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Curing of concrete with chemical additions in frosty weather

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TECHNICAL TRANSLATION
TRADUCTION TECHNIQUE

S.A. MIRONOV, A.V. LAGOIDA AND E.N. UKHOV

CURING OF CONCRETE WITH CHEMICAL ADDITIONS IN FROSTY WEATHER

BETON I ZHELEZOBETON, 14 (3): 1 - 4, 1968

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V. POPPE

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PREFACE

Winter construction is an accepted fact in Canada and the U.S.S.R. Winter concreting requires special precautions; much valuable experience has been accumulated over the years and new techniques and new materials have markedly affected the speed of winter construction.

Canadian and American specifications limit the amounts of admixtures to be used in concrete. The Russians, however, have allowed, under certain circumstances, large additions of admixtures including chlorides in winter construction. They are mainly added to lower the freezing point of water. In this paper the effect of large additions of potassium, sodium nitrites, sodium and calcium chlorides on the properties of concrete is described.

The translation was made by V. Poppe and checked for technical content by V.S. Ramachandran of the Division of Building Research.

Ottawa
March 1976

C.B. Crawford, Director
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Research

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TECHNICAL TRANSLATION 1854
TRADUCTION TECHNIQUE

Title/Titre: Curing of concrete with chemical additions in frosty weather
(Tverdenie betona s khimicheskimi dobavkami na moroze)

Authors/Auteurs: S.A. Mironov, A.V. Lagoida and E.N. Ukhov

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CURING OF CONCRETE WITH CHEMICAL ADDITIONS IN FROSTY WEATHER

Up to the early fifties it was prohibited in the U.S.S.R. and abroad to use chemical substances to lower the freezing point of water added to the concrete mix. Large additions of calcium and sodium chlorides (22.5% by weight of water in the mix) were used for the first time in the winter of 1951-1952 while concreting the plates on the Volga-Don canal^(1,2). The positive results of investigations and experimental applications of chlorides⁽³⁾ made it possible to recommend a new method of concreting in winter, i.e., the use of concrete containing chemical additions which harden at low temperatures. Owing to its low cost and simplicity, this method has been widely applied in construction in the U.S.S.R. in the last 15 years. On the basis of Soviet experience, the RILEM recommendations on winter concreting contain allowances for the use of chlorides to lower the freezing point of water to -10°C .

Further investigation⁽⁵⁻⁸⁾ have shown that, apart from chlorides, potash and sodium nitrate can also be used to lower the freezing point of water. Any new compound can be used if the construction and engineering properties of concrete containing it will not be inferior to those of concrete containing potash, sodium nitrite or sodium and calcium chlorides, and if addition of the new compound will not significantly affect the cost of concrete. The latter is calculated by adding up the costs of the additions per m^3 of concrete, the equipment required, the safety measures to control the effects of toxic substances and the costs related to the rate of curing of concrete, which determines the speed with which the structure can be completed.

The ability of a chemical compound to lower the freezing point of water is a necessary but not the only criterion which determines whether a substance can

be used as a frost-resistant addition. The data in Table I show that FeCl_3 and $\text{Ca}(\text{NO}_3)_2$ have practically no effect on the rate of hardening of concrete based on portland cement with a high aluminum content even at a temperature as high as -5°C , although they do produce a nonfreezing liquid phase.

In our opinion, the rate of hardening of concrete containing frost-resistant additions depends on two factors: the amount and the bonding of water molecules with the ions (molecules) of added compounds, and the participation of these compounds in the hydration processes. The latter becomes decisive at very low temperatures, when large additions of frost-resistant substances result in an abrupt reduction in the number of "free" water molecules due to the transition of most water to a solvate state. In this connection it would be interesting to examine the hydration of cement, since in the absence of physical factors (in this case freezing) the mechanism of hydration and the properties of new hydrate phases determine the properties of the concrete mix and the hardened concrete, and hence the limits of application of concretes containing various additions.

On adding aqueous solutions of calcium chloride to a concrete mix, the lime produced during the hydrolysis of tricalcium silicate reacts with the salt forming calcium oxychloride (predominantly $3 \text{CaO} \cdot \text{CaCl}_2 \cdot 15 \text{H}_2\text{O}$). Being a crystalline compound, the oxychloride fulfills the same function as $\text{Ca}(\text{OH})_2$ in the hardening of concrete under normal conditions. Formation of insoluble calcium oxychloride brings about a more rapid and more complete hydration of tricalcium silicate, since lime is immediately removed from the liquid phase. This, together with the lowering of the freezing point of water, is responsible for the fact that calcium chloride is an effective frost-resistant addition^(9,10).

The presence in the liquid phase of calcium chloride capable of producing

complex salts with calcium hydroaluminates accelerates to a certain extent the formation of the primary structure of the cement stone. Once the bonding of gypsum is complete, the aluminate components of cement react with CaCl_2 forming calcium hydrochloraluminate, the most probable composition of which is $3 \text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCl}_2 \cdot 10 \text{H}_2\text{O}$. Evidently, the formation of this compound does not change the kinetics of hardening of concrete, since instead of hexagonal calcium hydroaluminates there is calcium hydrochloraluminate of the same crystalline form. Because of this, the rate of hardening of concrete increases with the content of tricalcium silicate in the cement and is little dependent on the amount of tricalcium aluminate.

The aforementioned new hydrate forms, i.e., calcium oxychloride and hydrochloraluminate, are formed in the presence of sodium chloride. But because of a slow interaction of sodium chloride with calcium oxyhydrate, which occurs only as a result of calcium chloride forming complex crystallohydrates, the nonfreezing liquid phase is constantly retained in the concrete. This explains the higher strength of concrete containing NaCl rather than CaCl_2 at not very low temperatures (see Table I).

To produce the liquid phase at low temperatures, large amounts of chlorides must be added to concrete. If NaCl only is used, then, due to the fact that large amounts of water are used up to form solvates and there is not rapid interaction of the salt with the products of hydration of cement, the strength of concrete increases only slowly. On the other hand, addition of CaCl_2 only may result in an early freezing of concrete due to formation of calcium oxychloride. To avoid this at temperatures below -5°C , both chlorides should be added to concrete simultaneously⁽¹¹⁾.

Calcium oxychloride decomposes if the temperature rises above 0°C and even in the case of prolonged hardening of concrete at low temperatures. If calcium oxychloride is the dominating compound in the structure of the cement stone, its decomposition results in a considerable reduction in the strength of concrete. It has been found that a stable structure may be obtained by adding chlorides to the concrete mix in amounts not exceeding 7.5% by weight of cement, and this determines the lowest permissible temperature of concrete (-15°C) when using chlorides as frost-resistant additions. The metastability of calcium oxychloride, as well as the presence of free sodium chloride, limits the range of application of concretes containing chlorides⁽¹¹⁾, since free chlorides corrode the reinforcements and lead to efflorescence on the surface of concrete.

In the solutions of potash, the interaction of the salt with calcium oxyhydrate results in the formation of insoluble calcium carbonate and hydrocarbonate $\text{CaCO}_3 \cdot 6 \text{H}_2\text{O}$ ⁽¹²⁾. If potash is added in amounts ranging from 5 to 7% by weight of cement, the hydration of cementing material becomes too rapid, which may weaken the structure of the cement stone. Formation of a defective structure manifests itself externally in an almost instantaneous setting of cement and a rapid loss of mobility of the concrete mix.

If the salt content increases due to the increased alkalinity of the liquid phase, the formation of calcium carbonates slows down. A dense structure of the cement stone is formed. Calcium hydrocarbonate in the form of large prismatic crystals reinforces the structure and, together with the hydration products of the aluminate components of cement, determines the strength of concrete in the initial period of hardening. Later this crystallohydrate fulfills the same function as calcium oxychloride when chloride solutions are added to the concrete mix, except that $\text{CaCO}_3 \cdot 6 \text{H}_2\text{O}$ decomposes to CaCO_3 and H_2O only if the temperature

of concrete rises above 0°C.

Potassium hydroxide is formed simultaneously with calcium carbonate and hydrocarbonate. Its aqueous solution has a lower freezing point (Figure 1). The increase in the alkalinity of the liquid phase due to conversion of some K_2CO_3 to KOH slows down the hydration of cement, but at the same time reduces the amount of potash required to produce an aqueous solution which will not freeze at a specified below-zero temperature. Since the rate of formation of KOH in the initial hardening period depends first of all on the amount of rapidly hydrating minerals (tricalcium aluminate and tetracalcium aluminoferrite), the required amount of potash decreases with the increase in the content of these minerals in cement.

Since calcium carbonates are practically insoluble in water, they can be formed until almost all potash has reacted with calcium hydroxide. If there is not enough hydrolytic lime for this interaction, then it may be expected that lime will be produced by calcium hydrosilicates which determine the strength of concrete. Considering the degree of hydration of portland cements and their content of tricalcium silicate, the amount of potash added to the concrete mix must not exceed 15% by weight of cement to avoid the decomposition of calcium hydrosilicates⁽¹³⁾.

Hydration of the aluminate components of portland cement in the presence of potash results in formation of calcium hydrocarboaluminate $3 CaO \cdot Al_2O_3 \cdot CaCO_3 \cdot 11 H_2O$ ⁽¹⁴⁾. In contrast to calcium hydrochloraluminate, this compound plays a more important role in determining the strength and other properties of concrete, since calcium hydrosulfoaluminate is unstable in potash solutions⁽¹⁵⁾. Furthermore, $3 CaO \cdot Al_2O_3 \cdot CaCO_3 \cdot 11 H_2O$ may form intergrowths with $CaCO_3$ and thus strengthen the crystalline lattice of the cement stone⁽¹⁶⁾. Therefore, the strength of

concretes containing potash can be increased by using high-aluminate portland cements.

Studies of the kinetics of hardening of concrete containing sodium nitrite indicate that as a rule the hardening rate slows down with the increasing content of this salt. The highest strength is obtained on adding at a given low temperature the least possible amount of sodium nitrite (Table II).

This may be explained by the fact that if the salt content is increased, the composition of the solution will approach the eutectic solution, which is characterized by the absence of "free" water molecules (the latter are bonded together to form solvates). This is accompanied by a slowing down of the interaction of water with the cement minerals. The interaction practically stops at temperatures below -15°C .

Sodium nitrite may participate in hydration of the aluminate components of cement and formation of calcium hydronitrialuminate $3 \text{ CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Ca}(\text{NO}_2)_2 \cdot 10 \text{ H}_2\text{O}$. Formation of this compound may take place once all gypsum has been combined into calcium hydrosulfoaluminate. Like calcium hydrochloraluminate, calcium hydronitrialuminate has little effect on the strength of concrete. However, NaOH is also present and is adsorbed on the surface of the siliceous aggregate better than the salt. Probably because of this, more NaNO_2 must be added at the same low temperature to increase the strength of concrete containing cement with an increased content of tricalcium aluminate.

Investigations of the strength of concrete based on portland cements from seven plants made it possible to determine the optimum additions of frost-resistant salts (see Table III).

Sodium nitrite and potash do not increase the corrosion of steel reinforcements.

However, they cannot be used if the aggregate contains active silica, since this would lead to formation of caustic alkalies during hardening of concrete^(17,18). When using nonreacting aggregates, one may hope for a long-time performance of concretes containing potash, since silica is uniformly distributed in concrete. Addition of finely ground pozzolan would distribute active silica more evenly and hence eliminate the alkaline corrosion of concrete, even if the latter contains reacting aggregates⁽¹⁹⁾. However, lack of experimental data does not permit lifting the ban on the use of potash as a frost-resistant addition in concrete structures in aqueous or strongly humid media.

In contrast to sodium chloride and sodium nitrite, potash and calcium chloride may lead to a rapid loss in mobility of the concrete mix due to their active participation in the hydration of cement. An excessively rapid setting of the concrete mix can be effectively slowed down by adding surface-active substances to it (naft soap, sulfite-alcohol distillery waste, etc.). The amount of the setting-time retarders may reach 2% by weight of cement. In contrast to concrete without additions, such large amounts of surface-active substances do not result in an excessive retardation of setting and hardening, which is due to the interaction of frost-resistant additions with organic compounds. It is interesting that in the case of low-aluminate portland cements, the surface-active substances may be used to accelerate the setting process which is considerably slowed down in the presence of large amounts of potash (Figure 2).

The hydration processes discussed above obviously do not provide a complete explanation of the effect of frost-resistant additions on the hardening of concrete. The salts bring about changes in the solubility of initial minerals and hydrate phases, in the stability of the latter and in practically all physico-chemical processes which occur during the hardening of concrete. However, the

large amounts of salts added to the concrete mix and the fact that the efficiency of these additions is noticeably dependent on the mineralogical composition of cement are sufficient reasons to assume that the hardening rate of concrete is strongly dependent on the ability of the additions to participate in the hydration of cement and formation of new phases, which in turn have a positive effect on the formation of the structure of the cement stone in concrete.

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Table I

Strength of concrete based on Spassk portland cement at -5°C

Addition		1:1.5:3:0.45, $R_{ss} = 432 \text{ kg/cm}^2$				1:2:3.5:0.55, $R_{ss} = 318 \text{ kg/cm}^2$			
Compo- sition	% by wt. of cement	Compression strength in % of R_{28} after (days):							
		7	14	28	$\frac{+28 (-5^{\circ})}{+28 (+20^{\circ})}$	7	14	28	$\frac{+28 (-5^{\circ})}{+28 (+20^{\circ})}$
—	—	9	13	21	53	9	15	21	62
NaCl	1	30	38	63	112	28	46	48	116
	3	36	55	67	124	26	40	61	113
	5	36	49	70	117	29	55	69	116
KCl	2	22	33	35	63	17	28	38	57
	3	23	38	50	74	24	38	49	71
	5	24	49	62	100	27	54	63	120
CaCl ₂	2	14	21	23	71	9	13	22	70
	3	14	20	33	75	16	19	29	104
	5	23	35	47	93	23	29	32	105
FeCl ₃	2	3	6	16	48	2	3	5	37
	3	4	7	30	49	4	9	20	77
	5	5	21	32	75	9	16	23	83
NaNO ₂	2	40	70	97	98	40	61	100	112
	3	40	74	88	93	23	52	68	111
	5	23	62	80	102	27	54	81	112
K ₂ CO ₃	2	30	36	52	104	33	48	56	119
	3	31	45	61	98	27	44	64	103
	5	34	59	59	96	53	73	89	104
Ca(NO ₃) ₂	3	9	19	19	61	6	10	15	38
	5	10	14	24	50	8	10	14	59
	7	14	20	25	78	7	7	14	50

Table II

Strength of concrete containing potash and sodium nitrite

Temp. of concrete, °C	Addition		Spassk port-land cement						Chernorechensk portland cement					
	Compo-sition	% by wt. of cement	1:1,5:3; :0,45; $R_{1,2}$ = = 432 kg/cm²			1:2:3,5; :0,55 $R_{1,2}$ = = 318 kg/cm²			1:1,5:3; :0,45; $R_{1,2}$ = = 401 kg/cm²			1:2:3,5; :0,55; $R_{1,2}$ = = 309 kg/cm²		
			Compression strength in % of R ₂₈ after (days):											
			7	14	28	7	14	28	7	14	28	7	14	28
-5	K ₂ CO ₃	2	30	36	52	33	48	56	53	57	81	58	70	
		3	31	45	61	27	44	64	56	68	56	55	77	69
		5	34	59	59	53	73	89	49	59	—	60	69	74
		10	27	34	40	26	34	39	20	28	47	16	25	45
		12	32	38	42	24	37	45	27	35	61	18	27	47
-25		15	40	44	53	28	37	50	24	39	28	19	32	58
-5	NaNO ₂	2	40	70	97	40	61	100	23	33	37	18	26	38
		3	40	74	88	23	52	68	21	43	50	20	38	46
		5	23	62	80	27	54	81	30	47	62	18	43	56
		8	21	38	48	17	28	36	11	22	31	9	18	24
		10	23	38	48	19	34	44	11	14	28	9	18	23
-15		12	22	38	43	24	38	44	9	11	28	6	18	19

Table III

Strength of concrete based on portland cements
with optimum amounts of frost-resistant salts

Temp. of concrete, °C	Addition		Compression strength in % of specified strength (days):			
	Comp.	% by wt. of cement	7	14	28	90
-5	NaCl + CaCl ₂	3,0+0,0	30—40	60—70	70—90	90—100
-10		3,5+1,5	20—30	30—40	40—50	60—80
-15		3,0+4,5	10—20	20—30	30—40	40—60
-5	NaNO ₂	4—6	20—40	40—60	60—80	80—100
-10		6—8	10—30	20—50	40—70	60—80
-15		8—10	10—20	20—30	30—40	40—60
-5	K ₂ CO ₃	5—6	40—60	60—70	70—80	90—100
-10		6—8	20—40	40—60	60—80	80—100
-15		8—10	20—30	30—50	50—70	70—90
-20		10—12	20—30	30—50	40—70	60—80
-25		12—15	10—30	20—40	40—60	50—70

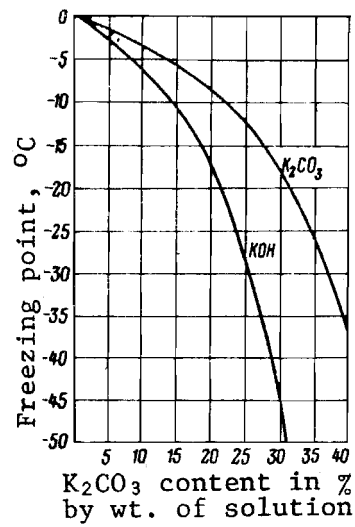


Fig. 1

Freezing point of aqueous K_2CO_3 and KOH solutions vs. the potash content of solutions

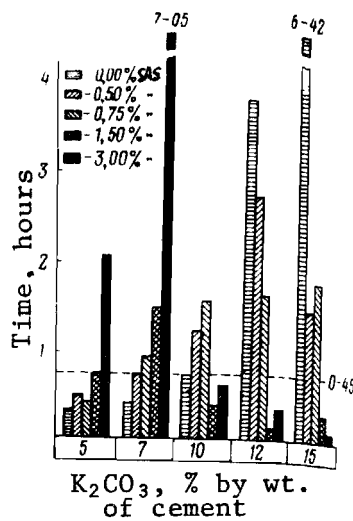


Fig. 2

Start of setting of Bryansk portland cement at 20°C vs. addition of potash and surface-active substances
SAS = surface-active substances