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Research of the Retarder Hardening of Tamponation Solutions «DEXTRIN»

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Abstract

This article is performed owing to the necessity to extend the range of domestic retarding reagents of thickening and hardening of tamponation solutions while cementing oil and gas wells. The piece of work is dedicated to the research of technological properties of tamponation materials with additions of reagent "dextrin" under conditions simulating reservoir conditions. The goal was set to substitute import of the reagent base of tamponation solutions, in particular, of the retarding reagent of hardening of tamponation solutions of nitrilotrimethylphosphonic acid (NTPHA) manufactured in Russia. The following requirements are placed to the new retarding reagent of hardening and thickening of tamponantion solutions: wide temperature interval of application, cost effectiveness and environmental compatibility of application.

Keywords: *Tamponation solution; Technological properties of tamponation solutions; Rheological parameters; Hydration rate; Hardening retarder.*

1. Introduction

In connection with the limited range of reagents used to regulate the properties of tamponation solutions, it has become urgent to study the possibilities of using some industrial products as reagents for oil and gas industry. The main properties of tamponation solutions that require regulation include pumping time (pumpability), rheological parameters, the amount of water separation, etc.

The stone formed in the process of hardening (curing) should have high physical and mechanical parameters and low permeability. As a rule, when one of the parameters of the tamponation solution (stone) is changed, its other properties also change, and not always in the desired direction. According to the existing classifications [1-9], all known retarding reagents, which create shielding films around the cement particles in the tamponation solution and prevent the process of hydration hardening, can be divided into several main groups according to their chemical structure. Among them are as follows:

– oxy-aminocarboxylic acids and their salts;
– borates, phosphates;
– natural tannin products;
– derivatives of polysaccharides;
– sugars;
– lignin derivatives;
– humates;
– products based on vinyl and acrylic polymers and others.

Thinners of tamponation solutions include substances of the first six groups.

When studying the effect on the hardening times of tamponation solutions of organic substances, it was established that reagents containing hydroxyl and carboxyl groups, as well as sulfo-, amido-, and amino groups and belonging to mono- and dibasic carboxylic acids, sulfonic acids, amines, low-molecular amides, mono-, di- and triatomic aliphatic alcohols have little impact on the hardening time of cement mortars [10].

An increase in the degree of polymerization increases the retarding effect of organic substances on the hardening time of tamponation solutions. Polyhydric polyphenols, dibasic aliphatic oxyacids having at least two COOH groups, monobasic cyclic oxyacids having at least three hydroxyl groups, cyclic oxysulphonic acids with the total number of sulphogroups and hydroxides at least three to one benzene, naphthalene, or the second cyclic core.

In later classifications of retarding reagents [10-11], a group of organic phosphonic complexes additionally appears, which includes, in particular, oxyethylidene diphosphonic acid (OEDA) and nitrilotrimethylphosphonic acid (NTPHA).

Today, there is no consensus on the effectiveness of some reagents-additives to tamponation solutions, so we conducted research on some reagents influencing the hardening time of these solutions. In the process of searching for the optimal retarding reagents, several reagents were studied, in particular, molasses, green molasses, green molasses+NaCl, pentaerythritol, calcium gluconate, polyethylene glycol, polyacrylamide, citric, adipic, boric acids, etc., dextrin, sodium tripolyphosphate, borax.

The results of laboratory studies we obtained do not always coincide with the data given in the literary sources, and some reagents, for example, calcium gluconate, green molasses, borax, do not correspond at all to the characteristics given in the researchers' sources.

Dextrin reagent turned out to be the only one suitable for use as a retarder of thickening and hardening of tamponation solutions. **The goal** of this work is to study the retarding reagent for thickening and hardening of the "dextrin" tamponation solutions. To achieve this goal, the following research tasks are set:

– study of the technological properties of tamponation solutions containing the reagent "dextrin";
- study of impact of the "dextrin" reagent on the technological properties of the tamponation stone.

2. Experimental (Methods and materials)

When researching the technological properties of tamponation solutions, the main parameters: mobility, density, stability were determined in accordance with the standard methods (DSTU B V.2.7-86-99 Oil-well cements. Test methods). Water separation of tamponation solutions was studied using a measuring cylinder with capacity of 250 cm^3 with applied graduations [12]. The rate of solidification of tamponation solutions was determined on a Vick device, and the pumping time at temperatures and pressures simulating reservoirs was determined on KC-3 consistometer [13-14]. The rheological properties of tamponation solutions were studied on VSN-3 rotary viscometer [15-16].

During the formation of samples of tamponation stone in conditions simulating reservoirs (temperature, pressure), they were stored in autoclaves on AU-1-71-IE installation, designed by the Poltava branch of the UkrNDRI (temperature fluctuation ± 5^{o}C, pressure ± 2 MPa) [13, 17-19]. The cylindrical samples were formed with height and diameter of 3 cm.

The mechanical strength of tamponation stone was studied on PSU-10 laboratory press (accuracy of measurements ± 0.25 MPa) [10,13-14,17, 19].

Adhesion of tamponation stone to metal was determined using PSU-10 laboratory press and a special attachment for it [14]. The principle of measuring adhesion is based on the extrusion of a rod made of cement stone formed under appropriate thermodynamic conditions (temperature, pressure).

The gas permeability of tamponation stone was investigated using GK-5 installation according to the method described in the sources [10,13,17,20].

3. Results and discussion

We will describe the reagents and materials used in the investigation of the retarder of thickening and hardening of tamponation solutions.

The "dextrin" reagent belongs to the class of polysaccharides and is obtained during heat treatment of corn, potato starch or starch of other origin in the presence or absence of chemical agents. When dry starch is heated up to 200–250°C, its partial decomposition occurs and a mixture of less complex polysaccharides, in particular, dextrins, is obtained. Dextrin is also formed when starch is heated for 10 minutes at 180–200°C. After cooling, the resulting dextrin is easily separated from starch by dissolving it in water (dextrin has better solubility in water than starch, and therefore is easily separated from unreacted starch) [21–23].

Depending on the type of starch used to manufacture the reagent, dextrins are divided into corn and potato dextrins, and depending on the type of catalyst, they are divided into acid and alum dextrins. Dextrins are of the highest, first and second grade. The cheapest dextrin of the second grade is suitable as a reagent for slowing down the thickening and hardening of tamponation solutions. Corn dextrin is light yellow, potato dextrin is light brown. Both dextrins do not significantly differ in their effect on tamponation solution.

Dextrin dissolves in water by approximately 80% with the formation of a stable colloidal solution with pH = 6-7. It belongs to the fourth class of danger. Copper vitriol or sulphate in the anhydrous state of $CuSO_4$, which was used as an additive to the tamponation mixture, is a white, fine-crystalline substance. When water is absorbed, copper sulphate acquires a blue or bluish colour. Copper sulphate is made from copper scrap [24].

Copper sulphate dissolves well in water. From an aqueous solution, it crystallizes in the form of crystal hydrate $CuSO_4 \cdot 5H_2O$, known as copper sulphate (blue stone). At the temperature above 96°C, in equilibrium with an aqueous solution, it turns into the trihydrate: $CuSO_4 \cdot 3H_2O$.

Portland tamponation cement for moderate temperatures PCTI-100, as well as a mixture of PCTI-100 with acid ash of Kurakhiv and Ladyzhyn state regional power stations (SRPS), were studied as tamponation materials. Portland cement tamponation for moderate temperatures PCTI-100 is manufactured by industry in accordance with DSTU B V. 2.7-88-99 Portland tamponation cement. Technical specifications [25].

Ash from Kurakhiv SRPS is a dark grey powder with density of 1950-2050 kg/m^3 and specific surface area of 350-400 m^2/kg. Chemical composition by oxides, %: SiO_2 – 52.0 – 54.0; Al_2O_3 – 15.0 – 24.0; Fe_2O_3 – 17.0 – 23.0; CaO – 2.2 – 2.8; MgO – 2.0 – 3.0; K_2O+Na_2O – 1.7 – 3.4; l.m.c. – 3.4 – 3.7.

Ash from Ladyzhyn SRPS is a greenish-grey powder with density of 2300-2500 kg/m^3 and specific surface area of 230-250 m^2/kg.Chemical composition by oxides, %: SiO_2 – 50.0 – 55.0; Al_2O_3 – 21.0 – 24.0; Fe_2O_3 – 10.0 – 12.0; CaO – 2.0 – 2.2; MgO – 1.9 – 2.1; K_2O+Na2O – 1.8 – 2.3; l.m.c. - 5.1 - 5.4.

Among the main properties of tamponation solutions, density, mobility, water separation and time of thickening (pumpability) and hardening should be noted.

In the process of researching tamponation solutions containing the retarding reagent "dextrin", the influence of the reagent on the technological properties of solutions based on Portland tamponation cement PCTI-100 and tamponation mixtures PCTI-100 with acid ashes from thermal power station (TPS) in the ratio of ingredients - 50 : 50 mass fractions % was studied (Table 1). From Table 1, one can see that the "dextrin" reagent slightly increases the mobility of tamponation solutions, but at the same time reduces their stability. Addition of copper sulphate to the tamponation solution slightly reduces its mobility and significantly increases sedimentation resistance.

The graphs (Fig. 1 and 2) show that the reagent "dextrin" reduces the rheological parameters of tamponation solutions - dynamic shear stress (τ) by 1.3-3.3 times and plastic viscosity (η) by 1.1- 1.45 times. At the same time, the tendency of decreasing rheological parameters increases with increasing content of "dextrin" in the tamponation solution.

Table 1. Technological properties of tamponade solutions containing the retarding reagent "dextrin".

Composition of tamponation solution, mass shares %					V/M	Mobility, m	Water separation, ml	Dynamic shear stress, Pa	Plastic viscosity, Pa·c
PCTI-100	SA_K	SA_L	Dextrin	Copper sulphate					
100	–	–	–	–	0.50	0.20	3.75	2.0	0.045
100	–	–	0.3	–	0.50	0.23	5.0	1.1	0.040
100	–	–	0.5	–	0.50	0.25	8.75	0.6	0.031
50	50	–	–	–	0.55	0.19	1.25	10.1	0.075
50	50	–	0.3	–	0.55	0.21	1.25	7.5	0.069
50	50	–	0.5	–	0.55	0.22	6.25	5.6	0.060
50	50	–	0.3	0.3	0.55	0.19	1.25	8.4	0.078
50	50	–	0.4	0.4	0.55	0.18	0	9.9	0.085
50	–	50	–	–	0.47	0.22	3.75	5.6	0.058
50	–	50	0.3	–	0.47	0.23	4.25	4.1	0.050
50	–	50	0.5	–	0.47	0.24	10.5	3.2	0.041
50	–	50	0.3	0.3	0.47	0.22	3.75	4.8	0.053
50	–	50	0.4	0.4	0.47	0.20	2.5	5.0	0.064

Theoretically, a significant drop in τ leads to a decrease in the boundary of the transition of the tamponation solution flow regime in the closed space from laminar to turbulent, which contributes to a better replacement of the flushing liquid with the tamponation solution. In Table 2 the main results of research on the pumpability of tamponation solutions containing the retarding reagent "dextrin" are shown.

From the given data, it can be seen that the reagent "dextrin" is a retarder of hardening of tamponation solutions at temperatures up to 100°C. Addition of the reagent "dextrin" varies from 0.1% of the mass of dry material at 75°C to 0.5% at 100°C. A further increase in the concentration of the reagent "dextrin" in the tamponation solution does not lead to an extension of its pumping period.

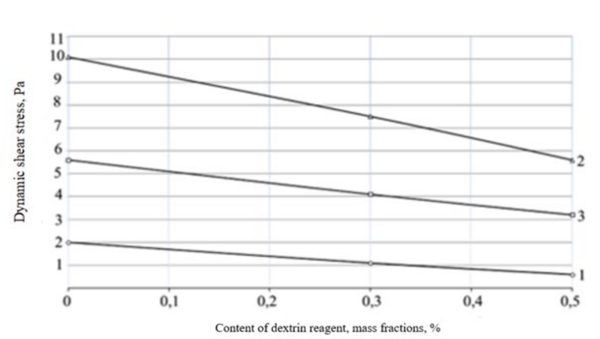

Figure 1 Dependence of dynamic shear stress on the content of the "dextrin" reagent in the tamponation solution
1 – PCTI-100, V/M = 0.5; 2 – PCTI-100 : SA_K – 50 : 50, V/M = 0.55;
3 – PCTI-100: SA_L – 50 : 50, V/M = 0.47

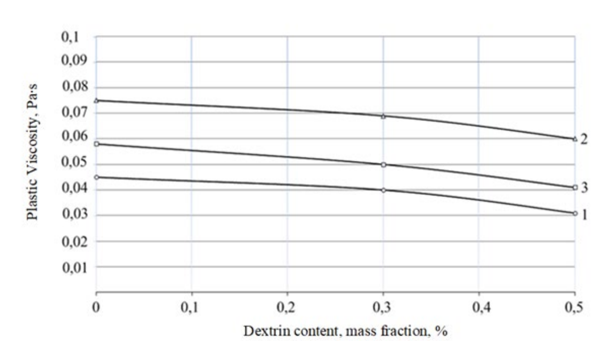

Figure 2. Dependence of plastic viscosity on the content of the reagent "dextrin" in the tamponation solution
1 – PCTI-100, V/M = 0.5; 2 – PCTI-100 : SA_K – 50 : 50, V/M = 0.55;
3 – PCTI-100 : SA_L – 50 : 50, V/C = 0.47

It is known from the practice of cementing wells that some reagents can enhance the effect of others, for example, at elevated temperatures, so-called synergistic effect [10].

Copper sulphate ($CuSO_4 \cdot 5H_2O$) was chosen to extend the thermal range of the retarding effect of the reagent "dextrin". Studies of tamponation solutions containing the reagent "dextrin" in combination with copper sulphate have yielded positive results.

Table 2. Time of pumping (pumpability) and hardening of tamponation solutions containing retarding reagent "dextrin".

Composition of tamponation mixture, mass shares %						Pumpability, h.-min.			Longing time, h.-min., by t=75°C P=0,1 MPa	
PCTI100	SA_K	SA_L	Dextrin	Copper sulfate	V/M	t=75 °C P=40 MPa	t=100 °C P=60 MPa	t=120 °C P=80 MPa	start	end
100	–	–	–	–	0.50	1-10	0-50	–	1-30	2-00
100	–	–	0.10	–	0.50	4-30	1-20	–	6-00	8-00
100	–	–	0.50	–	0.50	5-40	2-50	–	8-00	–
50	50	–	–	–	0.55	1-30	1-00	–	1-50	2-40
50	50	–	0.05	–	0.55	3-00	1-20	–	3-50	4-50
50	50	–	0.10	–	0.55	6-00	1-30	–	8-00	–
50	50	–	0.50	–	0.55	6-00	3-30	2-00	8-00	–
50	50	–	0.30	0.30	0.55	6-00	5-10	3-00	8-00	–
50	50	–	0.40	0.40	0.55	6-00	6-00	4-10	8-00	–
50	–	50	–	–	0.47	1-30	1-00	–	1-40	2-30
50	–	50	0.05	–	0.47	3-20	1-40	–	4-05	5-20
50	–	50	0.10	–	0.47	6-00	1-50	–	8-00	–
50	–	50	0.50	–	0.47	6-00	4-10	2-30	8-00	–
50	–	50	0.30	0.30	0.47	6-00	5-25	3-15	8-00	–
50	–	50	0.40	0.40	0.47	6-00	6-00	4-25	8-00	–

From the research results (Table 2), one can see that when using a combined additive - the reagent "dextrin" + copper sulphate, the temperature range of the thickening and hardening retarder of tamponation solutions "dextrin" expands up to 120°C.

Today, strength is the main indicator of the quality of tamponation stone. Table 3 shows the main physical and mechanical properties of tamponation stone containing the "dextrin" reagent. When considering them, some regularities can be noted. In particular, at the temperature of 75°C, after 2 days of hardening of the tamponation solution, a drop in strength is observed in samples containing the reagent "dextrin". With the combined addition of the reagent "dextrin" + copper sulphate, after 2 days of hardening, the stone, as a rule, has not formed yet.

When the temperature rises up to 100°C after 2 days of hardening of the tamponation solution, the absolute strength values increase slightly. With the combined addition of the reagent "dextrin" + copper sulphate, after 2 days of hardening, the stone has time to form.

When the temperature increases up to 120°C after 2 days, there is a significant intensification of hardening of the tamponation solution and, as a result, the stone strength indicators increase significantly. At the same time, the strength indicators of the stone containing reagent "dextrin" are almost equal to the samples that do not contain reagent "dextrin", and the samples with a combined additive (reagent "dextrin" + copper sulphate) have an excess of strength over the samples that do not contain the reagent – retarder "dextrin".

Therefore, addition of the retarding reagent "dextrin" to the tamponation solution at the first stages of hardening slows down hydration and the process of stone formation.

As a rule, the gas permeability of tamponation stone decreases with increasing temperature. As the strength of the stone increases, gas permeability naturally decreases. Table 4 shows data of the adhesion to metal tamponation stone containing the "dextrin" reagent.

Table 3. Technological properties of tamponation stone containing retarding reagent "dextrin".

Composition of tamponation mixture, mass shares %						Strength of stone in 2 days, MPa			Gas permeability in 2 days, μm^2×10^{-3}		
PCTI-100	SA_K	SA_L	Dextrin	Copper sulphate	V/M	t=75 °C P=40 MPa	t=100 °C P=60 MPa	t=120 °C P=80 MPa	t=75 °C P=40 MPa	t=100 °C P=60 MPa	t=120 °C P=80 MPa
100	–	–	–	–	0.40	19.5	15.2	–	0.80	1.14	–
100	–	–	0.5	–	0.40	2.1	10.1	–	–	2.15	–
100	–	–	–	–	0.50	12.8	9.3	–	1.75	2.10	–
100	–	–	0.5	–	0.50	0.8	3.7	–	–	3.25	–
50	50	–	–	–	0.55	10.0	15.2	18.0	1.55	1.01	0.88
50	50	–	0.3	–	0.55	1.0	14.4	15.7	–	1.43	1.05
50	50	–	0.5	–	0.55	0.3	12.8	15.5	–	1.61	1.11
50	50	–	0.3	0.3	0.55	–	11.7	18.7	–	2.08	0.95
50	50	–	0.4	0.4	0.55	–	7.9	22.8	–	3.01	0.53
50	–	50	–	–	0.47	12.1	13.2	20.2	1.57	1.60	0.75
50	–	50	0.3	–	0.47	3.6	10.1	16.1	3.85	3.12	1.30
50	–	50	0.5	–	0.47	1.2	6.9	14.0	–	3.70	1.40
50	–	50	0.3	0.3	0.47	–	8.8	19.9	–	2.48	0.46
50	–	50	0.4	0.4	0.47	–	5.7	24.8	–	3.25	0.33

Table 4. Adhesion to the metal of tamponation stone containing the retarding reagent "dextrin".

Composition of tamponation mixture, mass shares %						Adhesion to metal in 2 hours, MPa		
PCTI-100	SA_K	SA_L	Dextrin	Copper sulphate	V/M	t=75 °C P=40 MPa	t=100 °C P=60 MPa	t=120 °C P=80 MPa
100	–	–	–		0.50	3.3	5.3	–
100	–	–	0.5		0.50	1.0	4.0	–
50	50	–	–	–	0.55	4.4	5.2	13.0
50	50	–	0.5	–	0.55	1.5	5.7	7.5
50	50	–	0.3	0.3	0.55	–	7.3	12.6
50	–	50	–	–	0.47	5.0	5.4	11.5
50	–	50	0.3	–	0.47	2.9	4.6	12.4
50	–	50	0.5	–	0.47	2.0	5.0	12.7
50	–	50	0.3	0.3	0.47	–	5.5	14.6

At a temperature of 75°C, there is a decrease in the adhesion to metal of the tamponation stone containing the reagent "dextrin" compared to stone samples that do not contain reagent "dextrin". As the temperature increases, the adhesion to the metal of tamponation stone containing reagent "dextrin" almost equalizes with the stone samples that do not contain reagent "dextrin", and in the samples with a combined additive (reagent "dextrin" + copper sulphate), an excess of the adhesion to the metal is observed over stone samples that do not contain the "dextrin" reagent.

In the conducted studies, the relationship between the strength of the tamponation stone and the adhesion of the stone to the metal is monitored - as the strength increases, the adhesion index also increases (Table 4).

The industrial introduction of the retarding reagent "dextrin" took place during the cementing of the second section of the technical column in well No. 8-Krasnozavodska under downhole conditions: reservoir temperature (t) = 65°C, reservoir pressure (P) = 68 MPa. For cementation, a cement-ash mixture was used: PCTI-100: ZK_L - 50:50 with the addition of the reagent "dextrin"- 0.07% of the mass of dry binding material. The density of the tamponation solution was 1730 kg/m^3, the pumping time on the KC-3 consistometer was 3 hours 45 minutes. Cementing was successful.

Also, the retarding reagent "dextrin" was introduced during the installation of a cement bridge in the interval 3230 - 3150 m in the well No. 4-Selyukhivska under well conditions: t = 65^{o}C, P = 47 MPa. For the installation of a cement bridge, a cement-ash mixture was used: PCTI-100: ZK_L – 50: 50 with the addition of the reagent "dextrin" – 0.08% of the mass of the dry binding material. The density of the tamping solution was 1730 kg/m3, the pumping time on the consistometer KC-3 – 4 hours 15 minutes. The cement bridge was successfully installed on the first attempt.

3. Conclusions

A study of the technological properties of tamponation solutions containing the reagent "dextrin" established that the reagent "dextrin" is an optimal retarder of tamponation solutions, which slows down thickening and hardening of solutions at temperatures up to 100^{o}C, and in combination with copper sulphate at temperatures up to 120^{o}C. Reagent "dextrin" slightly reduces the rheological parameters of tamponation solutions (dynamic shear stress and plastic viscosity). The value of adding "dextrin" to the tamponation solution, depending on the reservoir conditions of use, is in the range of 0.05 to 0.5% of the mass of dry material.

The study of the effect of "dextrin" on the technological properties of the tamponation stone allows us to conclude that "dextrin" slows down the hydration of cement in the early stages of hardening. However, the reagent "dextrin" does not generally reduce the rate of growth of the technological properties of the tamponade stone (strength, adhesion, gas permeability) during the period of waiting for the cement to harden (48 hours). Cement stone with additives of the reagent "dextrin" has high physical and mechanical indicators and low permeability.

Competitive advantages from the introduction of the reagent "dextrin" are due to its relatively low price, environmental friendliness (IV hazard class). The introduction of a new reagent of multifunctional action "dextrin" will ensure high quality of well fastening.

Symbols

NTPHA	*nitrilotrimethylphosphonic acid;*
DSTU	*State Standard of Ukraine;*
PV UkrDGRI	*Poltava branch of the Ukrainian State Geological Exploration Institute;*
PCTI-100	*tamponation Portland cement for moderate temperatures;*
SDPS	*State District Power Station;*
l.m.c.	*oss of mass during calcination;*
TPP	*thermal power plant;*
SA_K	*sour ash of Kurakhiv SDPS;*
SA_L	*sour ash of Ladyzhyn SDPS;*
W/M	*water mixture ratio.*

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