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SYNTHESIS AND DESCRIPTION OF A POLYCARBOXYLATE SUPERPLASTICIZER BASED ON POLYOXYETHYLENE ISOPENTENYL ETHER

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Abstract

This research was conducted to evaluate a new type of polycarboxylate superplasticizer. For the synthesis of a polyarboxylate superplasticizer with high water-lowering capacity, polyoxyethylene ether (TPEG) and acrylic acid (AC) were used as monomers, ammonium persulfate as an initiator, and thioglycolic acid (TGC) as a chain transfer agent. The work discussed the influence of additives on the flowability and shrinkage of the concrete cone. The structure of the water-reducing additive based on carbonic acids was described using infrared spectroscopy. The study also assessed the influence of various series of polyhydroxyethylene ether (TPEG)), the amount of acrylic acid (AA), and ammonium persulfate.

Keywords: *polycarboxylate superplasticizer, carboxyl density, cement paste flowability*

Polycarboxylate superplasticizers are widely used in modern concrete production to increase the flowability of fresh mixtures, as well as to reduce water demand for achieving the specified flowability. It has been demonstrated that they can reduce heat generation during hydration and improve fluidity, which leads to increased strength and durability of hardened concrete products (Witt J., Plank J.). Polycarboxylate superplasticizers are the most promising category of polymer dispersants. From the perspective of chemical structure, these polymers offer numerous advantages: good flowability and spreadability of polycarboxylate superplasticizers in small quantities, wide compatibility with cement, high water reduction efficiency, greater variability of molecular structure, diversity of synthesis technologies, and significant potential for improvement have been studied (Sakai E., Ishida A., Ohta A.; Magarotto K., Torresan I., Zeminian N., 2003; Yamada K., Ogawa S., Hanebara S., 2001). The history of studying and applying polycarboxylate superplasticizers spans only a decade. Research efforts are focused on selecting reactive monomers, determining synthesis temperatures, establishing initiator types, optimizing technological conditions, as well as reducing production costs and controlling product prices (Li S., Yu Q., Wei J., et al., 2011; Lange A. A., Plank J., 2012). Our understanding and knowledge about the structure of polycarbonates, their dispersion properties, rheological characteristics, as well as the relationship between hydration theory and practical applications in construction remain limited (Hirata T., Lange A., Plank J., 2012; Ismailov F. S., 2025). In this study, a number of polycarboxylate superplasticizers were synthesized. Isoamyl alcohol (IPEG) of polyoxyethylene ether and acrylic acid in various quantities were used as monomers, and ammonium persulfate as an initiator and chain carrier. The influence of monomer type, carboxyl group density, initiator quantity, chain-transmitting agent quantity, and pH on the dispersion properties of the superplasticizer was studied by measuring the flowability of the cement paste and the workability of the concrete mix.

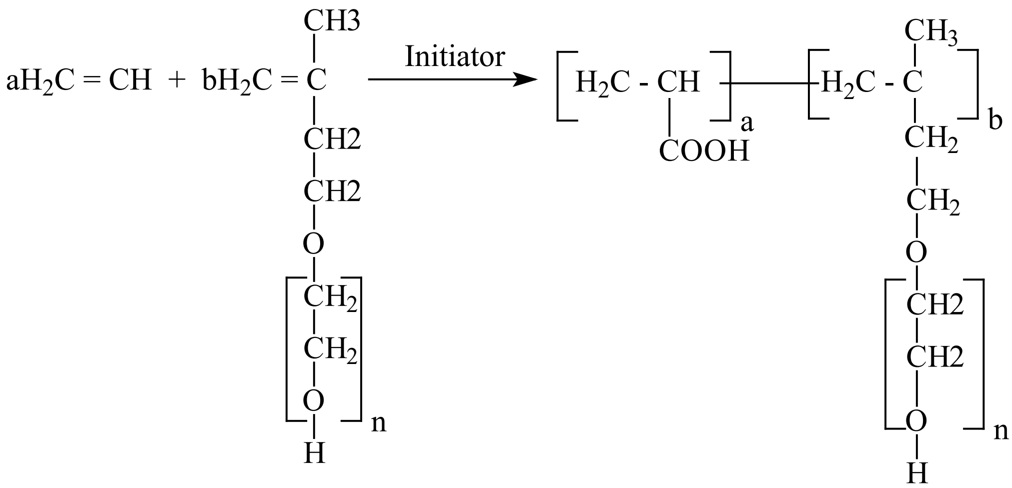
Methodology

When synthesizing a polycarboxylate superplasticizer based on TPEG, 180 liters of distilled water are added to a stainless steel heater reactor. For the first reactor, a solution is prepared by mixing 120 kg of TPEG, 14 kg of acrylic acid, 6 grams of HEA, and mercaptopropionic acid in 180 liters of water, stirring at a rate of 50 rpm until a homogeneous mass is obtained. 180 liters of distilled water is poured into the third reactor, heated to 50 °C, and for 3–4 hours, the contents are pumped from the first reactor to the second using a special chemically resistant pump for 2 hours. The second reactor is constantly heated, stirring at a rate of 100 rpm. The temperature is constantly monitored. Time and temperature play a crucial role in the course of the reaction. After loading the first part of the reagents into the second reactor, 5 liters of hydrogen peroxide solution is introduced through another opening for 40 minutes. After supplying the solution, hydrogen peroxide is maintained at a temperature of 60 °C for 45 minutes. During the synthesis process, a copolymerization reaction of monomers occurs. The amount of dry residue and pH are determined in the resulting product.

Results 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 the production of concrete and reinforced concrete, plasticizers occupy the first place. The literature analyzes published works on the influence of plasticizing additives on the workability and strength of concrete mixtures, and examines the mechanism of action of plasticizing and superplasticizing additives in cement systems.

Figure 1. *The process of obtaining a semi-carboxylate superplasticizer based on TPEG was carried out according to the following reaction scheme*



The dependence of the TPEG-based polycarboxylate superplasticizer yield on time is presented in Figure 2. As shown in Figure 2, the highest efficiency compared to other ratios is achieved with an HEA, acrylic acid, and TPEG ratio of 3.88:1:1. The synthesis process takes 4 hours. In experiments lasting longer than 4 hours, the superplasticizer yield decreases. This can be explained by the fact that the raw materials exist in various aggregate states for 4 hours or more. The presence of interacting substances in two different aggregate states reduces their interaction.

Table 1. *Amount of substances for the synthesis of the superplasticizer half-carboxylate based on TPEG*

No.	Substances used in synthesis	Quantity (kg)
	1 part solution	
1.	TPEG	120
2.	water	360
3.	acrylic acid	14
4.	HEA	6
5.	MPA-3. 95%	0.5
	2 part solution	
6.	water	5
7.	APS, 99%	0.5

Figure 2. *Time dependence of the yield of a polycarboxylate superplasticizer based on TPEG*

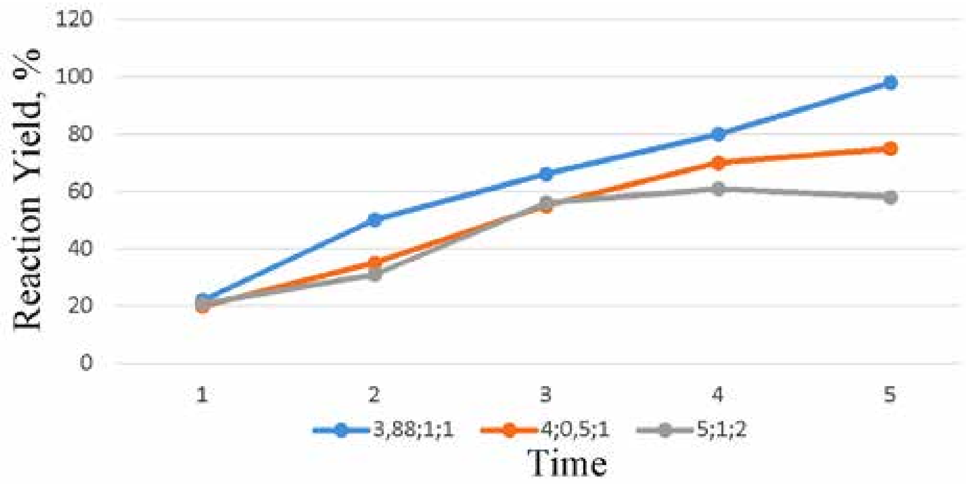


Figure 3. *Dependence of the yield of TPEG-based polycarboxylate superplasticizer on the molar ratio*

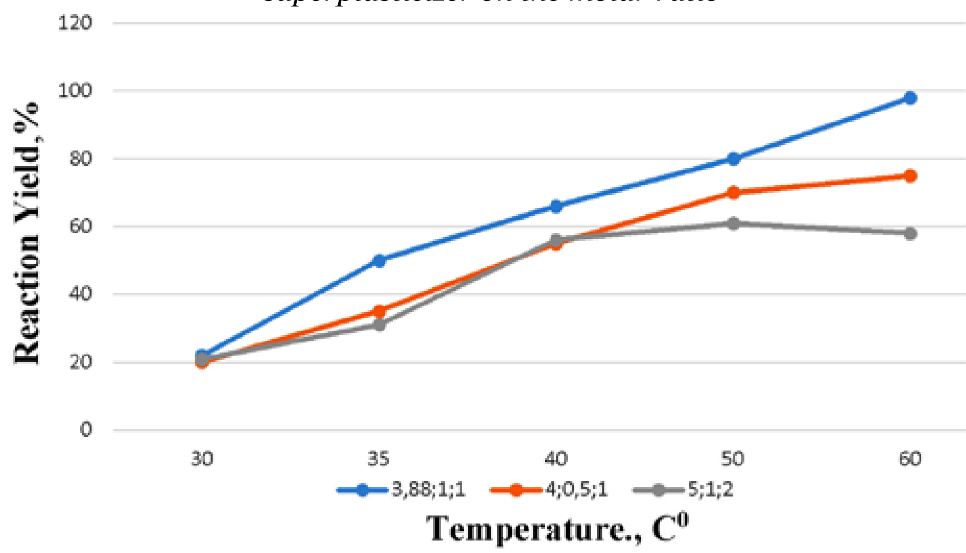


Figure 3. Dependence of the yield of a polycarboxylate superplasticizer obtained on the basis of TPEG on the molar ratios of acrylic acid, HEA, and TPEG As can be seen from Figure 3, the optimal ratio of components in the production of a polycarboxylate superplasticizer is 3.88:1:1, under these conditions, the profitability of the superplasticizer is 90%. The highest dry residue is obtained at a ratio of the initial products of 4:0.5:1 and 5:1:2, but the plasticizing effect of the obtained superplasticizers is low. Based on this, the optimal temperature of 60 ˚C was chosen for obtaining superplasticizer. Reaction time also plays an important role in the production of superplasticizers. To choose the optimal reaction yield temperature, all three initial product ratios were selected. The following figure shows the dependence of superplasticizer yield on reaction time at a temperature of 60 ˚C. As can be seen from the figure, as a result of carrying out the reaction under optimal conditions for 5 hours, a superplasticizer yield of 90% 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), which leads to a decrease in the plasticizing effect of superplasticizing additives.

Table 2.

No.	Mole ratios	Time	Reaction Yield, %	No.	Mole ratios	Time	Reaction Yield, %
1.	5:1,5:1,5		26.2	11	5:1,5:1,5		49.4
2.	5:2:2,5		37.3	12	5:2:2,5		65.8
3.	4:0,5:1	2	47.4	13	4:0,5:1	4	72.5
4.	5:1:2		54.3	14	5:1:2		78.7
5.	3,88:1:1		56.5	15	3,88:1:1		**79.8**
6.	5:1,5:1,5		35.5	16	5:1,5:1,5		49.5
7.	5:2:2,5		60.5	17	5:2:2,5		65.9
8.	4:0,5:1	3	70.5	18	4:0,5:1	**5**	72.6
9.	5:1:2		72.4	19	5:1:2		78.7
10.	3,88:1:1		75.5	20	**3,88:1:1**		90

Figure 4. *IR spectrum of a polycarboxylate superplasticizer based on TPEG*

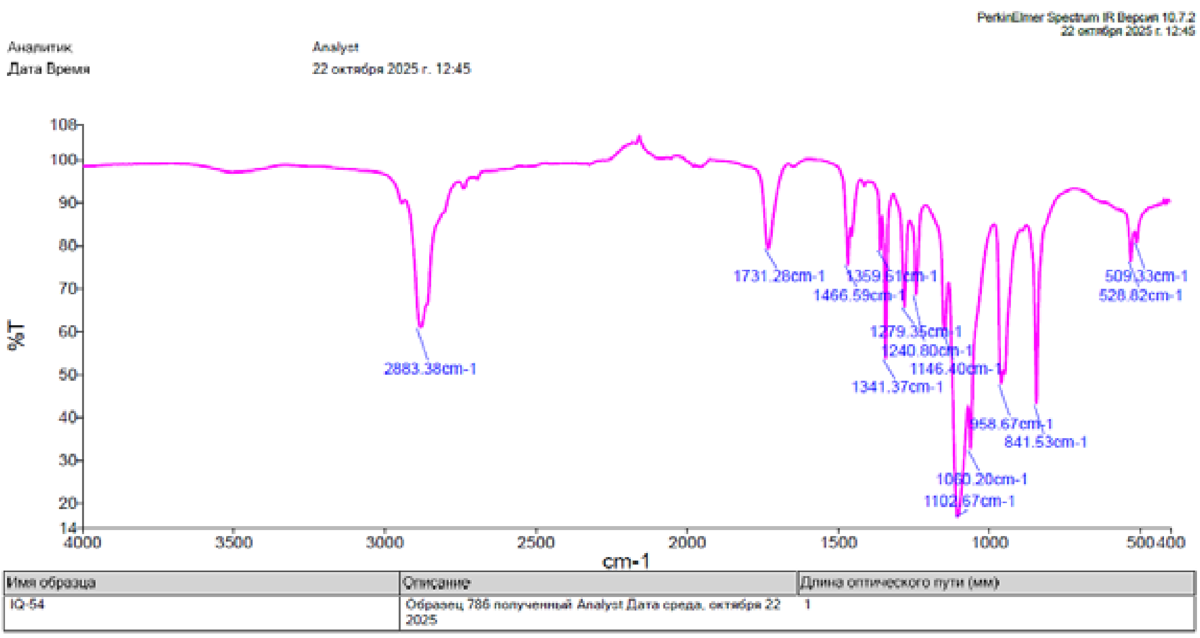


Table 2. presents the influence of various factors on the superplasticizer yield based on the presented results: time and molar ratios of the initial substances. As can be seen from Figure 2, compared to others, acrylic acid and NEA are obtained at the highest TPEG ratio of 3.88:1:1, however, the resulting product contains derivatives of di-naphthylsulfones. 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 at a molar ratio of acrylic acid and HEA, TPEG 3.88:1:1, and a process duration of 5 hours, the yield of the polycarboxylate superplasticizer was the highest.

Figure 4 shows that the broad peaks from 2500 sm^{-1} to 3000 sm^{-1} are attributed to the stretching vibrations of the O-H group. The peaks at 1731 sm^{-1} and 1466 sm^{-1} exhibit characteristic absorption zones of the polyethylene oxide group (CH2CH2O). A typical absorption peak of stretching vibrations of the C-O-C structure was observed at 1060 $sm^{-1.}$ The characteristic absorption peak of the COO-group appeared at 1466 sm^{-1}. The absence of resonance for the carbonyl bond in AA and TPEG accounts for the observed shift towards higher wavenumbers. Thus, the vibrational signals observed in the IR spectra confirm the successful synthesis of the polycarboxylate superplasticizer.

Conclusion

The results of the conducted research showed that concrete with the addition of superplasticizer is characterized by high strength and durability under conditions of reduced water consumption. Today, increasing attention is being paid to concrete and concrete products with the addition of superplasticizers. Using macromonomers and monomers, it is possible to synthesize a high-performance polycarboxylate superplasticizer and provide a new superplasticizer. By analyzing various factors influencing the effectiveness of polycarboxylates, optimal conditions for the synthesis process of a new type of polycarboxylate superplasticizer with high dispersity were determined. Factors influencing the reaction include acid ratio, initiator concentration, pH level, and additives. There is an opportunity to introduce this method into industrial production.

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