

UDK 666.972.3

ABBASOVA S.I., IBRAHIMOV K.A.

Azerbaijan University of Architecture and Construction
saida.abbasova@azmiu.edu.az

EFFECT OF POLYCARBOXYLATE-BASED SUPERPLASTICIZERS ON THE PROPERTIES OF CEMENT SYSTEMS

1. Introduction. It is known that the water-cement ratio significantly affects the physical-mechanical and operational properties of concrete [1]. Various plasticizing and water-reducing admixtures are used to reduce the water-cement ratio during concrete mixture preparation [2, 3].

Among the plasticizing admixtures currently used in cement systems, the most effective are superplasticizers, which can reduce the water demand of the concrete mixture by up to 40%. Unlike superplasticizers based on naphthalene and melamine sulfonates, polycarboxylate-based superplasticizers have a significant water-reducing and plasticizing ability, while ensuring the production of highly flowable and self-compacting concrete mixtures that retain high rheological properties [4-7].

The stronger water-reducing and plasticizing effect of polycarboxylate-based superplasticizers is explained by their structure. Unlike polynaphthalenesulfonates and polymelaminesulfonates, which have a linear polymer chain, polycarboxylates are characterized by molecules with a spatial structure having side chains, which leads to more efficient dispersion of cement flocs due to the steric effect.

2. Materials and research methods

2.1. Materials. In the experimental studies, during the preparation of cement stone and self-compacting concretes, CEM I 52.5N Portland cement produced at the HOLCIM cement plant according to the AZS EN 197-1 standard was used as a binder.

The specific surface area of the Portland cement is 400 m²/kg, the normal consistency of the cement paste is 28.5%, the initial setting time is 120 min, the final setting time is 150 min, and the compressive strength of spe-

cimens hardened for 28 days is 55.9 MPa.

During the research, Bahramtepe river sand was used as a fine aggregate for the preparation of the concrete mixture. The properties of the sand were determined according to the method given in GOST 8736-2014. The average density of the sand is 1.51 g/cm³, and the true density is 2.65 g/cm³.

During the research, crushed stone of 5-20 mm fraction obtained from crushing river stones at the Guba quarry was used as a coarse aggregate. The properties of the crushed stone were determined according to the method given in GOST 8269.0-97. The average density of the crushed stone is 1.56 g/cm³, and the true density is 3.0 g/cm³.

In this work, microsilica was used as an active mineral admixture, and as plasticizing admixtures, a superplasticizer based on sulfonated naphthalene-formaldehyde polycondensate from NANOKIM company and a polycarboxylate-based superplasticizer were used. Polycarboxylate-based admixtures are aqueous solutions modified with polymers of various molecular structures and have different water-reducing, flowability retention, and early strength characteristics.

Naphthalenesulfonate-based Nanocon SP 900 can reduce the water demand of the concrete mixture by up to 50-60 liters compared to conventional concrete.

Polycarboxylate-based Nanocon HP 1010 can reduce the water demand of the concrete mixture by up to 80-100 liters compared to conventional concrete; the concrete mixture has greater plasticity, acquires self-compacting properties, and can have a denser structure and higher strength.

2.2. Research methods. The normal consistency and setting time of the cement paste were determined using a Vicat apparatus ac-

according to the method given in AZS 196.3.

The compressive strength of the cement-sand mortar was determined on 40×40×160 mm prism specimens according to AZS 196.1. The compressive strength of concrete specimens was determined on 100×100×100 mm cube specimens according to GOST 10180-2012.

The selection of compositions of self-compacting concrete mixtures was carried out according to GOST 27006-2019, taking into account the requirements for the ratio of components.

To evaluate the workability and flowability of self-compacting concrete mixtures, their spread from a standard cone was determined according to GOST R 58002-2017/EN 12350-8:2010. The viscosity of self-compacting concrete mixtures was determined by the time of spreading of the concrete mixture up to a diameter of 500 mm (T_{500}). Cube specimens of 100×100×100 mm were prepared from the concrete mixture. After hardening under normal conditions for 2 and 28 days, their compressive strength was determined according to GOST 10180-2012.

Studies on the flowability retention of self-compacting concrete mixtures were carried out at room temperature of 23±2 °C according to GOST 10181-2014 and GOST 30459-2008. The flowability was evaluated by the spread diameter of the concrete mixture from a standard cone. The first determination of flowability was carried out immediately after the completion of mixing the concrete mixture, and subsequent ones every 60 minutes from the moment of their preparation. Before each test, the concrete mixture was re-mixed.

3. Experiments and results. In the initial stage of the research, the effect of polycarboxylate-based superplasticizers on the plasticity of cement was studied. The tests were carried out in comparison with a non-admixed cement paste with a water/cement ratio of $W/C=0.29$. The amount of superplasticizer in the mixture varied between 0.4-0.7% by weight of cement. Superplasticizers were added to the cement mixture together with water. The spread of the cement paste

was monitored at a constant water-cement ratio. The spread of the cement suspension was determined 5 and 60 minutes after the preparation of the mixture.

The results regarding the effect of superplasticizers on the spread of the cement paste are shown in Figures 1 and 2.

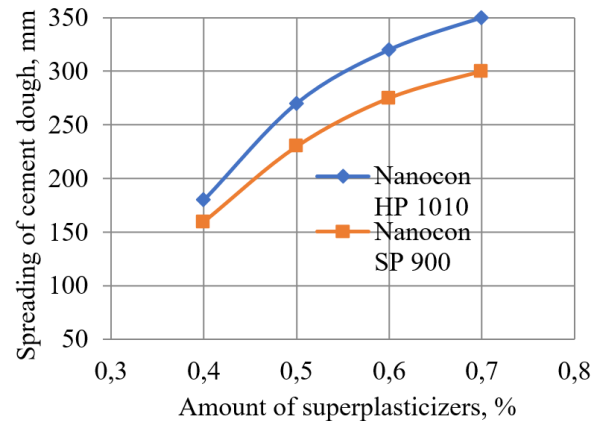


Figure 1. Effect of superplasticizers on the spreading of cement paste: (immediately after preparation of the mixture)

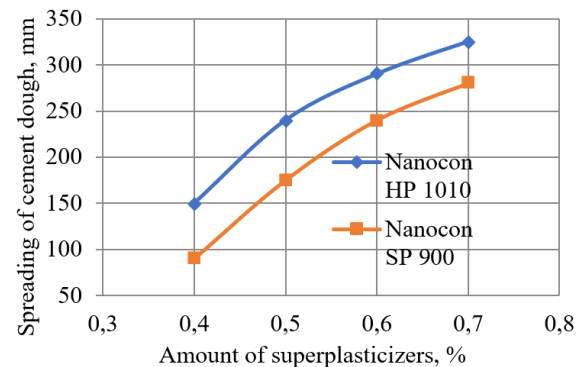


Figure 2. Effect of superplasticizers on the spreading of cement paste: (60 min. after preparation of the mixture)

The studies revealed that in cement mixtures without microsilica, the plasticizing ability of superplasticizers with different molecular structures differs.

It can be seen from Figure 1 that when microsilica is not used, all plasticizers have a significant effect on the spread of the cement paste. In the initial stage, when the amount of superplasticizer Nanocin SP 900 was 0.4%, the spread of the cement paste was 160 mm,

while at 0.7% it increased to 300 mm. However, when using this superplasticizer, it was observed that the cement paste lost its flowability 60 minutes after preparation. Based on the research results, it was determined that the best effect on the cement paste is provided by Nanocon HP 1010, ensuring a flowability of 350 mm (Figure 2). The studies show that the best retention of flowability is possible when using the Nanocon HP 1010 admixture. Namely, after 60 minutes, when using Nanocin SP 900, the flowability of the cement paste was 90 mm, whereas when using Nanocon HP 1010, it was 150 mm.

In the next stage of the research, the effect of complex organic-mineral admixtures on the properties of self-compacting concrete mixtures and concretes was studied. The experiments were carried out with compositions of self-compacting concrete mixtures prepared with more efficient ratios of coarse and fine aggregates. The consumption of microsilica was 10% by weight of cement. The consumption of superplasticizer was taken as 1% of

the total binder mass. The amount of mixing water was selected according to GOST 59714-2021, provided that self-compacting concrete mixtures of class YS2 in terms of ease of placement are obtained, which do not require vibratory compaction during molding [4].

To compare the properties of self-compacting concrete mixtures with a complex admixture based on microsilica and superplasticizer, a concrete mixture with BM 10-01 organic-mineral modifier was prepared. The BM organic-mineral modifier is a powdery product with a bulk density of 800 kg/m³, consisting of particles ranging in size from 2 μm to 400 μm. The mineral part of the modifier (90%) consists of microsilica, and the organic part (10%) consists of a superplasticizer based on sulfonated naphthalene-formaldehyde polycondensates. At the same time, control compositions were prepared from a concrete mixture without modifying admixture, with a flowability class of P4.

The compositions and properties of the concrete mixtures are given in table 1.

Table 1

Compositions and properties of concrete mixtures with modifying admixtures

№	Composition	Component amounts, kg/m ³							W/C	Slump, mm	Spread, mm	Viscosity T ₅₀₀ , sec
		Cem	BM	PKE	MS	Sand	Crushed stone	W				
1	Non-admixed	550	–	–	–	621	1012	248	0.45	170	–	–
2	BM	500	55.5	–	–	778	951	200	0.36	180	–	–
3	BM	500	111	–	–	729	891	220	0.36	–	710	5.8
4	PCE	550	–	5.5	–	828	1012	170	0.31	–	730	8.0
5	CA	500	–	5.5	50	819	1001	170	0.31	–	690	8.2

BM – organic-mineral modifier (based on microsilica and naphthalene sulfonate), PCE – polycarboxylate ether superplasticizer, MS – microsilica, CA – complex admixture (microsilica + PCE)

Based on the results obtained from the determination of the flowability of the concrete mixture (Table 1), it was found that obtaining self-compacting mixtures is only possible with the use of plasticizing admixtures. In the non-admixed composition (Composition 1), even with a high amount of cement paste, the slump of the concrete mixture was 170 mm (flowability class P4). The use of BM organic-mineral modifier based on microsilica and sulfonated naphthalene-formaldehyde (Composition 3) allows obtaining self-compacting con-

crete mixtures only when a very high amount of the admixture (22%) is used. The use of a small amount of BM admixture does not allow obtaining self-compacting concrete mixtures (Composition 2). Moreover, the application of the modifier based on sulfonated naphthalene-formaldehyde superplasticizer does not significantly reduce the water content in the concrete mixture.

The use of polycarboxylate superplasticizer (Composition 4) allows significantly reducing the water content in the concrete mix-

ture, and due to the strong plasticizing and water-reducing effect of the admixture, it is possible to obtain a self-compacting mixture even at a water-cement ratio of $w/c=0.31$.

The use of a complex organic-mineral admixture (Composition 5) allows, due to its high water-retaining capacity, to reduce water separation by microsilica, to increase the homogeneity and stability of the self-compacting concrete mixture without significantly reducing its flowability.

An important technological characteristic of self-compacting concrete mixtures is the retention of flowability and the prevention of segregation of the concrete mixture over time. The study of the flowability retention of self-compacting concrete mixtures was carried out at room temperature according to GOST 10181-2014 and GOST 30459-2008. Flowability is evaluated by the spread diameter of the concrete mixture from a standard cone. The first determination of flowability is carried out immediately after the completion of mixing of the concrete mixture, and subsequent ones every 60 minutes from the moment of their preparation. Each determination of the mixture's flowability is carried out on a new sample of the concrete mixture.

Figure 3 shows the results regarding the flowability retention time of self-compacting concrete mixtures prepared with modifying admixtures.

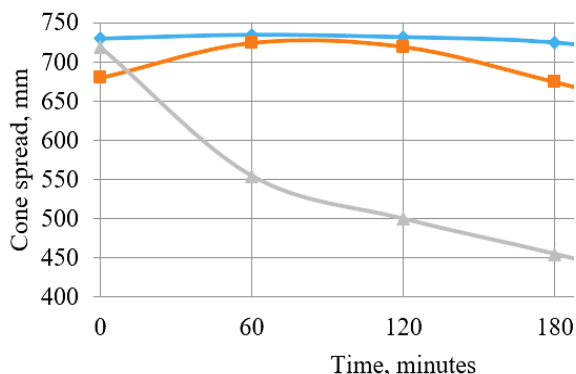


Figure 3. Flow retention of self-compacting concrete mixtures with modifiers.
1- BM, 2- PKE, 3- KƏ

It was determined that the use of BM organic-mineral modifier based on MS and SNF leads to a rapid loss of flowability of the

concrete mixture, even at a high amount of the admixture. The flowability retention of the self-compacting concrete mixture with BM admixture is less than 30 minutes when the ease of placement class is YS2 (Figure 3-1), which does not allow the concrete mixture to be transported over long distances during the construction of monolithic structures.

The use of polycarboxylate superplasticizer ensures long-term retention of flowability of the concrete mixture even at low values of the W/C ratio (Figure 3-2).

In this case, a slight increase in flowability of the self-compacting concrete mixture with PCE is observed 60 minutes after re-mixing. This may be due to the characteristics of the molecular structure of the polycarboxylate superplasticizer used. Researchers note that PCE adsorbs slowly onto cement particles and has a delayed plasticizing effect. It is also noted that self-compacting concrete mixtures containing polycarboxylate superplasticizer are prone to segregation and water separation over time if stabilizing and water-retaining admixtures are not used.

The use of a complex organic-mineral admixture (Figure 3-3) allows reducing water separation, increasing the homogeneity and stability of the self-compacting concrete mixture without significantly reducing its flowability. At the same time, it was determined that the application of the complex admixture leads to acceleration of cement hydration, shortening of the setting time of the cement paste, and a reduction in flowability retention of the concrete mixture compared to PCE. In this case, the obtained self-compacting concrete mixture with complex admixture has high flowability, with a cone spread of more than 700 mm, and its flowability retention exceeds 3 hours when the ease of placement class is YS2. This allows such concrete mixtures to be used in the construction of monolithic structures.

In the next stage of the research, the effect of complex organic-mineral admixtures on the physico-mechanical properties of self-compacting concretes was studied.

The results of the conducted experiments are given in table 2.

Table 2

Properties of concretes with modifying admixtures

№	Composition	Compressive strength, MPa			Concrete class	Concrete density, kg/m ³
		1 day	2 days	28 days		
1	Non-admixed	12.0	25.1	48.0	B35	2353
2	BM	15.4	36.5	68.4	B50	2379
3	PCE	26.5	52.5	74.9	B55	2498
4	CA	31.6	56.5	92.5	B70	2530

Figure 4 shows the hardening kinetics of self-compacting concretes with modifying admixtures.

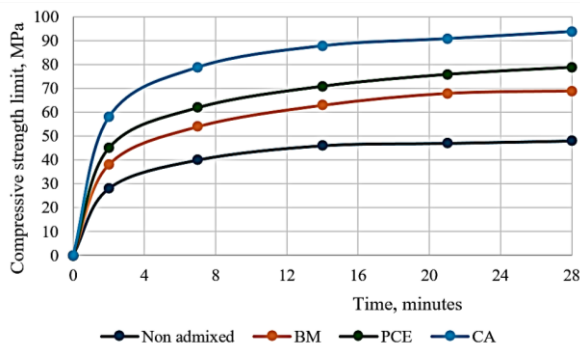


Figure 4. Curing kinetics of self-compacting concrete with modifying additives

According to the strength results obtained, the strength of the control non-admixed concrete composition after 28 days of hardening was 48.0 MPa, which corresponds to class B35.

The use of BM organic-mineral modifier allows increasing the 28-day strength of concrete by 43% compared to non-admixed control specimens and obtaining self-compacting concrete of class B50.

The use of polycarboxylate superplasticizer, due to a significant reduction in the water demand of the concrete mixture, allows increasing the strength of self-compacting concrete compared to BM-admixed self-compacting concrete by 72% at 1 day of hardening, 44% at 2 days, and 10% at 28 days. In this case, although the self-compacting concrete mixture with polycarboxylate superplasticizer retains its flowability for a long time, the hardening kinetics

of the concrete is characterized by rapid early strength gain. The application of superplasticizer based on polycarboxylate ethers allows obtaining self-compacting concrete with a cement content of 550 kg/m³ having strength classes of B20 at 1 day, B40 at 2 days, and B55 at 28 days of hardening.

The use of complex admixtures based on microsilica and polycarboxylate superplasticizer allows increasing the strength of self-compacting concrete compared to PCE-admixed self-compacting concrete by 19% at 1 day of hardening, 8% at 2 days, and 23% at 28 days. The increase in strength is associated with the high pozzolanic activity of microsilica, the low amount of portlandite in the structure of the cement stone, and the increase in the amount of amorphous low-basic calcium hydrosilicates. The use of the complex admixture also leads to a densification of the cement stone structure.

Compared to BM-modified self-compacting concrete, the complex admixture leads to an increase in concrete strength of 105% at 1 day of hardening, 55% at 2 days, and 35% at 28 days, which allows obtaining self-compacting concrete of class B70 with a cement content of 500 kg/m³.

CONCLUSION

1. Among the superplasticizers studied, Nanocon HP 1010 (polycarboxylate-based) increased the spread of cement paste to 350 mm and retained flowability at 150 mm even after 60 minutes. This is significantly higher

compared to Nanocin SP 900 (90 mm after 60 minutes).

2. The combined application of microsilica (10% by weight of cement) and polycarboxylate superplasticizer (1%) exhibits a synergistic effect. This complex admixture reduces the water separation capacity of the self-compacting concrete mixture, increases its homogeneity and stability, while maintaining high flowability (spread 690–730 mm).
3. The self-compacting concrete mixture with complex admixture retains its flowability for more than 3 hours when the ease of placement class is YS2. This allows the concrete mixture to be transported over long distances and processed for a long time during the construction of monolithic structures.
4. The 28-day compressive strength of concrete specimens obtained with the complex organic-mineral admixture reached 92.5 MPa (class B70). This is 23% higher than the strength obtained with polycarboxylate superplasticizer alone, and 35% higher than concrete with BM (naphthalenesulfonate-based) modifier. The early strength (1 day) was 31.6 MPa, which enables rapid formwork removal.
5. For the production of self-compacting concrete with high flowability, long workability retention, and high strength, it is advisable to use a complex admixture consisting of microsilica (MK-85) and Nanocon HP 1010 superplasticizer. This approach densifies the concrete structure, increases the amount of low-basic calcium hydrosilicates as a result of the pozzolanic reaction, and enhances the overall durability of the concrete.

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UDK 666.972.3

Abbasova S.I., İbrahimov K.A.

Azərbaycan Memarlıq İnşaat Universiteti
saida.abbasova@azmiu.edu.az

**Polikarboksilat əsaslı
superplastikləşdiricilərin sement
sistemlərinin xüsusiyyətlərinə təsiri**

XÜLASƏ

Polikarboksilat əsaslı superplastikləşdiricilərin sement sistemlərinin reoloji və fiziki-mexaniki xassələrinə təsiri tədqiq edilmişdir. Su/sement nisbəti 0,29 olan sement xəmirinə müxtəlif molekulyar quruluşlu superplastikləşdiricilər (Nanocin SP 900 və Nanocon HP 1010) 0,4–0,7% miqdarda əlavə edilmişdir. Təcrübələr göstərmişdir ki, Nanocon HP 1010 ən yaxşı plastikləşdirici effektdə malikdir – ilkin yayılma 350 mm, 60 dəqiqədən sonra isə axıcılıq 150 mm olmuşdur. Mikrosilika ilə birlikdə istifadə edilən polikarboksilatlı superplastikləşdirici özüyərləşən beton qarışığının axıcılığını (yayıma 690–730 mm, özlülük 8–9 saniyə) və dayanıqlığını yaxşılaşdırır, eyni zamanda suayırmanı azaldır. Betonun 28 günlük sıxılmada möhkəmliyi 92,5 MPa (B70 sinfi) təşkil etmiş, axıcılığın saxlanma müddəti 3 saatdan çox olmuşdur. Beləliklə, mikrosilika və polikarboksilat əsaslı superplastikləşdiricidən ibarət kompleks üzvi-mineral əlavə yüksək axıcılığa, seqreqasiyaya davamlılığa və uzun iş qabiliyyətinin saxlanmasına malik özüyərləşən betonların istehsalı üçün ən effektiv additividir.

Açar sözlər: polikarboksilat, superplastikləşdirici, özüyərləşən beton, mikrosilika, reoloji xassələr, möhkəmlik

UDK 666.972.3

Redaksiyaya daxil olma/Received 21.04.2026

Çapa qəbul olunma/Accepted for publication 25.05.2026

Abbasova S.I., İbrahimov K.A.

*Azerbaijan University of Architecture and
Construction*
saida.abbasova@azmiu.edu.az

**Effect of polycarboxylate-based
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ABSTRACT

The effect of polycarboxylate-based superplasticizers on the rheological and physico-mechanical properties of cement systems has been investigated. Superplasticizers with different molecular structures (Nanocin SP 900 and Nanocon HP 1010) were added in an amount of 0.4–0.7% to a cement paste with a water/cement ratio of 0.29. The experiments showed that Nanocon HP 1010 has the best plasticizing effect – initial spread of 350 mm, and after 60 minutes the flowability was 150 mm. The polycarboxylate superplasticizer used in combination with microsilica improves the flowability (spread 690–730 mm, viscosity 8–9 seconds) and stability of the self-compacting concrete mixture, while reducing water separation. The 28-day compressive strength of the concrete reached 92.5 MPa (class B70), and the retention of flowability exceeded 3 hours. Thus, the complex organic-mineral admixture consisting of microsilica and polycarboxylate-based superplasticizer is the most effective additive for producing self-compacting concretes with high flowability, segregation resistance, and long workability retention.

Keywords: polycarboxylate, superplasticizer, self-compacting concrete, microsilica, rheological properties, strength

Məqaləyə AzMİU-nun “Meliorasiya və su təsərrüfatı tikintisi” kafedrasının dosenti N.A. Səfərova rəy vermişdir.