

Research of the Superplasticizer of Tamponation Solutions «C-3»

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Abstract

This study aims to investigate a domestically produced plasticising reagent for cementing slurries. The research focuses on evaluating the technological properties of plugging materials with additives of plasticising reagent C-3 under simulated formation conditions. The C-3 plasticising reagent for cementing slurries is required to demonstrate a wide temperature range of applicability, cost efficiency and environmental safety.

Keywords: *Cementing slurry; Cement slurry spreadability; Cement slurry density; Cement stone strength; Superplasticiser C-3.*

1. Introduction

In recent years, the drilling depths of oil and gas wells have reached 6,000 to 7,000 m and beyond. Consequently, cement slurries are now subject to more stringent performance requirements. They must exhibit favourable rheological properties to reduce pressures when squeezing cement slurry and to prevent complications during casing cementing operations. Furthermore, reduced rheological properties will ensure a turbulent flow for cement slurries, which is the most effective flow regime for the complete displacement of flushing fluid by the cement slurry. The use of efficient plasticisers as admixtures in cement slurries provides an effective approach to addressing these challenges.

Plasticisers are chemical reagents used to process cement slurries, enhancing their physical and chemical properties, plasticity, flowability and strength. These surface-active substances primarily function by altering the molecular links within the cement solution. The properties of plasticisers include the ability to decrease slurry viscosity and improve its flowability. In addition, plasticisers enable a reduction in the water-to-cement ratio (W/C), i.e. the percentage of water in the cement solution.

Plasticisers are classified into four main groups:

- 1) melamine formaldehyde resins;
- 2) products derived from the condensation of formaldehydes with naphthalene sulfonic acid;
- 3) purified lignosulphonates (free from sugars);
- 4) polycarboxylate-based group.

This classification represents the most widely used types of plasticisers. However, other, less common classification systems also exist. For example, some groups include naphthalene derivatives, urea-formaldehyde resins, formaldehyde derivatives, dicarboxylic acids, polyethylene glycols, cellulose ethers, aromatic hydrocarbons, fatty alcohols, and carboxylates, among others.

A broader classification distinguishes six groups of plasticisers: *oxy-amino carboxylic acids* and their salts (citric, tartaric, trioxylglutaric, gluconic, heptanoic, ethylenediaminetetraacetic, 2,4-dioxybenzoic, 3,4,5-trioxybenzoic, etc.); sugars (sucrose, glucose, galactose, sodium glucoheptonate, etc.); borates and phosphates (boric acid, borax, sodium hexametaphosphate, pyrophosphate, trisodium phosphate, etc.); lignin derivatives (activated hydrolytic lignins (nitrolignin, chlorolignin, sulphonated nitrolignin, H₂O₂-oxydised lignin, etc.), lignosulphonates (calcium lignosulphonate, ferrochrome lignosulphonate (FCLS), chrome lignosulphonate (oxyl), evaporated sulphite waste liquor (ESWL); natural tannin-based products and synthins (quebracho extract, chestnut extract, sulphite-bark reagent (silkor), synthin-5, pitch reagent (pitch), polyphenolic wood-chemical reagent (PPWC), tanning agent D-4, etc.); humates (sulphonated nitrohumate reagent (SNHR), carbon-alkaline reagent (CAR), oxidised humate reagent (OHR) [1-15].

During the 1970s, a wide range of plasticising agents was employed in various types of cement slurries. These included sulphite-alcohol liquor (SAL), lignosulphonates such as ESWL, ESWL+ CaCl₂, hydrolysed polyacrylonitrile, CMC, GKZh-10, Trilon B (disodium salt of ethylenediaminetetraacetic acid), complex reagents such as furfural+liquid glass (FLG) and furfural+ESWL (FES), polysaccharide dextrin, xylose, alkaline-treated petasan products with present chromates, ferrochrome lignosulphonates (FCLS) and others, admixtures based on resorcinol-formaldehyde resin FR-50A [1-2,6].

The American company Halliburton developed and applied polymeric reagents CFR-1 and CFR-2 for high-temperature conditions above 100°C. These reagents halve the critical velocity (the speed at which the flow transitions from a structured to a turbulent regime), while reducing pressure losses by 66% [1,6]. In the United States, admixtures based on sulphonated complex anhydrides were employed to enhance the hydrodynamic performance of cement slurries during the rapid pumping of large volumes [1,7].

In the early 1970s, concrete admixtures known as superplasticisers were developed in Germany, Japan and the United Kingdom. These admixtures proved far more effective than conventional plasticisers. The addition of a few kilograms of superplasticiser per cubic metre of concrete can increase flowability severalfold, improve the physical and chemical properties of the concrete and reduce overall cement consumption. Germany developed superplasticisers based on water-soluble anionic melamine resins, such as Melment F10, Melment F300, and Melment L10 [16-17].

Superplasticisers are primarily based on sulphonated melamine-formaldehyde or naphthalene-formaldehyde condensation resins, as well as modified lignosulphonates. Their incorporation can reduce the maximum water consumption in concrete by 20–35%, resulting in an increase in one-day compressive strength of 50–75% [18]. The goal of the study is to investigate the superplasticising reagent C-3 for cement slurries.

To achieve this goal, the following objectives were defined:

- to research the technological properties of cement slurries containing the C-3 superplasticiser;
- to research the technological properties of cement rock containing the C-3 superplasticiser.

2. Experimental (Methods and materials)

2.1. Materials

The superplasticiser C-3 is a sodium naphthalene sulfonate, produced by the condensation of formaldehyde with naphthalene sulfoxy acids. It is a mixture of sodium salts of polymethylene naphthalene sulfonic acids with varying molecular weights. C-3 is produced either as a dry substance or as an aqueous solution and belongs to the class of plasticising water-reducing modifiers, i.e. plasticising thinners. The dry C-3 reagent is a light reddish-brown powder, readily soluble in water in any proportion. It contains less than 0.2% chloride compounds, has a density of 1.08 g/cm³ at 20°C, and a pH of 7–9.

The liquid form of C-3 plasticiser is a dark brown substance with a faint characteristic odour, a density of 1.18 g/cm³ at 20°C, and a water content not exceeding 68.0%.

The composition and properties of the C-3 superplasticiser remain unchanged under normal storage conditions for up to one year. In this study, the dry form of C-3 reagent was used as the superplasticiser.

The cementing materials investigated included packing Portland cement for moderate temperatures, grade PTsT1-100 (DSTU ББ.2.7-88-99. Packing cements. Technical specifications) and slag-sand cement ShPTsS-120 (TUU B.2.7-26.5-32312060-002:2007. Modified blended packing slag-sand cement ShPTsS-120M) [19].

2.2. Methods, research devices and equipment

The technological properties of cement slurries, including density, flowability and thickening time, were determined using standard methods. Density was measured with a pycnometer, flowability was assessed using a slump cone, and thickening time was determined using a KC-5 consistometer [2,20]. Cement stone samples were formed in specially designed containers (baths) at controlled temperatures [21]. Specimens were formed as cylinders with a height and diameter of 3 cm, and as beams measuring 0.04×0.04×0.16 m. Moulds were arranged in batteries by operating volume of the autoclaves and placed in a preheated environment.

Autoclaving was performed using the AU-1-71-IE unit, developed by the Poltava branch of UkrSGRI. The system is rated for a maximum operating pressure of 100 MPa and a maximum temperature of 523 K. The autoclaving unit comprises autoclaves, electrical and hydraulic equipment, monitoring and documenting instruments (for temperature, pressure and current in the heating elements) [21]. The mechanical strength of the cement stone was evaluated using standard methods. Flexural strength was measured according to DSTU ББ.2.7-86-99 on a testing device for beam specimens under bending, while compressive strength was determined using a PSU-10 press [21-24].

3. Results and discussion

Plasticiser is a highly effective additive in cement slurries. Its functional principle lies in dispersing binder particles, which significantly improves the slurry's rheological properties. Let us consider the plasticisation process of the cement mixture on its molecular level. The plasticiser molecules within the cement slurry are highly flexible and capable of conformational changes. Their enhanced mobility allows them to combine rapidly with the molecules of the cement slurry, resulting in a more homogeneous structure. This leads to increased flowability of the cement slurry and greater strength in the set cement.

The mechanism of action of the C-3 plasticiser involves adsorption onto the surface of hydrating cement particles, forming a monomolecular layer that reduces internal friction within the cement-water system. It also promotes the peptisation of cement particle flocs. The increase in specific surface area due to peptisation intensifies hydration, enhancing the strength of the cement stone or concrete even with no losses in water content within the system [25]. With this principle in mind, studies were conducted to investigate the effect of C-3 superplasticiser concentration on the mobility of cement dispersions. The research showed that low dosages, the superplasticiser adsorbs onto active centres of binder particles, covering only some portions of their surface and having minimal effect on internal friction. As the superplasticiser's concentration increases, internal friction decreases, and flowability improves until the particle surfaces are fully coated with a monomolecular layer of C-3. Further increases in C-3 concentration have no effect on mobility. These results are consistent with observations that, during the early stages of hydration, C-3 superplasticiser forms hydrated adsorptive films, and the low strength of contacts between polymer molecules reduces friction, resulting in the observed plasticising effect.

The plasticising effect of superplasticiser additives, which act simultaneously as surfactants, dispersants and polyelectrolytes, is based on the adsorption of oligomeric molecules on the surfaces of cement particles [26]. These molecules, composed of both hydrophilic and hydrophobic parts, are expected to facilitate the passage of water and hydration products through the hydrophilic areas while blocking interactions at the hydrophobic areas during the binder-water interaction.

Table 1 presents the research results on the effect of the C-3 superplasticiser on the flowability of cement slurries based on PTsTI-100 and ShPTsS-120 at standard temperature and pressure conditions (NTP – 20°C, 1 atm). The results (Table 1, Fig. 1) indicate that the addition of a small amount of C-3 (0.4–0.43% of the binder mass) significantly enhances slurry workability. Slurry density also increases due to the reduction in the W/C associated with the plasticising (thinning) effect of C-3.

Table 1. Effect of the C-3 superplasticiser on the properties of cement slurries.

Cement Slurry	C-3 Additive, %	W/C	Cement Slurry Flowability, m	Cement Slurry Density, kg/m ³
PTsTI-100	–	0.50	0.210	1850
PTsTI-100	0.5	0.40	0.215	1960
PTsTI-100	0.6	0.40	0.220	1960
PTsTI-100	0.7	0.40	0.230	1960
ShPTsS-120	–	0.43	0.185	1820
ShPTsS-120	0.3	0.40	0.185	1830

The study demonstrated that the C-3 superplasticiser has minimal impact on the setting time of cement slurries. Consequently, in subsequent experiments, known setting retarders – including alcohol-sulphite lye concentrate (ASLC), synthetic tartaric acid (STA) and bichromate – were added to the mixing water. Table 2 summarises the effects of the C-3 superplasticiser, in combination with these retarders, on the technological properties of ShPTsS-120 cement slurries.

The results (Table 2) indicate that the addition of a small amount of C-3 (0.3–0.43% of the binder mass) markedly improves slurry mobility. Slurry density also increases due to the reduction in the W/C resulting from the plasticising action of C-3. Moreover, the thickening time at $t = 120^\circ\text{C}$ and $P = 40 \text{ MPa}$, measured at consistencies of 30 CCU and 50 CCU, is significantly extended by the inclusion of the setting retarders (ASLC, bichromate and STA).

Table 2. Effect of the C-3 superplasticiser on the technological properties of cement slurries.

Cement Slurry	Reagents	Reagent content, %	W/C	Flowability, m	Density, kg/m ³	Thickening time, at $t=120^\circ\text{C}$, $P=40 \text{ MPa}$, hrs-min	
						to 30 CCU	to 50 CCU
ShPTsS-120	–	–	0.43	0.185	1820	0-43	0-50
ShPTsS-120	C-3	0.80	0.30	0.185	2040	0-35	0-38
ShPTsS-120	C-3	0.80	0.43	0.220	1820	3-10	3-20
	ASLC	0.25					
ShPTsS-120	bichromate	0.20	0.30	0.200	2040	3-35	3-50
	C-3	0.80					
ShPTsS-120	ASLC	0.30	0.30	0.185	2020	1-45	2-00
	bichromate	0.30					
ShPTsS-120	C-3	0.80	0.30	0.185	2020	2-15	2-40
	STA	0.20					
ShPTsS-120	C-3	0.80	0.30	0.185	2020	2-15	2-40
	STA	0.30					

Table 3 presents the results of studies on the effect of the C-3 superplasticiser on the properties of cement slurries and cement stone. The results (Table 3, Fig. 2) indicate that the addition of a small amount of C-3 (0.2–2.0% of the binder mass) significantly improves slurry mobility. The physical and mechanical properties of cement stone at 75°C and 0.1 MPa (after 2 days of setting) show that, with an increase in C-3 content from 0.5% to 1.0% of the binder mass, the stone’s flexural strength slightly increases at the same W/C ratio.

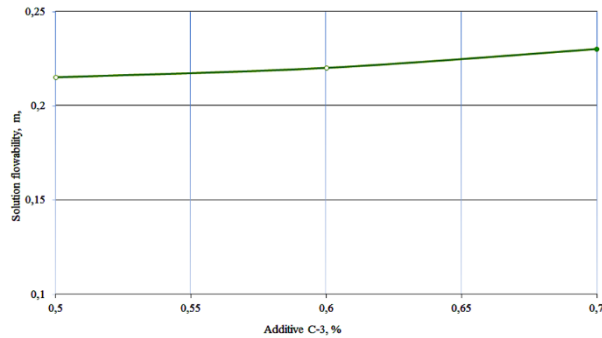
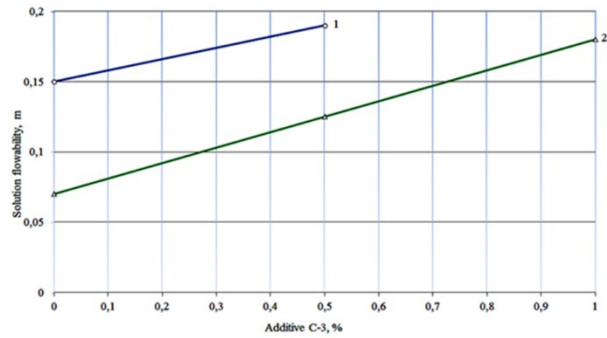


Figure 1. Effect of the C-3 superplasticiser on the flowability of the cement slurry based on PTsTI-100 at W/C = 0.40.



Effect of the C-3 Superplasticiser on the Flowability of Cement Slurry Based on PTsTI-100; 1 – W/C = 0.36; 2 – W/C = 0.28.

Table 3. Effect of the C-3 superplasticiser on the properties of cement slurries and cement stone.

Cement slurry	C-3 Addition, %	W/C	Flowability, m	Cement stone strength, MPa, at $t=75^{\circ}\text{C}$, $P=0.1$ MPa, 2 days of setting	
				Flexural	Compressive
PTsTI-100	–	0.40	0.135	8.93	41.8
PTsTI-100	0.25	0.39	0.180	8.77	39.5
PTsTI-100	–	0.36	0.150	9.57	46.6
PTsTI-100	0.50	0.36	0.190	10.60	44.0
PTsTI-100	0.75	0.31	0.195	10.13	49.2
PTsTI-100	–	0.28	0.070	11.23	51.4
PTsTI-100	1.00	0.28	0.180	11.43	69.4
PTsTI-100	–	0.25	–	11.37	68.0
PTsTI-100	1.50	0.25	0.190	10.47	63.6
PTsTI-100	–	0.24	–	10.13	69.4
PTsTI-100	2.00	0.24	0.200	11.10	52.4

Promising directions for future research include the application of C-3 cement slurries in horizontal wells [27-30], as well as in complex oil, gas and geothermal wells that integrate fluid extraction with geothermal heat recovery [31-34].

4. Conclusions

Investigations of the technological properties of cement slurries containing the C-3 superplasticiser admixtures demonstrated that the addition of a small amount of C-3 (0.25–2.0% of the binder mass) significantly improves slurry mobility. The research on the technological properties of cement stone containing C-3 superplasticiser admixtures showed that increasing the C-3 content from 0.5% to 1.0% of the binder mass enhances both the flexural and compressive strength of the resulting cement stone.

Symbols

PTsTI-100	packing Portland cement for moderate temperatures
DSTU	State Standard of Ukraine
ShPTsS-120	heat-resistant packing cement based on granulated metallurgical slag
TUU	Technical Specifications of Ukraine
UkrSGRI	Ukrainian State Geological Research Institute
W/C	water-to-cement ratio
t	temperature, $^{\circ}\text{C}$
P	pressure, MPa
CCU	conventional consistency units

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