in the later stages of hardening, provided that the cement contains a pozzolanic additive.

Table 5. Time of ultrasound propagation through cement stone of different composition depending on freezing and thawing cycles.

Number of freeze-thaw cycles	0	1	3	5	7	9	11	13
Pure Portland cement	18.82	18.96	19.04	19.05	19.21	19.31	19.43	19.53
Portland cement with the addition of 10% ground natural zeolite	19.75	19.34	19.43	19.51	19.82	19.91	20.10	20.19
Portland cement with the addition of 30% ground natural zeolite	21.66	21.31	20.57	21.15	21.36	21.69	21.85	22.32
Portland cement with the addition of 50% ground natural zeolite	21.93	21.07	20.83	21.33	21.52	21.76	21.91	22.26

Table 6. Strength of cement stone of different compositions as a function of freezing and thawing cycles.

Number of freeze-thaw cycles	0	1	3	5	7	9	11	13
Pure Portland cement	32.74	32.32	32.08	32.04	31.57	31.27	30.90	30.60
Portland cement with the addition of 10% ground natural zeolite	29.94	31.18	30.90	30.66	29.74	29.48	28.91	28.64
Portland cement with the addition of 30% ground natural zeolite	24.23	25.28	27.49	25.78	25.13	24.15	23.67	22.27
Portland cement with the addition of 50% ground natural zeolite	23.44	26.00	26.71	25.23	24.66	23.95	23.50	22.45

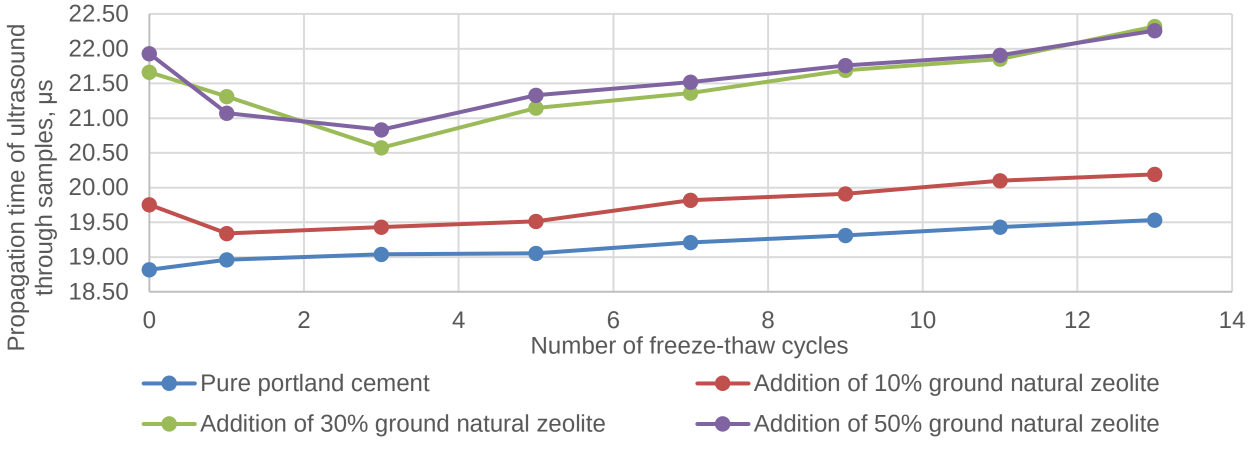

Figure 2. Time of ultrasound propagation through cement stone of different compositions as a function of freezing and thawing cycles.

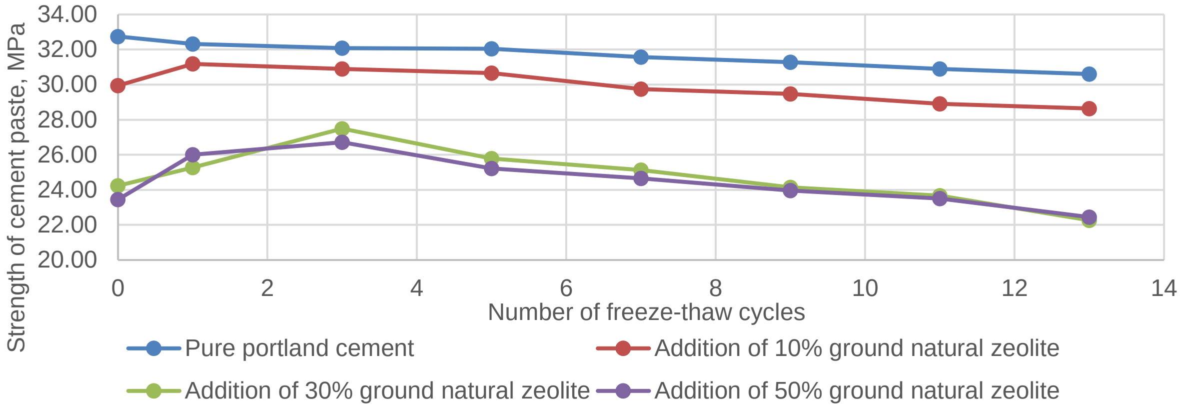

Figure 3: Strength of cement stone of different compositions as a function of freezing and thawing cycles.

In order to determine frost resistance of cement stone samples of each binder composition according to the interstate standard GOST 26134-2016, on logarithmic diagrams $\left(N-N_m\right)-\left(t-t_m\right)$ the changes of average total values of ultrasound spreading time through 4 channels of samples sounding have been plotted versus the minimum fixed value taken as tm and corresponding to it freeze-thaw cycles value – N_m (Fig. 4–7). The obtained points are divided into two groups of corresponding points – before and after sharp increase of ultrasound propagation time, linear dependences for each group of points are found by regression method.

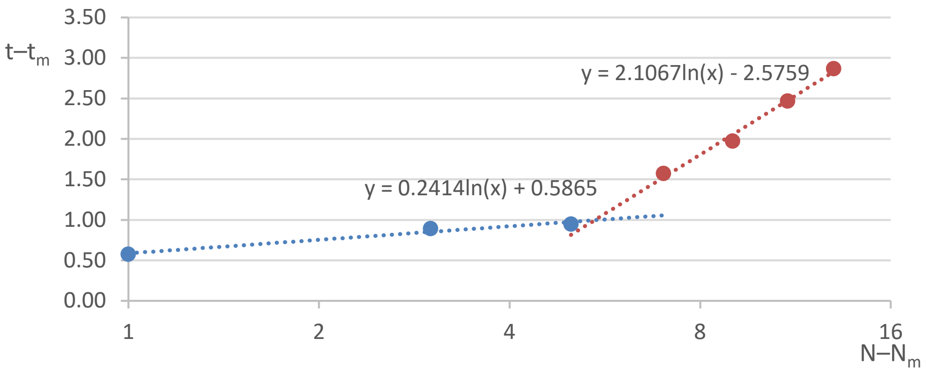

Figure 4: Graph of ultrasonic measurements of pure Portland cement.

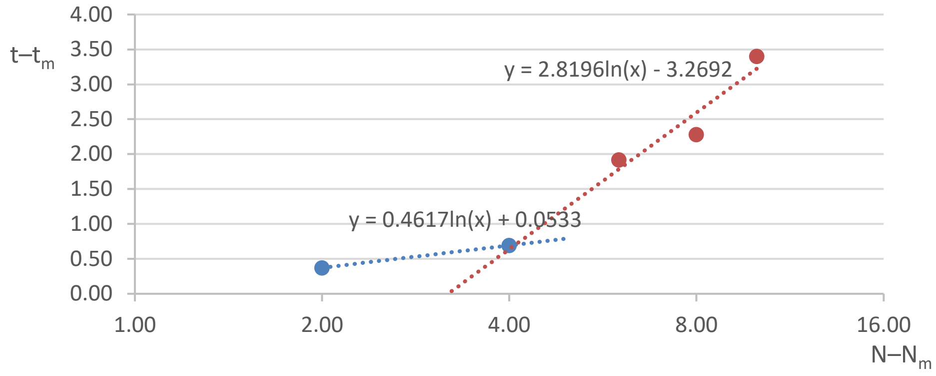

Figure 5. Graph of ultrasonic measurements of Portland cement with the addition of 10 % ground natural zeolite.

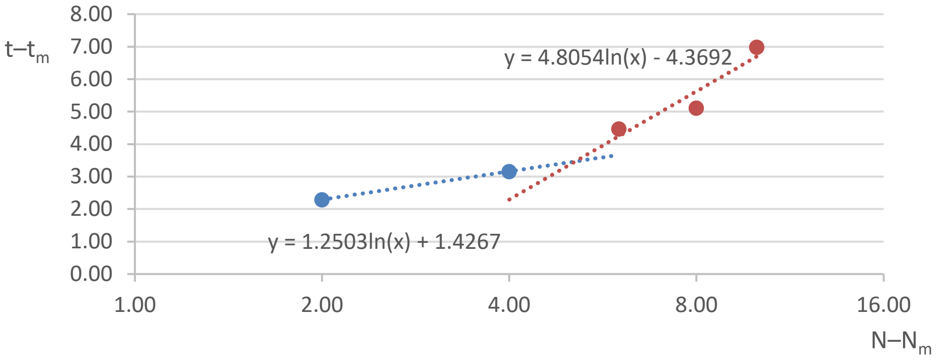

Figure 6: Graph of ultrasonic measurements of Portland cement with the addition of 30 % ground natural zeolite.

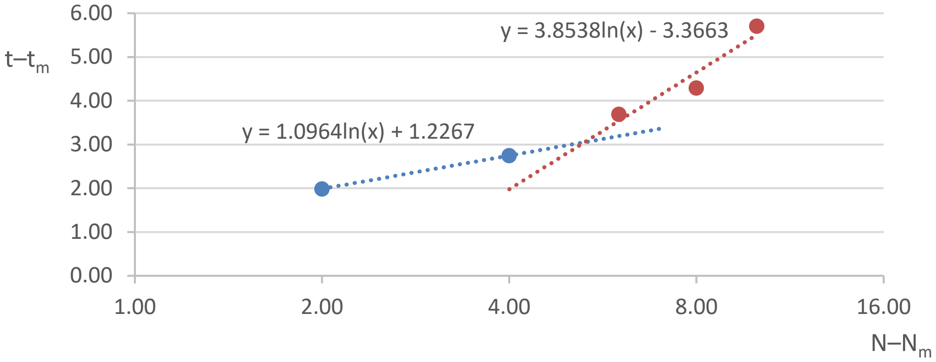

Figure 7. Graph of ultrasonic measurements of Portland cement with the addition of 30 % ground natural zeolite.

The given equations are used for determining breaking points (K), values of critical number of freezing-thawing cycles (M), after which there is a sharp increase of ultrasound spreading time in the sample and corresponding index of frost-resistance grade according to GOST 26134-2016 for each binder composition (Table 7). The addition of 30 % or more of ground natural zeolite into Portland cement results in increase of critical number of freeze-thaw cycles, due to which freeze-thaw resistance grade of cement increases from $F_1$300 to $F_1$400. Adding a smaller amount of additive does not change the critical number of freeze-thaw cycles. Thus, the introduction of pozzolanic additive in the form of natural zeolite can increase the long-term strength of the cement stone, which leads to an increase in frost resistance. Thus, the introduction of a pozzolanic additive in the form of natural zeolite makes it possible to increase the long-term strength of the cement stone, which leads to an increase in frost resistance, which is consistent with the studies of Chinese scientists, who also noted that frost resistance may not depend on the degree of concrete strength [27].

Table 7. Data on the frost resistance of cement stone of different compositions.

Number of freeze-thaw cycles	t_m	N_m	K	M	Mark frost resistance
Pure Portland cement	75.27	0	5.45	5	$F_1$300
Portland cement with the addition of 10% ground natural zeolite	77.36	1	4.09	5	$F_1$300
Portland cement with the addition of 30% ground natural zeolite	82.29	3	5.11	8	$F_1$400
Portland cement with the addition of 50% ground natural zeolite	83.33	3	5.28	8	$F_1$400

4. Conclusion

Thus, the process of changing the strength of cement stone during freeze-thaw cycles has been studied on the basis of which the following was established:

1. Graduation dependence "time of ultrasound spreading - strength" with reliability of approximation 0.87 has been revealed. This allowed us to obtain dependences of cement-stone samples durability with different amount of additives on cycles of alternate freezing and thawing.

2. A delayed growth of the strength of binders with natural zeolite addition allows you to continue to gain strength of cement stonedamaged by cycles of freezing and thawing. Thus, if pure cement is characterized by strength reduction after the first cycle, then in case of addition of ground natural zeolite the strength keeps growing in spite of being exposed to freezing-thawing cycles. The addition of 10 % of ground natural zeolite causes strength to decrease after the 3rd cycle, and 30 % and 50 % after the 5th cycle.

3. The increase of long term strength of cement stone by adding natural zeolite, results in increase of frost resistance. The addition of 30 % or more of ground natural zeolite into Portland cement results in increase of critical number of freeze-thaw cycles, due to which increase of cement stone frost-resistance grade from $F_1$300 to $F_1$400 is possible. Adding a smaller amount of additive does not change the critical number of freeze-thaw cycles. This fact is associated with longer processes of pozzolanic reactions that go beyond the generally accepted cement hydration rate of 28 days. Moreover, a larger amount of natural zeolite makes it possible to maintain the strength growth of the damaged cement stone with a large number of freeze-thaw cycles.

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