# The Influence of Salt Composition from Soda Manufacture Wastes upon the Properties of Grouting Mortars

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## Abstract

An effect of the salt composition obtained from soda manufacture wastes on the hydration of mineral binding agents and the hardening of grouting mortars has been studied. Recipes for grouting mortars developed on the basis of salt compositions are presented.

**Key words:** salt composition, grouting mortars, portland cement, calcium chloride, sodium chloride, hydration

### INTRODUCTION

The hydration solidification of cement at all the stages is connected with chemical and physicochemical processes. The properties of a grouting mortar and a cement stone under formation could be changed effecting on the reactions proceeding therein. The most widespread and efficient method to influence the process of cement hardening consists in the use of various kind of active admixtures [1–4].

The salt composition obtained from large-tonnage liquid waste products of soda manufacture those need to be recycled consist of calcium and sodium chlorides. The mass fraction value for calcium ions therein is not less than 18 %, that for chlorine ions being not less than 51 %, whereas the fraction of water-insoluble residue does not exceed 2 % [5]. According the composition and properties this salt mixture is close to a well-known reagent "Technical grade calcium chloride" (State Standard GOST 450–77) widely applied as an active admixture for oil-and-gas producing well operation. In this connection the studies on possible utilizing the salt composition obtained from waste products of soda manufacture in order to substitute this reagent are of current importance.

It is known that calcium chloride as one of the components of grouting mortars used for oil- and-gas well operation performs the functions of an accelerator of stiffening and hardening, increasing the durability of oil-well plugging stone and adjusting the viscosity parameters of the mortar for well cementation. There are various well operation conditions in the oil-and-gas region of West Siberia, which requires for the development of grouting mortars with the composition and properties optimal for a particular deposit [6].

The choice of either way of activation is basically determined by the following technological tasks: realizing the potential capability of the cementing agent for reduction of cement consumption; the creation of a more plastic system with no application of organic plasticizing agents decelerating the hydration and lowering the durability; the necessity to provide the accelerated hardening at a certain stage of the structurization (during the period of stiffening, after the operations of pumping or laying the mortar, *etc.*). However, irrespective of the specificity of the problem concerning the regulation of certain properties of the system, first of all it is necessary to provide obtaining a hardened material corresponding to the features of a particular deposit as much as possible [7].

The purpose of the present work consisted in the investigation of the effect of salt composition additives on physicomechanical and technological of the mortar and stone obtained basing on PCT-D20-50 oil-well portland cement with additional introduction of other additives.

## EXPERIMENTAL

As base cement a PTsT-D20-50 oil-well portland cement was used. It is known [3, 4, 8] that the addition of sodium and calcium chlorides to grouting mortars results in the increase in the cement stone durability as well as in the reduction of the presetting period. As the basic criteria for the estimation of the quality of cement mortar (stone) we have chosen strength characteristics and presetting period under simultaneous maintaining the pumpability of the mortar obtained.

The composition of the salt mixture includes calcium chloride (60–65 %) and sodium chloride (35–40 %) those exert different influence upon the processes of cement mortar hardening [9]. The most efficient reagent among the aforementioned ones is $CaCl_2$. So, at a higher than 5 % NaCl content in cement mortar the rate of setting and hardening of straight cement mortar decreases, which is not observed in the case of adding the same amount of $CaCl_2$ though the strength properties of cement could worsen [10].

In the experiments with PTsT-D20-50 oil-well portland cement the content of the salt composition additive was varied within the range from 1 up 20 %, the value of $H_2O$/cement ratio amounted to 0.55–0.60. The values of mortar mobility, presetting period values, mechanical bending strength were measured. The additive of the salt composition was introduced into the tempering liquid wherein the PTsT-D20-50 oil-well portland cement and other mixtures on its base were then dissolved. The bending strength was determined for the values of stone age amounting to 2, 7, 14, 28 days. The influence of the salt composition additive upon the properties of the mortar and stone based on PTsT-D20-50 was studied at the cement hardening temperature equal to 22 °C.

A series of experiments was carried out for establishing the influence of the salt composition additive upon the characteristics of grouting mortars (stone) based on various types of cements such as calcium aluminate cement, ferrite-alinite cement and expanding cements on the base of PTsT-D20-50. The experiments were carried out at the hardening temperature values amounting to −5 °C and 22 °C. The content of the salt composition was varied within the range from 5 to 15 %.

We have also studied the possibility of soluble glass use as an active additive. For obtaining this kind of additive the ratio between the components (cement + salt composition mixture and soluble glass) amounted to 1 : 1, 3 : 1 and 5 : 1. The intergrinding time of the components was varied within the range from 10 to 40 min. The mixtures obtained were dissolved in water (the temperature of water being at 80 °C).

For the investigation of the hydration kinetics of the cementing systems, to a monomineral cementing agent ($3CaO \cdot SiO_2$, $3CaO \cdot Al_2O_3$, $2CaO \cdot Fe_2O_3$, oil-well portland cement) was added 5 and 15 % of a salt composition. In 2, 5 and 8 h sampling was performed, the samples taken being then analyzed by means of a thermogravimetry assay in order to determine the hydration level. We determined the value of hydration rate constant and the activation energy for the hydration process for various cementing agents [15].

## RESULTS AND DISCUSSION

The results of the studies on the salt composition additive influence upon the properties of the mortar and stone based on PCT-D20-50 oil-well portland cement are presented in Table 1.

One can see that the optimum content of the salt composition in the mortar ranges within 5–10 %. A lower content of the salt composition does not exert any significant effect on the reduction of the presetting period, whereas the content higher than 10 % results in worsening the strength properties of the cement stone as well as in abrupt lowering the mobility of the cement mortar.

The results of the studies on the influence of the salt composition on the characteristics of grouting mortars (stone) based on calcium

TABLE 1

Influence of salt composition added upon the properties of mortar and stone based on oil-well portland cement PTsT-D20-50 ($T$ = 22 °C)

| Additive content | Water/cement ratio | Spreadability, cm | Presetting period, h-min | | Bending strength, MPa, after period of time, days | | | |
|---|---|---|---|---|---|---|---|---|
| | | | Start | End | 2 | 7 | 14 | 28 |
| 0 | 0.50 | 20 | 7-35 | 11-05 | 2.5 | 5.1 | 6.0 | 7.0 |
| 1 | 0.50 | 19 | 6-55 | 8-15 | 2.9 | 6.5 | 7.1 | 7.3 |
| 5 | 0.55 | 18 | 3-40 | 4-55 | 3.6 | 5.6 | 6.7 | 7.0 |
| 10 | 0.60 | 16 | 1-00 | 2-20 | 2.8 | 3.9 | 4.8 | 6.6 |
| 20 | 0.60 | 14 | 0-15 | 1-15 | 1.9 | 3.3 | 4.5 | 6.2 |

aluminate cement, ferrite-alinite cement and expanding cements on the base of PCT-D20-50 are presented in Table 2.

It is apparent that the highest durability is exhibited by calcium aluminate cement with the salt composition content equal to 5 %. However, with the increase in the salt composition content up to 15 % we observed a decrepitation of the samples connected, to all appearance, with the occurrence of an internal stress as the result of growth and formation of calcium hydrochloroaluminate crystals under the conditions of closed pore space within a cement stone. High strength parameters of the stone based on calcium aluminate cement are connected both with the features of cementing agent hardening, and with the character of pore structure of the stone. Furthermore, the increase in durability of the stone based on calcium aluminate cement with the salt composition added could be caused by the electrolytic influence of calcium and sodium chlorides upon the processes of initial phase dissolution and crystalline hydrate formation.

In the studies on the compositions of expanding cements and the salt mixture with the use of devices such as PNG we have also investigated the processes of volume expansion during 1 day of hardening, as well as contact stress and bending strength for the stone after 2 days of hardening (Table 3). One can see that with the introduction of the salt composition the volume expansion value amounts to 8 %, the contact stress being 3 MPa. At the same time, when 15 % of the salt composition added, the durability of the sample abruptly falls resulting in “blowing up” under internal stress.

TABLE 2

Physicomechanical properties of grouting mortars based on various cements containing the salt composition

| Cement | Additive content, % | Hardening temperature: 22 °C — Presetting period, h-min, start/end | Hardening temperature: −5 °C — Presetting period, h-min, start/end | Bending strength, MPa* |
|---|---|---|---|---|
| Calcium aluminate cement | 5 | 3-05/4-30 | 5-50/9-45 | 4.8/1.2 |
| | 10 | 1-45/2-00 | 4-35/8-15 | 0.9/1.9 |
| | 15 | 0-40/1-15 | 3-05/7-40 | 0.9/2.1 |
| Ferrite-alinite cement | 5 | 5-00/7-30 | 8-05/14-20 | 3.4/0.8 |
| | 10 | 4-35/7-10 | 7-25/12-45 | 2.9/1.8 |
| | 15 | 3-50/6-25 | 6-30/12-45 | 1.6/2.5 |
| PTsT-D20-50 | 5 | 3-55/5-10 | 7-35/11-30 | 3.5/0.3 |
| | 10 | 1-50/2-15 | 6-40/10-20 | 2.9/0.9 |
| | 15 | 1-55/2-20 | 5-15/10-05 | 2.2/1.0 |

*The first and second values correspond to the hardening temperature of 22 °C and −5 °C, respectively.

TABLE 3

Influence of adding the soda manufacture salt composition upon the properties of expanding cement

| Additive content, % | Volume expansion in 24 h, % | Contact stress, MPa | Bending strength in 2 days, MPa |
|---|---|---|---|
| 0 | 3.0 | 1.0 | 2.5 |
| 5 | 4.0 | 1.6 | 3.8 |
| 10 | 6.5 | 2.5 | 3.1 |
| 15 | 8.0 | 3.0 | – |

The increase in the expansion effect with the introduction of the salt composition could be caused by a preliminary repacking of water molecules of in the tempering liquid, by changing the conditions of accretion new growth hardening cement as well as by an excess amount of calcium ions in the interstitial liquid of the mortar (stone). The overall combination of these factors results in a considerable increase in the volume of the mortar (stone) [11].

The results we have obtained indicate that the salt composition could be used as an accelerator of cement hardening, as well as for intensifying the effect of expansion and self-stressing of expanding cements. However, the most promising direction could be presented by the obtaining of multipurpose lightening additives on the base of the salt composition. Moreover, the demand of oil-and-gas producing branch for such lightened cements or for the additives to traditionally used portland cements is very high.

A number of methods for reducing the density of grouting mortars is widely applied for today [10, 12]. However, as the result of using the majority of known lightened grouting mortars there is a considerable decrease in durability and a dilution of hydrate ligament in the bulk of stone, which causes the porosity and permeability of the cement stone to increase. As a result, the operational characteristics of the lightened cements used for stabilizing oil wells, considerably deteriorate [13]. There are various technological decisions known to eliminate these disadvantages, *e.g.* obtaining colloidal suspensions for tempering the cements. Depending on the type of conditioned phase obtained in the tempering liquid the suspensions could accelerate or decelerate the process of cement hardening.

Another advantage of the mortars with conditioned solid phase consists in the additional repacking of water molecules. Due to this fact there is a decrease in the contraction of the cement under hardening and thus the reduction of porosity and permeability is provided.

TABLE 4

Time of colloid formation on the base of the salt composition and soluble glass

| Component ratio | Grinding time, min | Sieve residue*, % | Colloid formation time, min | Colloid density, kg/m$^3$ |
|---|---|---|---|---|
| 1 : 1 | 10 | 20 | 360 | 1300 |
| | 20 | 15 | 240 | 1280 |
| | 40 | 5 | 180 | 1280 |
| 3 : 1 | 10 | 10 | 200 | 1200 |
| | 20 | 5 | 120 | 1200 |
| | 40 | 2 | 60 | 1200 |
| 5 : 1 | 10 | 8 | 240 | 1150 |
| | 20 | 3 | 80 | 1150 |
| | 40 | – | 40 | 1150 |

*Mesh aperture being 0.8 mm in diameter.

TABLE 5

Influence of the lightening additive containing lime-belite mixture (LBM) (active CaO content of 35 %) upon physicomechanical properties of mortar (stone) based on PTsT-D20-50

| LBM content, % | Tempering liquid density, kg/m$^3$ | Water/cement ratio | Spreadability, cm | Grouting mortar density, kg/m$^3$ | Bending strength, MPa |
|---|---|---|---|---|---|
| – | 1200 | 1.5 | 18 | 1570 | 1.2 |
| 5 | 1100 | 1.1 | 18 | 1530 | 1.5 |
| 10 | 1070 | 0.8 | 18 | 1510 | 2.1 |
| 20 | 1040 | 1.2 | 18 | 1500 | 2.0 |

Soluble glass belongs to well-known chemical compounds those are capable to form the analogues of portland cement hardening products due to interaction with calcium chloride, being available over the Ural and Siberia territory. We have investigated the dissolution duration for the mixture based on oil-well portland cement with various proportions of components (salt composition : soluble glass ratio = 1 : 1, 3 : 1, 5 : 1) after intergrinding the components and dissolving in water at the temperature of 80 °C (Table 4). It is apparent that at the ratio of components equal to 1 : 1 the mixture obtained represents an activator of slag cements only, whereas the mixture with the ratio of components equal to 5 : 1 already plays the role of a lightened additive.

Thus, we have demonstrated the feasibility of obtaining a lightening additive that improves physicomechanical properties of stone at low concentrations in grouting mortar, reducing the mortar density at the additive content higher than 5 % and providing the stone strength characteristics better than the standard values meeting the requirements for oil-well portland cements (State Standard GOST 1581–96). Even provided the salt composition and soluble glass being in the ratio 1 : 1, under normal conditions the hardening rate value for slag cements exhibits an increase.

However, the use of soluble glass as an additive for the salt composition is complicated due to its hygroscopic and blocking properties whereby a caking of the mixture obtained occurs on open-air keeping during 1 day. Furthermore, the presence of NaCl in the salt composition and additional formation of this substance in the course of the exchange reaction causes the additive efficiency to decrease. In this connection we have attempted to increase the efficiency of soluble glass action through the introduction of a lime-containing additive into the mixture. Firstly, colloidal calcium hydrosilicates are formed due to lime interaction with soluble glass, which causes the possibility of inert sodium chloride formation to decrease; secondly, high lime ability with respect to hydration prevents the additive from caking.

The mixture with the salt composition to soluble glass ratio 3 : 1 was modified *via* the introduction of lime-belite mixture (LBM) based on solid wastes from soda manufacture amounting to 5, 10, 20 % as a lime-containing additive (Table 5). One can see that with increase in the content of LBM up to 20 % the density of grouting mortar exhibits a decrease down to 1500 kg/m$^3$. The lightening additive containing the salt composition could influence the surface properties of cement to activate them accelerating the hardening of cement. This is especially important at high water content of in

TABLE 6

Hydration level of the mixtures under investigation for various hardening time

| Composition No. | Hydration level at hardening time*, h 2 | 5 | 8 |
|---|---|---|---|
| 1 | 1.13/1/21 | 1.11/1.30 | 1.30/1.39 |
| 2 | 1.43/1.45 | 1.59/1.66 | 1.62/1.68 |
| 3 | 2.30/2.41 | 2.71/2.85 | 2.70/2.90 |
| 4 | 3.20/3.34 | 3.30/3.38 | 3.50/3.71 |
| 5 | 1.56/1.64 | 1.98/1.98 | 1.83/1.96 |
| 6 | 1.70/1.76 | 1.90/2.04 | 2.01/2.10 |
| 7 | 1.18/1.54 | 1.30/1.61 | 1.35/1.80 |
| 8 | 1.84/1.88 | 1.91/2.88 | 1.25/2.27 |

*The first and second values were observed for hardening at 20 and at 50 °C, respectively.

the cement mortar in obtaining mortars of lowered density. The introduction of the salt composition could promote the destruction of water structure as well as accelerate the process of cement dissolving.

In order to study the influence of the salt composition upon the hydration kinetics for cementing systems, a series of experiments was carried out with the addition of the salt composition (the content amounting to 5 and 15 %) to a monomineral binder such as tricalcium silicate cement ($C_3S$), tricalcium aluminate ($C_3A$) cement, dicalcium ferrite ($C_2F$) cement and oil-well portland cement. After 2, 5 and 8 h passed since the beginning of the experiment, a sampling was carried out and the samples were prepared for thermogravimetric analysis according to well-known techniques. The recipes of the compositions under investigation are presented below (the amount of the salt composition being expressed as percentage):

| Composition No. | Recipe of mixture |
|---|---|
| 1 | $C_3S$ + 5 % |
| 2 | $C_3S$ + 15 % |
| 3 | $C_3A$ + 5 % |
| 4 | $C_3A$ + 15 % |
| 5 | $C_2F$ + 5 % |
| 6 | $C_2F$ + 15 % |
| 7 | PTsT-D20-50 + 5 % |

The hydration level of the mixtures within various hardening time was determined according to the data of thermogravimetric analysis of hardened mortar samples (Table 6). Table 7 demonstrates the values of hydration rate constants determined according to [14]. One can see that the hydration rate increases with the increase in the hardening temperature. Moreover, with the increase in the content of the salt composition additive for all the kinds of binders the rate value for the processes of cementing agent interaction with water is observed to increase. It is especially exhibited for the hydration of $C_3S$ and PTsT-D20-50.

There are activation energy ($E$) and preexponential factor ($K_0$) values presented below for the hydration of various cementing agents [15] determined basing on a known hydration rate dependence on the temperature:

| Composition No. | $K_0$, $h^{-1}$ | $E$, J/mol |
|---|---|---|
| 1 | 1.325 | 3491.9 |
| 2 | 0.533 | 629.0 |
| 3 | 1.182 | 1360.8 |
| 4 | 1.339 | 1076.5 |
| 5 | 0.718 | 1043.8 |
| 6 | 0.806 | 1193.6 |
| 7 | 5.326 | 6702.6 |
| 8 | 1.551 | 2828.1 |

Table 8 displays the hydration rate constants calculated from these values for the temperature range from 20 to 60 °C. The data obtained indicate that the most abrupt decrease in the activation energy of the hydration process is inherent in $C_3S$ and PTsT-D20-50. This fact confirms the hypothesis we offered earlier concerning the mechanism for the influence of salt

TABLE 7

Hydration rate constants for the mortars at various temperatures and different hardening time

| Composition No. | Hydration rate constant, $h^{-1}$ at hardening duration, h | | | Average |
|---|---|---|---|---|
| | 2 | 5 | 8 | |
| 1 | 0.565/0.650 | 0.222/0.260 | 0.162/0.174 | 0.316/0.361 |
| 2 | 0.715/0.725 | 0.318/0.332 | 0.202/0.210 | 0.412/0.422 |
| 3 | 1.150/1.205 | 0.542/0.570 | 0.338/0.362 | 0.676/0.712 |
| 4 | 1.600/1.670 | 0.660/0.676 | 0.438/0.464 | 0.899/0.937 |
| 5 | 0.780/0.820 | 0.396/0.396 | 0.229/0.245 | 0.468/0.487 |
| 6 | 0.850/0.880 | 0.380/0.408 | 0.251/0.262 | 0.494/0.517 |
| 7 | 0.590/0.770 | 0.260/0.322 | 0.169/0.225 | 0.340/0.439 |
| 8 | 0.920/0.940 | 0.382/0.400 | 0.156/0.284 | 0.486/0.541 |

*Note*. The first and second values were observed for hardening at 20 and 50 °C, respectively.

TABLE 8

Hydration rate constants for the compositions under investigation at various hardening temperatures, $10^{-2} \cdot h^{-1}$

| Composition | Hardening temperature, °C | | | | |
|---|---|---|---|---|---|
| No. | 20 | 30 | 40 | 50 | 60 |
| 1 | 0.316 | 0.331 | 0.350 | 0.361 | 0.375 |
| 2 | 0.412 | 0.415 | 0.418 | 0.422 | 0.425 |
| 3 | 0.676 | 0.689 | 0.701 | 0.712 | 0.723 |
| 4 | 0.899 | 0.912 | 0.925 | 0.937 | 0.948 |
| 5 | 0.468 | 0.474 | 0.481 | 0.487 | 0.492 |
| 6 | 0.494 | 0.502 | 0.510 | 0.517 | 0.524 |
| 7 | 0.340 | 0.372 | 0.405 | 0.439 | 0.473 |
| 8 | 0.486 | 0.505 | 0.523 | 0.541 | 0.558 |

composition chlorides on the process of cement hardening [16].

The addition of the salt composition promotes the acceleration of both hydration and structurization processes (due to calcium chloride) [17], which provides a considerable extension of the practical application field. In particular, the salt composition use allows one to obtain quickly thickening grouting mortars for the liquidation of various troublesome zones.

The hydrated calcium ions are capable to incorporate into a solvate sphere, for example, belonging to clay particles. Therefore, clay (mud powder) should be added to the mixture for structurizing a tempering liquid as well as for fast oil-well portland cement structure formation. We have investigated the composition with mud powder added to the cement, and with the salt composition added to the tempering liquid. This "quickly structurizing composition" was tested according to the techniques recommended by the State Standard GOST 26798.1–96 (Table 9).

Thus, the salt composition can be used instead of calcium chloride. Moreover, in some cases the use of the former could be more preferable due to the presence of NaCl and amorphized silica therein; those intensify the structurization of cement. It is especially important for the isolation of thief zones with considerable fissure opening when the grouting mortar deg-

TABLE 9

Influence of the salt composition upon the properties of the mortar based on "quickly structurizing composition" (testing temperature 20 °C)

| Mixture composition, % | | | Water/cement | Spreadability, | Thickening |
|---|---|---|---|---|---|
| PTsT-D20-50 | Clay | Salt composition | ratio | cm | time, h-min |
| 100 | – | 5 | 0.5 | 20 | 2-20 |
| 100 | – | 10 | 0.5 | 18 | 0-30 |
| 100 | – | 15 | 0.5 | 18 | 0-15 |
| 95 | 5 | 5 | 0.7 | 18 | 4-20 |
| 95 | 5 | 10 | 0.6 | 18 | 1-10 |
| 95 | 5 | 15 | 0.6 | 18 | 0-55 |
| 92 | 8 | 5 | 0.8 | 18 | 6-00 |
| 92 | 8 | 10 | 0.7 | 18 | 3-00 |
| 92 | 8 | 15 | 0.6 | 18 | 0-30 |
| 90 | 10 | 5 | 0.9 | 18 | 7-00 |
| 90 | 10 | 10 | 0.8 | 18 | 4-50 |
| 90 | 10 | 15 | 0.7 | 18 | 0-40 |

radation can happen earlier than the efficient mortar structurization, and it is not possible to provide a reliable isolation of a troublesome zone.

The thickening of the mortar on the basis of portland cement occurs earlier than it is registered according to the presetting period, *i.e.* for a certain time the mortar exists in a low-plasticity state. The adding of clay prevents this phenomenon reinforcing the coagulation structure formed due to additional formation of hydrogen bonds in the bulk of the mortar. It could be caused by the fact that clay exhibits acceptor properties in aqueous media. In this connection, owing to the presence of calcium ions as well as due to the donor action of oil-well portland cement it is possible to control additionally the structural properties of grouting mortars.

The studies on the processes of pure portland cement structurization in the presence of the salt composition indicates that there is a considerable rate of structurization with the subsequent destruction of the structure formed, especially at a low content of the salt composition in the bulk of the mortar. At the same time, the results obtained confirm the suggestions made earlier concerning the structurization processes from thermodynamic standpoint as well as taking into account the salt composition influence on the processes. The character of the structurization process first of all is determined by the properties of water those vary with portland cement hydration as well as with reagent (salt composition) adding. It is just this total influence which finally determines the properties of grouting mortars based on portland cement.

## CONCLUSION

1. It has been established that the properties of grouting mortars in the process of a salt composition introduction therein can be controlled through varying the properties of a tempering liquid (water) and, accordingly, the energy parameters of the process of cementing agent hydration. A decrease in the activation energy value for cement hydration has been experimentally observed.

2. A lightening additive is developed for the reduction of the density of grouting mortars on the basis of oil-well portland cement with the use of LBM product obtained by calcining the solid waste of soda manufacture (5–20 %) as well as the salt composition and soluble glass being in the ratio 3 : 1.

3. It has been demonstrated that the introduction of the salt composition into the tempering liquid allows one to control efficiently the structurization process for oil-well portland cement and the mixtures on its base.

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