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PROPERTIES OF MONOLITHIC CEMENT CONCRETE, HARDENING AT TEMPERATURES CLOSE TO ZERO

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One of the main problems of hardening monolithic cement concrete, especially in the conditions of restoration and reconstruction of various objects, is the hardening of concrete in conditions of negative temperatures. Winter concreting technologies are divided into two main methods: the thermos method, in which the temperature of the environment in which the concrete hardens, must be above zero degrees Celsius, or the use of antifreeze additives. In the last case, it is advisable to introduce antifreeze chemical additives into the concrete composition, which lower the freezing temperature of water in concrete. However, previously conducted studies are known that show that even in the case of early freezing of concrete, its quality remains high. In this case, it is necessary that the beginning of setting of the cement paste occurs before the concrete can freeze. In addition, it has been suggested that concrete hardening at low temperatures can occur without using the thermos method. The article presents the results of the influence of hardening temperature on the setting time of cement paste. It is shown how hardening conditions affect on the strength and frost resistance of concrete.

Keywords: beginning of setting (BS), end of setting (ES), setting time (ST), strength, antifreeze chemical admixture, frost resistance.

Introduction

The flow of goods transported by worldwide road system increases every year. Loads on vehicle axles and road surfaces increase. Road transportation is becoming more and more energy-intensive, which leads to the need to develop modern programs and equipment that make the road more autonomous in energy production and consumption (Gnatov, Argun & Rudenko,2017). In these conditions, an important task, especially for Ukraine, is to expand the network of roads paved with cement concrete. This can be achieved by increasing the duration of the construction season and building in conditions of negative temperatures. Usually, very energy-intensive methods are used for this, in which artificial conditions of positive temperatures are created for the hardening concrete. This greatly increases the cost of construction. An important task in the construction of concrete roads remains to ensure their strength and durability, which depend from the conditions in which the concrete hardens. It is traditionally believed that high performance properties are ensured if the effect of negative temperatures on concrete occurs no earlier than the concrete structure acquires critical strength. If this does not happen, this will lead to a decrease not only in strength and frost resistance, but also in other properties of concrete. It is believed that concrete can obtain the necessary critical strength only when hardening under positive temperatures. At the same time, it is known that when antifreeze additives are introduced into concrete, concrete hardening can occur at temperatures below zero degrees (Shpynova & dr., 1985, Usherov-Marshak et al., 2002, Rixom et al., 1978). As for ensuring critical strength, research shows that the most effective technique is to accelerate the strength gain of concrete in the first day of hardening using heat (Brzozowski, Horszczaruk & Hrabiuk, 2017).

Long-term studies conducted in different countries in temperate climates such as Northern Europe show that freezing and thawing has a destructive effect on concrete, especially if it is not frost-resistant (Müller, Djuric & Haist, 2018). This is well known from intensive research into the frost resistance of concrete carried out in recent decades. However, despite the fact that knowledge about the mechanisms of frost destruction is constantly improving, some key aspects associated with their development over time have not yet been sufficiently studied. It is quite difficult to simulate the process of concrete destruction during freezing and thawing. It is necessary to take into account the influence of concrete strength on this process, the pore structure of the hardened cement paste and concrete as a whole, as well as the time during which changes can occur in concrete. These changes are caused by various reasons, which still does not allow predicting the behavior of concrete under long-term cyclic impacts. The only generally accepted reason for the destruction of concrete due to the action of freezing-thawing can be considered the formation of cracks in the microstructure and mesostructure of concrete.

In the last century, T. Powers showed that the cause of cracking is the water pressure during the ice formation process, during which its volume increases by about 9 %, as well as the direct pressure of the ice. Given that this occurs in the pores and capillaries of concrete, unless sufficient space for expansion is provided, cracking is inevitable. However, as numerous studies have shown, even with the formation of a microcrack, further destruction of concrete may not occur. To do this, it is necessary that the pore system has sufficient space for expansion. This theory explains that there is no direct correlation between the strength of concrete and its frost resistance and corrosion resistance. Therefore, air-entraining additives are introduced into road and airfield concrete to increase the number of air micropores.

The work (Müller et al., 2018 also provides data that allows us to conclude that during freezing-thawing, water is redistributed from larger pores to smaller ones. Research has shown that the maximum degree of pore saturation with water, which is equal to S = 1.0, is achieved both before the start of the test cycles and after 2 freeze-thaw cycles. During further tests, frost absorption of water into the pores occurs and it accelerates as the negative temperature decreases. This can be explained by the fact that microcracks can form in the concrete at the beginning of the tests, into which water or an aqueous solution enters.

The process of frost degradation is accelerated by the simultaneous action of salts, which are used on roads in winter (Oguchi & Yu, 2021). It is known that the destructive effect of negative temperatures is caused not only by ice pressure, but also by water pressure during ice formation. However, other authors have shown that the pressure due to the formation of ice in a closed pore space will decrease over time, since under the influence of pressure the temperature of the phase transition of water into ice shifts towards lower values (Ioannidou, Del Gado, Ulm & Pellenq, 2017). This suggests that structure formation in concrete can continue during freeze-thaw cycles. This is confirmed by studies that show that the amount of water in concrete at a distance of 2000 microns from the surface or more is quite large and can decrease after 56 freeze-thaw cycles (Kind et al., 2023).

Research by I.A. Kireenko (Kireenko,1919), which were carried out at the end of the 19th – beginning of the 20th century, showed that negative temperatures will not have an adverse effect on the final properties of concrete, even if it froze at an early stage of hardening. A necessary condition for this is that freezing of the concrete must occur either before the beginning of setting of the cement paste or after the end of setting. In this case, after thawing, the strength of concrete will be higher than the strength of concrete that hardened under normal conditions. Another important condition is that after defrosting, the concrete should not be re-frozen until the grade strength, or critical strength, is reached. At the end of the 19th century, under his leadership, several objects were built, including bridges, which were laid from concrete at temperatures down to –20 °C. At the beginning of the 20th century, summarizing the practical results of his work, he expressed a number of requirements for improving the quality of concrete that hardened at subzero temperatures. For example, concrete needs a reserve of heat to complete the setting of the cement, and antifreeze additives must be introduced into the concrete composition. He also suggested that the crystallization process can continue at temperatures down to – 12°C.

Our research did not allow us to establish a correlation between the strength of concrete and its frost resistance. At the same time, there is evidence that increasing the strength of concrete leads to an adequate increase in its durability (Zhang et al., 2020). These studies show that deterioration of class C20 concrete begins after 25 freeze-thaw cycles. Concrete classes C30 and higher can withstand up to 300 freeze-thaw cycles. The authors believe that the reason for this is a change in the size distribution of pores in the concrete structure. For example, the number of pores with a diameter of less than 20 nm in concretes of classes C50, C40, C30 and C20 was 89.13 %, 1.76 %, 1.11 % and 0.12 %, respectively. While the number of pores with a diameter of more than 200 nm in concretes of classes C50, C40, C30 and C20 was 4.3 2 %, 9.24 %, 7.65 % and 77.80 %, respectively. High-strength concrete had more small-sized pores, while low-strength concrete had more large-sized pores.

Materials and methods

The following materials were used in the studies: cements of the PC I 500 (CEM I – 42.5N) and PC I 400 (CEM I – 32.5N) brands; quartz sand of medium coarseness with fineness modulus Mfm = 2.0…2.2; crushed granite of fractions 5-10 mm, 10-20 mm and 20-40 mm. The strength grade of coarse aggregate was M1200; chemical additives: polycarboxylate type superplasticizer Sika 2508 HE (Sika Switzerland); a superplasticizer based on sulfonated melamine-formaldehyde resins FK88 (MC Bauchemie, Germany); air-entraining additive Lp 75 and antifreeze additive Frigidol (BASF, Germany).

The Frigidol additive was designed to lower the freezing point of water in concrete. An aqueous solution of the Frigidol additive with a concentration of 2 %, which was placed in a test tube with a diameter of 15 mm, froze at a temperature of – 12 °C, and in a test tube with a diameter of 6 mm at a temperature of – 18 °C. This confirmed the high antifreeze properties of this additive.

Studies of the properties of cement paste, strength and frost resistance of concrete were carried out in accordance with the testing standards that apply in Ukraine. The frost resistance of concrete cube samples measuring 70 x 70 x 70 mm was studied using the accelerated method at the age of 28 days. Before testing, the samples were soaked in a 5 % aqueous solution of sodium chloride for 4 days. After saturation, the samples were placed in a container with the same solution and kept at a temperature of – 50 ± 5 °C for 2 hours. Then the samples were placed in a container with a 5 % sodium chloride solution and defrosted for 2 hours at a temperature of +18 ± 2 °C. This constituted one test cycle. After 10, 20, 37 and 55 test cycles, the frost resistance coefficient of concrete, Kft, was determined. Kft is equal to the ratio of the compressive strength of concrete after a certain test cycles to the compressive strength of concrete before testing. 10, 20, 37 and 55 test cycles correspond to frost resistance grades F150, F200, F300. F400.

Results and Discussion

Considering the importance of knowledge about the process of cement setting, in the laboratory of the Department of Road Construction Materials (RCM) of the Kharkov National Automobile and Road University (KNARU), studies were carried out on the setting time of cement paste with the addition of polycarboxylate type superplasticizers Sika 2508 HE as well as based on sulfonated melamine-formaldehyde resins FK 88, and also without additives. During testing, cement paste samples were in different conditions (Table 1).

The results show that lowering the temperature of the environment at which setting of the cement paste occurs leads to an increase of the initial and final setting times. For cement paste of normal cosistency without additives, this elongation is 2...3 hours, or 50...60 %. The setting time of pastes with the addition of Sika 2508 increases by 23...40 %, and for pastes with the addition of FK 88 - by 20...32 %. The initial setting at the same water content in the paste without additives and in the paste with the addition of Sika 2508 increases by 33 %, and the final setting time by 23 %. In paste with the addition of FK88, the initial setting time increases by 33 %, and the final - by 24 %.

It is interesting that the setting time of cement pastes with superplasticizers, regardless of the mobility of the pastes and the temperature of the environment remains approximately the same. This makes it possible to determine the setting time of such pastes by testing at only one temperature. From a technological point of view, knowledge of the setting time makes it possible to determine the duration of thermos curing of hardening concrete at a temperature close to 0 °C.

Table 1

Setting times for cement paste under different conditions

#	Admixture	Ambient temperature	NC, %	Initial setting time	Final setting time	Setting time
1	-	+19 ºC	32.5	230 min	340 min	110 min
2	-	+5 ºC	32.5	370 min	510 min	140 min
3	Sika 2508 HE 0.8 %	+19 ºC	25.6	165 min	370 min	205 min
4	Sika 2508 HE 0.8 %	+5 ºC	25.6	230 min	455 min	225 min
5	FK 88 0.7 %	+19 ºC	28.0	110 min	365 min	255 min
6	FK 88 0.7 %	+5 ºC	28.0	145 min	435 min	290 min
7*	Sika 2508 HE 0.8 %	+19 ºC	32.5	300 min	530 min	230 min
8*	Sika 2508 HE 0.8 %	+5 ºC	32.5	400 min	650 min	250 min
9*	FK 88 0.7 %	+19 ºC	32.5	210 min	465 min	255 min
10*	FK 88 0.7 %	+5 ºC	32.5	280 min	575 min	295 min

Note: 1. The amount of mixing water in compositions 1, 7*, 8*, 9*, 10* was the same 2. Tests were carried out on PC I-400 (CEM I – 32.5 N) cement.

At the same time, it should be noted that the use of superplasticizers leads to a general increase in setting time at a temperature of + 19 °C by 2...2.5 times, and at a temperature of +5 °C by 1.6...2 times compared to cement paste without additives. This must also be considered when determining the duration of concrete curing.

Researchers note that after a short-term early freezing of concrete, if its further hardening took place under positive temperatures and sufficient environmental humidity, then the degree of hydration of cement in such concrete does not differ from cement hardening under normal conditions. The phase composition of new cement formations is also remains unchanged. Therefore, a decrease of the strength of concrete due to early freezing is most likely explained not by a change in the nature of physical and chemical processes, but by a change in the structure of concrete, for example, due to possible loosening of the structure (Collepardi, 2005).

In the period from 2009 to 2012, the international airport in the city of Kharkov was reconstructed. One of the sections of the airport with concrete pavement was laid at temperatures from + 6 °C to - 16 °C. Employees of the RCM department provided scientific support for the construction. At the same time, we studied the properties of concrete samples that hardened under the same conditions as the concrete of the site. In experiments, these samples hardened under real conditions, without using the thermos method.

When choosing a superplasticizer, it was found that the polycarboxylate additive is capable of involving an additional amount of air, which leads to instability of the properties of concrete and a decrease in its strength. Similar data were obtained later by other researchers (Łaźniewska-Piekarczyk, Miera & Szwabowski, 2017). Therefore, superplasticizer FK88 was chosen. Composition of the concrete mixture for pavements was as follows: cement – 380 kg/m^3, sand - 600 kg/m^3, granite 5-10 mm - 760 kg/m^3, granite10-20 mm - 500 kg/m^3.

Four concrete compositions were studied, which differed in the content of chemical additives. Composition 1 included the additives FK88 (0.6 wt. %) and Frigidol (1 wt. %). Composition 2 included the additives FK88 (0.6 wt. %) + Frigidol (1 wt. %) + Lp75 (0.1 wt.%). Composition 3 included the additive FK88 (0.6 wt. %). Composition 4 included the additives FK88 (0.6 wt. %) + Lp75 (0.1 wt. %).

Before testing, concrete of compositions 1 and 2 hardened for 45 days under construction site conditions. During the first 16 days, the temperature ranged from + 6 ºC to –7 ºC. The number of temperature transitions through 0°C was 8. Then the temperature was constantly negative and ranged from -6 °C to -16 °C. Concrete of compositions 3 and 4 hardened under normal conditions at a temperature of +18 ± 2 °C and a relative air humidity of 95 % in the laboratory.

Table 2

Compressive strength of concrete

# of mix	**Compressive strength, MPa at age, days**			
	3	**7**	**28**	**45**
1	4.5	8.0	22.5	46.0
2	3.0	7.0	20.0	42.0
3	19.5	30.0	39.0	43.0
4	15.0	25.0	35.0	38.5

It is obvious that the strength of concrete of compositions 1 and 2 is 3.5...5 times less than the strength of concrete of compositions 3 and 4 (table 2). However, with further hardening in conditions of negative temperatures, the difference in strength between concrete of compositions 1 and 2 and compositions 3 and 4 at the age of 28 days decreased to 1.7 times. And at the age of 45 days, concretes of compositions 1 and 2 had higher strengths than concretes of compositions 3 and 4.

Table 3

Bending strength of concrete

# of mix	**Bending strength, MPa at age, days**			
	3	**7**	**28**	**45**
1	1.45	2.65	4.10	6.15
2	1.00	2.15	3.50	5.10
3	3.00	4.80	6.00	6.43
4	2.70	4.10	5.15	5.75

It is interesting that the difference in the flexural strength of concrete at the age of 3...7 days of compositions 1 and 2 compared with concrete of compositions 3 and 4 is significantly less than the difference in the compressive strength of concrete and is 1.8...2.7 times (table 3). This shows that the formation of a contact zone between the aggregate and the hardened cement paste occurs quite intensively at both negative and positive temperatures. This can also be caused by the compaction of the cement gel due to water pressure during partial ice formation, which leads to an increase of the density of the contact zone and the entire concrete, and, accordingly, is reflected on an increase the strength, primarily flexural strength. Subsequently, the rate of gain of flexural strength of concrete that hardened in conditions of negative temperatures decreases, and by 45 days it becomes 5...12 % lower than the strength of concrete that hardened under normal conditions.

From the presented experimental data it is clear that during the initial period of hardening, concrete samples of compositions 1 and 2 containing the antifreeze chemical admixture Frigidol gained strength even under conditions of constant temperature fluctuations. During this period, at night and periodically during the first 10 days during the daytime, the temperature was negative.

It is likely that the acceleration of strength gain in concrete that hardened under conditions of transitions through 0 °C and at negative temperatures is caused by the fact that the hydration of cement in them occurred by two mechanisms: “through the solution” and by topochemical. According to the first mechanism, hydration occurs slowly, but most of the aqueous solution of chemical admixtures in concrete does not freeze, as previously shown. This allows hydrolysis and hydration reactions take place even at negatives temperatures. A similar opinion was expressed in the work (Trofimov, Kramar & Schuldyakov, 2017).

According to the second hardening mechanism, the formation of crystalline hydration products occurs due to the direct introduction of water into the crystalline lattice of the minerals and new formations. It is known that this mechanism is caused by the emergence of internal pressure due to the partial freezing of water and the formation of ice, as well as the pressure of crystallization of new formations. The presence of two hardening mechanisms under such conditions is indirectly confirmed by the well-known data on the increase in the strength of concrete when tested for freezing-thawing (Tolmachov, 2020; Powers, 1965; Moukwa, 1990).

The frost hardening of concrete is especially noticeable at a certain stage when it is tested using an accelerated method (freezing at – 50 °C). In this case, the strength of the tested concrete can increase by 20...30 % or more. The increase in strength is explained by the ongoing hydration of cement during the period of concrete thawing (according to the through-mortar mechanism) and hydrate formation due to the resulting pressures (according to the topochemical mechanism). Under real conditions, concrete samples of all compositions were tested using an accelerated method after 45 days of hardening.

Table 4

Freeze-thaw resistance of airfield concrete

# of mix	Frost resistance coefficient after number of cycles			
	10	20	37	55
1	1.01	0.98	0.93	-
2	1.02	1.05	0.98	0.87
3	1.05	1.09	1.12	0.93
4	1.08	1.10	1.14	1.02

It is known that at all stages of the life of concrete, two opposite processes occur in its structure – structure formation and destruction (Zhang et al., 2020). The data in Table 4 confirms that when concrete is tested for freezing-thawing, its strengthening occurs at the beginning, and the structure of concrete is compacted.

These studies also showed that in the process of testing for frost resistance and corrosion resistance, initial strengthening of concrete and subsequent destruction occurs, which is confirmed by research in the work (Zhang, Yang, Guo, Liu & Chen, 2018).

But the hardening period for each concrete is different. For example, in concrete of composition 1, after 10 test cycles, destruction begins. For concrete of composition 2, hardening continues up to 20 test cycles, and for compositions 3 and 4 - up to 37 and 55 test cycles, respectively. After this, destruction also prevails in their structures.

There are several reasons for this initial strengthening of concrete and the subsequent acceleration of its degradation under the cyclic action of frost and chloride salts. As our studies have shown, the initial increase in the strength of concrete can be explained by the fact that at the initial stage of negative temperatures, water and ice pressure arises, which stimulates the continuation of the cement hydration reaction (Tolmachov, 2020). In this case, the reaction proceeds through a topochemical mechanism and promotes the healing of pores and microcracks in the structure of hardened cement paste and concrete. In addition, from the saline solution in which the tests are carried out, sodium chloride crystallizes in the pores and capillaries of the concrete. Sodium chloride crystals fill these pores and other defects in the concrete structure, which leads to compaction of the structure. This compaction leads to an increase in the strength of concrete. Subsequently, after a certain number of freeze-thaw cycles, microdeformations accumulate in the concrete structure, and sodium chloride crystals begin to exert pressure on the walls of pores and capillaries, which enhances and accelerates the concrete degradation. Destruction is also facilitated by osmotic pressure, which increases as water binds during cement hydration, which continues during testing. A decrease in the amount of free water leads to an increase in the concentration of salts in the aqueous solution, and, therefore, an increase in osmotic pressure.

According to Ukrainian standards, it is considered that concrete has passed the freeze-thaw resistance test if its freeze-thaw resistance coefficient is not lower than 0.95. The minimum freeze-thaw resistance grade for road and airfield concrete is F200 (DSTU 8858:2019, 2020). Tests have shown that concretes of compositions 1 and 2, which initially hardened at negative temperatures, have freeze-thaw resistance that meets these requirements.

In 2021, we tested concrete of compositions 1 and 2, which were laid in 2009 and hardened under conditions of transitions through 0°C, as well as at negative temperatures (Table 5). Visually, the quality of the concrete has not changed. Samples were cut out of concrete, from which cube samples measuring 70x70x70 mm were made, which were tested for compressive strength and freeze-thaw resistance.

Table 5

Freeze-thaw resistance of airfield concrete

# of mix	Freeze-thaw resistance coefficient after number of cycles			
	10	20	37	55
1	1.03	1.01	0.94	0.85
2	1.04	1.06	1.02	0.97

Tests showed that the compressive strength of concrete samples of composition 1 increased from 46.0 MPa at the age of 45 days to 59.6 MPa. The compressive strength of concrete composition 2 also increased from 42.0 MPa to 53.8 MPa. The increase in the strength of concrete of both compositions over a period of 12 years was about 30 %.

Studies of the durability of concrete samples showed that over 12 years of operation, the freeze-thaw resistance of concrete composition 2 not only did not decrease, but even increased by one grade - from F300 to F400. As for concrete of composition 1, its frost resistance remained at the same level. These studies partially confirm the hypothesis expressed in (Zhang et al., 2020) that as the strength of concrete increases, its freeze-thaw resistance also increases. The most probable is that the pore structure of concrete, which was formed during the initial period of hardening, has not changed. Therefore, an increase in strength led to a corresponding increase in freeze-thaw resistance only for concrete that contained an air-entraining additive.

Conclusions

1. It is shown that there are different ideas about the possibility of creating durable monolithic concrete when they harden under conditions of negative temperatures. It is generally believed that in order to create high-quality concrete under these conditions, it is necessary to insulate it and introduce antifreeze additives into its composition.

2. Research has shown that the use of modern superplasticizers makes it possible to stabilize the setting period of cement pastes and ensure a setting duration that is slightly dependent on the temperature of the hardening medium in the temperature range of + 5...+ 19 °C.

3. Concrete, which contained a complex of superplasticizer and antifreeze additive, and which hardened for the first 16 days at negative and low temperatures, by 45 days of on-site hardening had a strength no lower than concrete that hardened under normal conditions. During hardening of these concretes no maintenance was carried out.

4. It has been established that concretes that have hardened under conditions of negative and low temperatures have frost resistance not lower than grade F200, which ensures their necessary durability.

5. The studies carried out did not allow us to establish a correlation between an increase in the strength of concrete and an adequate increase in frost resistance. Perhaps this only applies to concretes that contain additional entrained air.

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ВЛАСТИВОСТІ МОНОЛІТНОГО ЦЕМЕНТОБЕТОНУ, ЩО ТВЕРДНЕ ПРИ ТЕМПЕРАТУРАХ, БЛИЗЬКИХ ДО НУЛЯ

Однією з основних проблем тверднення монолітного цементобетону, особливо в умовах відновлення та реконструкції різних об'єктів, є тверднення бетону в умовах негативних температур. Технології зимового бетонування поділяються на два основні методи: метод термоса, при якому температура середовища, в якій застигає бетон, повинна бути вище нуля градусів за Цельсієм, або використання протиморозних добавок. В останньому випадку доцільно вводити до складу бетону незамерзаючі хімічні добавки, які знижують температуру замерзання води в бетоні. Однак відомі раніше проведені дослідження, які показують, що навіть в разі раннього замерзання бетону його якість залишається високим. При цьому необхідно, щоб початок схоплювання цементної пасти відбулося до того, як бетон зможе замерзнути. Крім того, було висловлено припущення, що затвердіння бетону при низьких температурах може відбуватися і без використання методу термоса. У статті представлені результати впливу температури затвердіння на час схоплювання цементного тіста. Дослідження показали, що застосування сучасних суперпластифікаторів дозволяє стабілізувати період схоплювання цементних паст і забезпечити тривалість схоплювання, яка незначно залежить від температури середовища твердіння в інтервалі температур + 5...+ 1 °С. Бетон, який містив комплекс суперпластифікатора і протиморозної добавки, і який тверднув перші 16 діб при негативних і низьких температурах, до 45 діб природного затвердіння мав міцність не нижче, ніж бетон, який тверднув в нормальних умовах. Встановлено, що бетони, які затверділи в умовах негативних і низьких температур, мають морозостійкість не нижче марки F200, що забезпечує їх необхідну довговічність Показано, як впливають умови затвердіння на міцність і морозостійкість бетону. Ці дослідження частково підтверджують гіпотезу про те, що зі збільшенням міцності бетону зростає і його морозостійкість. Найбільш вірогідним є те, що пориста структура бетону, який утворився в початковий період твердіння, не змінилася. Тому підвищення міцності призводило до відповідного підвищення морозостійкості тільки для бетону, в складі якого була повітровтягуюча добавка.

Ключові слова: початок схоплювання (ПС), кінець схоплювання (КС), час схоплювання (ЧС), міцність, протиморозна хімічна добавка, морозостійкість.