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STUDYING THE EFFECT OF SYNTHESIZED POLYCARBOXYLATE SUPERPLASTICIZER ON CEMENT STONE AND CEMENT PASTE

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Abstract

As a result of the rapid development of the construction industry, there was a widespread need for plasticizers used for various purposes. However, such shortcomings as high cost and complex synthesis process have limited the widespread use of modern superplasticizers. In this article, we conducted an experimental study of a highly effective superplasticizer based on polycarboxylate. Using a special process, monomer, and macromonomer, a new superplasticizer based on polycarboxylate was synthesized. The results showed low cost and high efficiency, indicating the possibility of using this new superplasticizer in high-quality concrete. Furthermore, it was shown that commercial concrete with the addition of this plasticizer loses less than 15% of its cone sediment within 2 hours.

Keywords: *Polycarboxylate superplasticizer, macromonomer, monomer, concrete mix, cement, flowability, flexural and compressive strength*

1. Introduction

Polycarboxylate superplasticizer has now become the most important product in the concrete mix market due to its remarkable advantages, such as high water reduction rate, better compatibility with cement, and better precipitation protection indicators (Huang X. Q., Li X. R., Zhang D. W., et al., 2017). The market demand for polycarboxylate superplasticizer is growing with the planning and implementation of projects in the field of atomic energy, water conserva-

tion, the construction of bridges, tunnels, and other major infrastructure, especially railway passenger lines. Polycarboxylate superplasticizer is a unique surfactant whose molecules contain carboxyl-bonded copolymers. Due to this unique molecular structure, it possesses many remarkable properties: low utilization, rapid reduction of water content, significant increase in strength at the initial stage, good preservation of bending, absence of retarding agents, minimal effect on concrete shrink-

age, and the addition regime does not affect its effectiveness (Tian W., Liu X. J., Fan L., 2016; Xu C., Huang Y. Q., Weng W. X., et al., 2011). With the development of the economy and the diversification of infrastructure projects, higher demands are placed on the effectiveness of polycarboxylate superplasticizers (Sakai, Etsuo, Ishida, et al., 2006). At the same time, due to the deterioration of the quality of cement, sand, gravel, and other materials in the market, the use of polycarboxylate superplasticizers becomes very sensitive to changes in the quality of concrete raw materials. Especially in winter, during low-temperature construction, the phenomenon of slow water separation in concrete with polycarboxylate superplasticizer is very common, which places even higher demands on the effectiveness of polycarboxylate superplasticizers (Li C. Z., Wang D. M., Song S. M., et al., 2005; Jiang J. Y., Gai G. Q., Kong L. W., et al., 2015). Currently, water-reducing agents available in the market cause early bleeding due to their high water reduction rate, while the stability of the retarding agent requires the development of a substance capable of effectively blocking concrete with high water retention capacity and stable flowability. In this work, starting with the molecular structure design

Song Z. B., Yao Y., Li T., et al., 2017; Li S., Wen Z. Y., 2009; Song Z. B., 2016; Chen M., 2015; Ismoilov F. S., Djalilov A. T., Karimov M. U., 2023), polycarboxylic acid molecule can effectively retain concrete by introducing a glucose ether structure into the polycarboxylic acid molecular chain and replacing the glucose ether structure with phosphate, amine, and carboxylate structures. Moisture controls the hydrolysis rate of the ether group, preventing bleeding while maintaining stable concrete flowability.

Methodology

When synthesizing a polycarboxylate superplasticizer based on HPEG, 180 liters of distilled water are added to a stainless steel reactor, the solutions added to reactor 1 are mixed with 120 g of HPEG, 12 g of acrylic acid, and 0.6 g of mercaptopropionic acid per 180 gr of water and stirred at a rate of 50 times/min until a homogeneous mass is

achieved, and the supply from the first reactor to the second reactor continues for 2 hours using a special pump designed for 3–4 hours, resistant to the chemical environment. Reactor 2 is constantly stirred up to 100 times/min. After loading the reagents from part 1 into reactor 2, we introduce a 60% hydrogen peroxide solution from another inlet of reactor 2 into the reaction mixture for 40 minutes. After the hydrogen peroxide solution is finished, stir for 45 minutes. During the synthesis process, a copolymerization reaction of monomers occurs. The dry residue content and pH of the resulting product are determined.

Table 1. Amount of substances for superplasticizer synthesis

No.	Substances used in synthesis	Quantity (g)
1 partial solution		
1.	HPEG	120
2.	water	180
3.	acrylic acid	12
4.	MPA-3: 95%	3.6
2 – partial solution		
5.	Water	180
6.	APS 99%	0.5

Result and analysis

Currently, cement concrete remains the main building material, therefore, the problems of increasing its technological strength and durability remain relevant. Among the additives widely used in concrete and reinforced concrete production, plasticizers occupy the first place.

The dependence of HPEG-based polycarboxylate superplasticizer yield on time is presented in Figure 2. As can be seen from Figure 2, the highest profitability compared to others is obtained when the ratio of acrylic acid to HPEG is 8:1. The synthesis process takes 5 hours. In an experiment lasting more than 5 hours, the superplasticizer yield decreases. This can be explained by the fact that the substances taken for raw materials are in various aggregate states for 5 hours or more. Because the presence of interacting substances in two different states of aggregation reduces their interaction.

Figure 1. HPEG-based polycarboxylate superplasticizer dependence of productivity on time

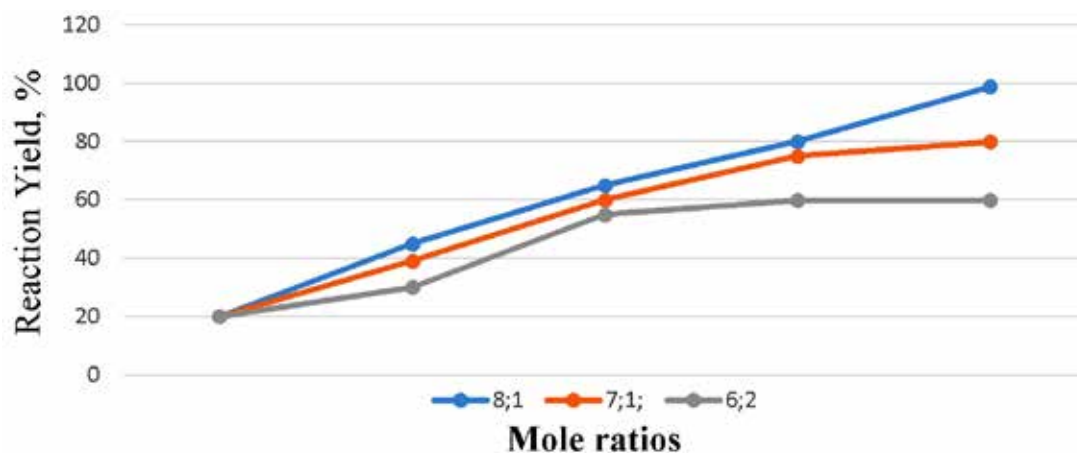


Figure 2. Dependence of the yield of HPEG-based polycarboxylate superplasticizer on the molar ratio

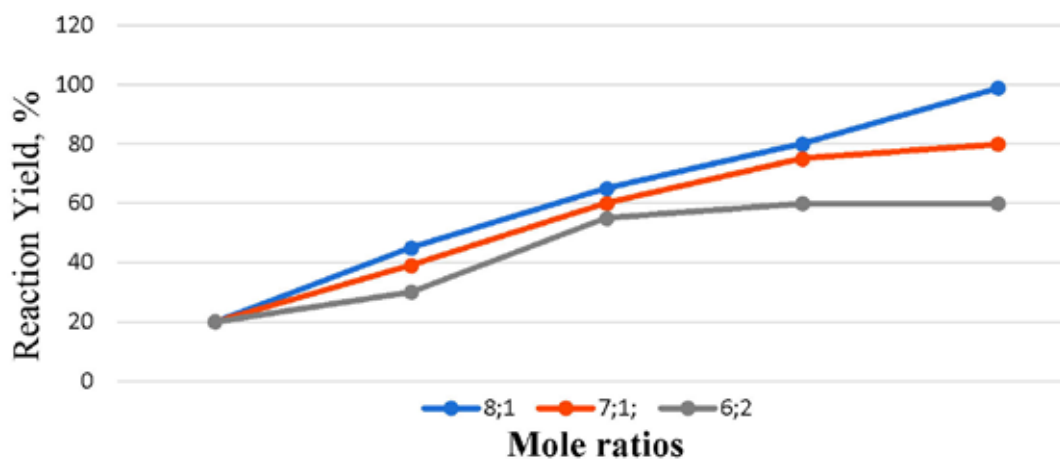


Figure 2. Dependence of the yield of a polycarboxylate superplasticizer obtained on the basis of HPEG on the molar ratio of acrylic acid and HPEG. As can be seen from Figure 2, the optimal ratio of components in the production of a polycarboxylate superplasticizer is 8:1, while the profitability of the superplasticizer is 90%. The highest dry residue is obtained at a ratio of the initial products 7:1 and 6:2, but the plasticizing effect of the obtained superplasticizers is low. Reaction time also plays an important role in the production of superplas-

ticizers. To choose the optimal reaction yield temperature, all three initial product ratios were selected. As can be seen from the figure, as a result of carrying out the reaction under optimal conditions for 5 hours, a 90% yield of superplasticizer is obtained. Under such conditions, further continuation of the reaction leads to a decrease in yield. This is due to the intensification of parallel reactions (for example, binding, decomposition, intermolecular interaction) leading to a decrease in the plasticizing effect of superplasticizing additives.

Table 2. Influence of the molar ratio of substances and time on product yield

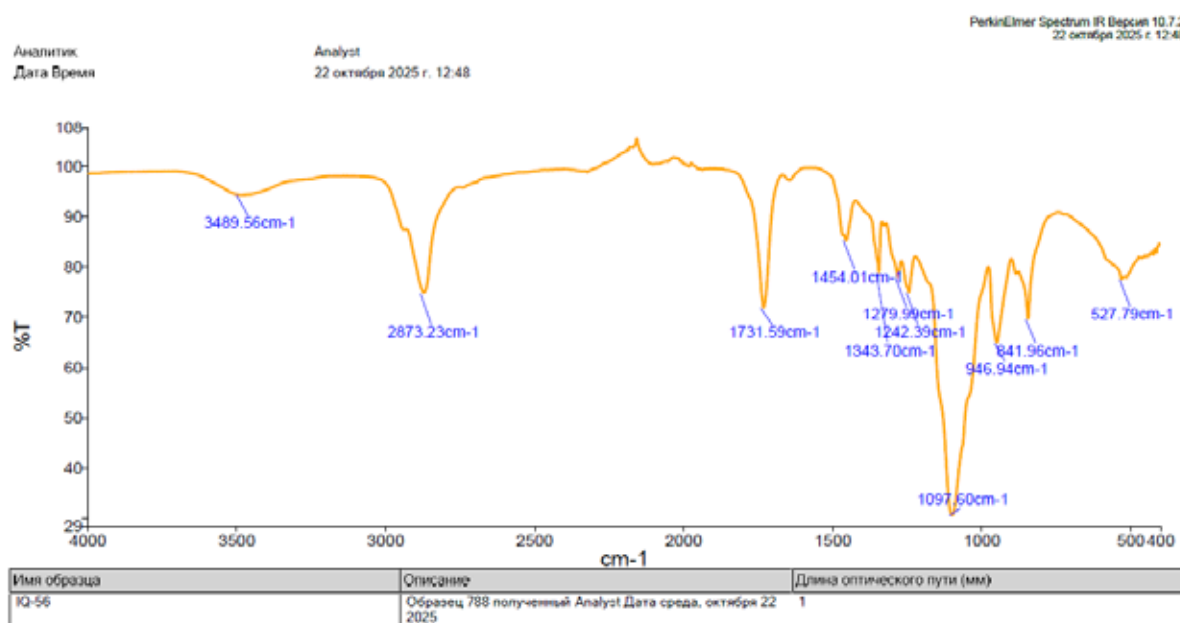
No.	Mass ratios	Time	Reaction Yield, %	No.	Mass ratios	Time	Reaction Yield, %
1.	3:1	2	26.2	11	3:1	4	49.4
2.	4:1		37.3	12	4:1		65.8

No.	Mass ratios	Time	Reaction Yield, %	No.	Mass ratios	Time	Reaction Yield, %
3.	6:2		47.4	13	6:2		72.5
4.	7:1		54.3	14	7:1		78.7
5.	9:1		56.5	15	9:1		79.8
6.	3:1		35.5	16	3:1		49.5
7.	4:1		60.5	17	4:1		65.9
8.	6:2	3	70.5	18	6:2	5	72.6
9.	7:1		72.4	19	7:1		78.7
10.	9:1		75.5	20	9:1		90

Table 2 presents the influence of various factors on the superplasticizer yield based on the presented results: time and molar ratios of the initial substances. As can be seen from Figure 2, compared to others, the allylicidial ether is obtained at the highest 8:1 ratio of acrylic acid and HPEG, but the resulting product contains derivatives of di-naphth-

ylsulfones. Therefore, when studying the plasticizing effect of mono-, di-, and other polyhydric alcohols, their derivatives are of particular importance. From the obtained results, it can be seen that the yield of superplasticizer was highest at an allylycidyl ether, acrylic acid, and HPEG ratio of 8:1 and a process duration of 5 hours.

Figure 3. IR spectrum of the HPEG-based polycarboxylate superplasticizer



IR spectrum: 3489 cm^{-1} (O-H); 1731 cm^{-1} , (C=O); 1097 cm^{-1} (C-O-C); 1343 cm^{-1} (C-O) It can be seen that the resulting low-flow polycarboxylate superplasticizer contains various functional groups such as hydroxyl groups, carbonyl groups, ether groups, and alkyl groups in its molecule, which are essentially consistent with the developed structure. The structure of the obtained product

largely corresponds to the molecular structure of the initially developed polycarboxylate superplasticizer.

The hydration of the binding substances showed the presence of a small amount of water, i.e., as a result of drying the samples under natural conditions, a large number of small crystals are formed. Mechanical mixing is carried out using an aqueous solution

of polycarboxylate, which improves the wetting properties of cement stone to provide

the binder with the necessary amount of water.

Table 3. Chemical composition of HPEG-based polycarboxylate superplasticizer based on elemental analysis

Element	Mass%	Energiya spektrometri	Atom%
C	55.17±0.04	K	62.36±0.05
O	43.96±0.09	K	37.30±0.08
S	0.21±0.00	K	0.09±0.00
Total	100.00		100.00

Table 4. Technical requirements for HPEG-based polycarboxylate superplasticizer

Name of indicators	Units of measurement	In solution form
Appearance	%	Light yellow liquid
Density 20 °C,	g/sm ³	1.05
Dry residue,	%	30%
Hydrogen ion activity indicator (Ph), aqueous solution	%	7
Dry matter does not exceed the mass fraction of chlorine ions	%	0.10
Water solubility 20 °C,	%	Completely dissolves
Reduce water consumption	%	30–35

This method is based on measuring the spread diameter of cement mortar under the influence of gravity. The effects of superplasticizers on cement mixtures were studied, and a superplasticizer based on HPEG was selected. The additives were introduced as aqueous solutions. The concentration of additives was calculated as a percentage ratio of the dry matter mass to the mass of the dispersed phase. Measurements were taken after mixing the solution for two minutes. The setting times of the cement mortar were determined in accordance with GOST 310.3–76. The effect of superplasticizers on concrete strength was investigated using 2 × 2 × 2 sm samples. The specimens were cured under room conditions for 28 days,

after which they underwent compression testing.

The effect of superplasticizer on concrete strength was determined using a press (GOST 8905–73) in accordance with GOST 10180–90. The influence of additives on the flowability of concrete mixtures was assessed using a standard slump cone as per GOST 10181.1–2000. The cone is filled with the concrete mixture being tested and compacted. After lifting the cone, the spread of the concrete mixture is measured with a measuring device to an accuracy of 0.5 sm. Concrete strength was determined on 10 × 10 × 10 cm samples at 28 days of age. Six samples are prepared for each test. The concrete mixtures are compacted on a type 435A vibrating table.

Table 5. Results of determining the flowability and average density of cement mortar with the addition of a polycarboxylate-based superplasticizer

No.	Cement quantity, gr	Cement mass addition, %	Water/cement ratio	Flowability, sm	Average density, gr/sm ³
1.	100	–	0.43	6	1.950
2.	100	0.2	0.42	8.5	1.950

No.	Cement quantity, gr	Cement mass addition,%	Water/cement ratio	Flowability, sm	Average density, gr/sm ³
3.	100	0.4	0.42	10	1.900
4.	100	0.6	0.42	11	1.855
5.	100	0.8	0.42	12	1.850
6.	100	1	0.42	13	1.850

Typically, superplasticizers are added to concrete mixtures in quantities ranging from 0.2% to 0.8% of the cement mass. Adding a plasticizer above 1% is considered impractical. In the experiments, PS400D20 grade cement was used. The flowability of the cement mortar was studied according to GOST 26798.1–96. When studying the physical and mechanical properties of concrete, the water-cement ratio typically

ranges from 0.2–0.8%, but at this amount of water, the change in cement spreadability is insignificant. Therefore, to objectively study the rheological properties of cement systems, we selected a water-cement ratio of 0.42.

As can be seen from Table 5, relatively high strength can be achieved by adding a polycarboxylate superplasticizer in an amount of 1%.

Table 6. *The effect of polycarboxylate superplasticizer on the water-cement ratio and strength of the cement system*

No.	Cement quantity, gr	Cement mass addition,%	Water/cement ratio	Strength, MPa
1.	100	–	0.24	18
2.	100	0.2	0.23	24
3.	100	0.4	0.23	29
4.	100	0.6	0.25	33
5.	100	0.8	0.22	36
6.	100	1	0.22	35

Further increase in the amount of polycarboxylate superplasticizer additive reduces the strength of the cement stone. The same effect is observed when adding a polycarboxylate superplasticizer. When this additive is introduced at 0.8%, the strength of the cement stone reaches 36 MPa. With the addition of polycarboxylate superplasticizer, the strength of the cement stone increases as the amount of additive increases. As evident from the above, the obtained polycarboxylate superplasticizer demonstrates good plasticizing properties.

Conclusion

The optimal ratios of HPEG and AK for the synthesis of a new experimental polycar-

boxylate superplasticizer have been determined. The optimal dose of initiators during the synthesis process was 1.0% of the total mass of monomers and was continuously added dropwise. The concentration of the synthesized superplasticizer was 35%, the reaction temperature ranged from 70 °C to 80 °C, and the reaction time was four hours. A highly effective polycarboxylate superplasticizer with a molecular structure containing carboxyl groups has been synthesized. The newly synthesized superplasticizer has a positive effect on cement compositions, reducing the water demand of the mixture by 15–25% due to high plasticization, while increasing strength properties by 15–25% and density.

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