

Матеріали пропонованої статті присвячені інтенсивній роздільній технології виробництва самоущільнюючого бетону (СУБ). Обґрунтовується запропонована технологія виробництва СУБ можливістю ефективного управління в'язкістю цементоводних композицій, що забезпечують, в основному, рухливість бетонних сумішей. Окреме приготування цементоводної композиції в швидкісному змішувачі з подальшим її змішуванням з заповнювачами в ординарному бетонозмішувачі в корені змінює пріоритети технології виробництва бетонної суміші. Обґрунтовується ідея про те, що за допомогою роздільної технології можливо оптимізувати режими швидкісного змішування і окремо готувати висококонцентровані цементовміщуючі суспензії в умовах інтенсивних гідродинамічних впливів на них. Особлива увага приділяється вивченню впливу вмісту суперпластифікатора полікарбоксилатного типу Релаксол-Супер ПК, мікрокремнезему і поліпропіленової фібри на ефективну в'язкість цементовміщуючої суспензії. Наведено порівняльний аналіз впливу вхідних рецептурних факторів на її значення. Встановлено, що механоактивація цементовміщуючої суспензії в присутності добавки Релаксол-Супер ПК призводить до граничного руйнування її початкової структури, що необхідно для рівномірного розподілу мікрокремнезему і поліпропіленової фібри в об'ємі.

Виявлено вплив наведених вище рецептурних факторів на механічні характеристики СУБ. Показано, що інтенсивна роздільна технологія виготовлення самоущільнюючих бетонних сумішей забезпечує отримання СУБ з міцністю при стиску в 28-и денному віці не менше 55 МПа та покращує характеристики його зі стираності та ударної міцності

Ключові слова: механоактивація, самоущільнюючий бетон, ефективна в'язкість, суперпластифікатор, полікарбоксилат, мікрокремнезем, поліпропіленова фібра

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MECHANO-ACTIVATION OF PORTLAND CEMENT IN THE TECHNOLOGY OF MANUFACTURING THE SELF-COMPACTING CONCRETE

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1. Introduction

Concrete, due to a number of properties, remains one of the most widely used construction materials in the world [1]. The availability of raw materials, strength, durability, efficiency, environmental safety, ensure high competitiveness of concrete in the construction industry. Among the varieties of concretes, special place belongs to the self-compacting concretes (SCC). Given the exceptional movability, a freshly prepared concrete mix can independently, under the influence of its own weight, fill the mold, releasing at the same time the air entrained in the agitation process. The advantages of laying the self-compacting concrete mixes to structures, when compared to conventional mixes that require vibrocompression, are obvious. These in the first place include a decrease in the mass of a formwork, an increase in the volume of laying a concrete mix per shift, a decrease in the pitch between reinforcing rods, and the improved adhesive strength between concrete and armature.

In the technology of SCC production, the quality of applied ingredients (cement, finely dispersed minerals, granite rubble, quartz sand) plays a special role. Deviation from the required characteristics can cause segregation of the concrete mix and, consequently, lead to a deterioration of

the homogeneity of the hardened concrete inside the volume of an article or a structure. In addition, the presence of a finely dispersed mineral additive and polypropylene fibers create additional technological constraints for ensuring their uniform distribution in the volume of the concrete mix. A comprehensive solution to the issues related to both the homogenization of a concrete mix and an increase in the potential of cement in the process of its mechanoactivation, is achieved by employing the intensive separation technology (IST) for preparing a concrete mix. The combination of positive properties of the proposed compositions of self-compacting concretes and IST opens new avenues in concrete engineering, provides a solution to today's tasks related to the production of concrete and reinforced concrete with improved characteristics.

2. Literature review and problem statement

The diameter of spreading a standard concrete cone of the self-compacting concrete mix is defined both by the amount of highly concentrated cement suspension in it and its effective viscosity. When addressing the technology to produce SCC, paper [2] substantiates the appli-

cation of a water-reducing additive based on polycarboxylate ethers. It should be noted, however, that this work lacks any information on the impact of the concentration of the additive on both the effective viscosity of suspension and the properties of a self-compacting concrete. In practical terms, this may cause technological problems both in the transportation of a concrete mix and when laying to a structure.

Self-compacting concrete is characterized by special features of its composition, which ensure high fluidity of the concrete mix. One such feature is the mandatory presence of a finely dispersed mineral admixture, which provides for an increase in the viscosity of the concrete mix [3]. It should be noted, however, that these studies contain no information on the impact of the amount of a mineral admixture on effective viscosity of the highly concentrated cement-containing suspension. One should also assume that the improved connectivity of the concrete mix through the introduction to its composition of the increased quantity of a finely dispersed mineral admixture would increase its water requirement and, consequently, reduce the mechanical characteristics of concrete [4]. It is possible to improve the strength of concrete by the additional dry milling of Portland cement with mineral admixtures [5]. Despite the practical importance of such a technological operation, it should be noted that the result of storing the cement after post-milling is the decline in its activity and, as a consequence, reduced strength of the cement stone and the concrete based on it. A known technological technique that makes it possible to increase the strength of concrete is the introduction of fibrous fillers to the composition of the mix. It follows from paper [6] that it is possible to improve the mechanical characteristics of concrete only when attaining a homogeneous distribution of fibrous filler in the bulk of the mix, which is not always possible.

A review of the scientific literature revealed that obtaining the self-compacting concretes involves a number of technological difficulties relating to, among others, the concrete mix homogenization.

The most effective technological impacts on the highly concentrated suspensions of a binder are exerted by those that make it possible to reach the limit of destruction of the original structure of the system, characterized by the minimal indicators of its effective viscosity [7]. There is every reason to believe that one of the ways to accomplish this task is the application of intensive hydrodynamic influences (mechanoactivation) to the highly concentrated suspensions of a binder in mixers-activators. Enabling the mechanoactivation of the Portland cement particles and a finely dispersed mineral admixture in the medium, with which they react, would increase the potential of the binder and, consequently, improve the mechanical characteristics of concrete [8].

3. The aim and objectives of the study

The aim of present research was to obtain, based on the intensive separation technology, a self-compacting concrete with improved mechanical characteristics for compressive strength, abrasion, and impact resistance.

To achieve the set aim, the following tasks have been solved:

- to determine the effect of mechanoactivation on change in the effective viscosity of cement suspensions, which contain the admixture Relaxol-Super PC, polypropylene fibers, and microsilica;

- to estimate the impact of mechanoactivation of the highly concentrated cement suspensions and consumption of the examined admixtures on the mechanical characteristics of SCC: compressive strength, abrasion, and impact resistance.

4. Materials and methods to study the properties of suspension, concrete mix, and concrete

4.1. Original raw materials and equipment used in the experiment

We conducted the study using the pure clinker Portland cement with an activity of 48.0 MPa, obtained by jointly milling the Portland clinker and gypsum dihydrate (5 %) at a laboratory ball mill. We used, as a water-reducing additive to the Portland cement, the polycarboxylate superplasticizer Relaxol-Super PC. The role of the active mineral admixture belonged to microsilica whose concentration ranged from 0 to 10 % by weight of the Portland cement. The fibrous filler was the polypropylene fibers, 12 mm long. The role of a fine filler in the concrete mix belonged to quartz sand, $M_s=2.5$. The coarse filler was granite rubble with fractions of 5–10 and 10–20 mm. Activation of the binder was implemented in a high-speed mixer at the rotor rotation speed of 2,800 rpm.

4.2. Procedures for determining the properties of concentrated suspension, concrete mix, and concrete

We accepted that the rheological characteristic of the cement-containing suspension was effective viscosity η , determined at a rotational viscosimeter with coaxial cylinders. Determining the spread of the concrete mix was carried out using the inverted Abrams cone. The strength of concrete at compression was determined by testing the samples, cubes with an edge of 10 cm. We determined the abrasion of concrete at the abrasion circle LKI-2. Impact resistance of concrete was determined at a laboratory impact testing machine.

5. Results of studying the effective viscosity of cement-containing suspension and the strength of concrete samples

It was interesting to determine the impact of separate and joint effects of the admixture Relaxol-Super PC, microsilica (MS), and polypropylene fibers (F), on effective viscosity of the highly concentrated cement-containing suspension ($W/C=0.3$). We examined suspensions based on the in mechanoactivated binder and on the binder, which was not exposed to mechanical activation. The activation time for the binder was chosen from the condition for the destruction limit of the suspension's structure, which was registered based on the minimum value of its effective viscosity. The values of effective viscosity of the cement-containing suspension, depending on the time of activation, the content of MS , F , and the admixture Relaxol-Super PC, are given in Table 1.

Table 1

Influence of formulation-technological factors on change in the effective viscosity of cement-containing suspensions

No.	PC, %	MS, %	Super PC, %	F, %	Suspension activation time, sec					
					0	30	60	90	120	150
1	100	0	0	0	1,500	504	255	191	175	198
2	95	5	0		2,400	1,049	692	632	680	747
3	90	10	0		2,900	1,688	1,437	1,425	1,459	1,493
4	100	0	0.5		831	281	143	107	96	112
5	95	5	0.5		1,515	599	371	337	357	403
6	90	10	0.5		1,869	951	752	742	751	787
7	100	0	1		150	58	30	21	19	24
8	95	5	1		608	149	47	35	42	51
9	90	10	1		812	214	63	45	58	66
10	100	0	0	1	1,741	703	404	358	348	371
11	95	5	0		2,653	1,275	890	822	850	881
12	90	10	0		3,130	1,920	1,694	1,673	1,703	1,734
13	100	0	0.5		1,187	380	188	153	147	156
14	95	5	0.5		1,697	804	528	484	498	509
15	90	10	0.5		2,019	1,155	913	871	883	899
16	100	0	1		187	137	122	108	116	122
17	95	5	1		671	295	150	121	128	137
18	90	10	1		947	355	183	148	155	166

Analysis of experimental data suggests that the mechanical activation of aqueous compositions of cement leads to a decrease in their initial effective viscosity. The introduction of the admixture Relaxol-Super PC to suspension enhances the destruction of its initial structure in the process of mechanical activation, Table 1. It has been observed that the activation of cement suspension without adding the Relaxol-Super PC over 120 seconds leads to a decrease in the effective viscosity from 1,500 to 175 cP, that is almost by 9 times. Introduction of a water-reducing additive in the amount of 1 % by weight of the Portland cement, which was not exposed to mechanical activation, leads to the reduced η of suspension, from 1,500 to 150 cP, and, given the mechanical activation, to 19 cP. This allows you to argue about synergy, that is the strengthening of destruction of the original structure of the suspension by mechanoactivation in the presence of the admixture Relaxol-Super PC.

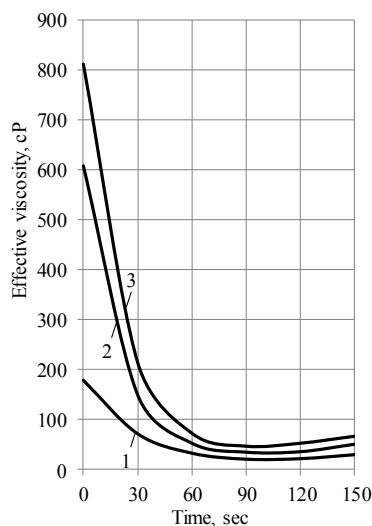


Fig. 1. Effect of activation time on the effective viscosity of suspension: 1, 2, 3 – concentration of MS 0; 5; 10 %

An increase in the duration of the Portland cement activation in suspension longer than 120 s is accompanied by the increased effective viscosity, which, in our opinion, is associated with the adsorption of free water at the newly formed surfaces of particles of the hardening cement slurry. A free water reserve reduction leads to an increase in the forces of particle-particle hardening and, consequently, to an increase in the viscosity of the cement-water composition. It should be noted that the introduction of microsilica to the Portland cement results in the increased effective viscosity of suspension at a preset water-cement ratio. This is especially noticeable for the suspensions that did not undergo mechanoactivation, Fig. 1. Thus, an increase in the concentration of MS from 0 to 10 % leads to the increase in η from 150 to 812 cP, that is by larger than 660 cP. After activating, for 90...120 seconds, the cement suspension that contains MS in the amount of 10 %, the value of effective viscosity decreases from 812 to 45 cP. The activation of the cement suspension without adding MS reduces η from 150 to 21.

The difference between the minimum values of effective viscosity of suspensions without adding MS and with it in the amount of 10 % is 24 cP, indicating that mechanoactivation largely eliminates the effect of MS on the effective viscosity of suspension. In the case of polypropylene fibers, their introduction to the composition of a cement-containing suspension has a minor effect on changing the effective viscosity. This is true for both the suspensions based on the mechanoactivated binder and the binder, which was not exposed to mechanical activation.

The next stage of the research relates to the impact of the original formulation-technological factors on the compressive strength of concrete, abrasion, and impact resistance.

We accepted as the base composition the concrete with the consumption of Portland cement in the amount of 460 kg/m³. Consumption of the fine and coarse filler was, accordingly, 785 kg/m³ and 835 kg/m³. We adopted the ratio between the mass fractions of crushed stone, 5–10 and 10–20, to be 1=0.82, which ensured its maximum bulk density. The amount of mixing water was chosen from the condition for obtaining a concrete mix with a spread of the cone not less than 60 cm. We performed two parallel series of experiments: one (control) – based on traditional technology, two – based on the separation technology with an activation in a high-speed mixer of cement suspension with the required amount of microsilica and polypropylene fibers. Upon activation, the suspension was fed to a regular concrete mixer, with the components of the concrete mix; their dosage was set in advance, quartz sand, granite rubble. We used, as a water-reducing additive to the concrete mix, Relaxol-Super PC in the amount from 0.5 to 1.5 % by weight of the binder. Compressive strength of concrete was determined at the age of 3 and 28 days of normal aging; the abrasion and impact resistance – at the age of 28 days. To determine the influence of the admixture Relaxol-Super PC, polypropylene fibers, and microsilica on the mechanical characteristics of SCC, we designed a 3-factor experiment. We accepted the following levels of variation in the above factors:

X_1 – content of polypropylene fibers in the binder, 0.5±0.5 %;

X_2 – content of microsilica in the binder, 5±5 %;

X_3 – content of the admixture Relaxol-Super PC in the binder, 1.0±0.5 %.

Table 2 gives the levels of independent factors, and the results of experiments.

Table 2

Levels of variation in the independent factors and the results of experiments

No.	Factor variation levels			Polypropylene fibers, % by weight of the binder	Microsilica, % by weight of the binder	Super PC, % by weight of the binder	Responses											
	X_1	X_2	X_3				f_{com}^c , MPa	f_{com}^m , MPa	f_m^c , MPa	f_m^m , MPa	G^c , g/cm ²	G^m , g/cm ²						
													Age					
													3 days	28 days	3 days	28 days	28 days	28 days
0	1	2	3	4	5	6	7	8	9	10	11	12						
1	-	-	-	0	0	0.5	10.0	24.7	11.9	29.4	0.33	0.27						
2	-	+	-	0	10	0.5	13.7	33.2	16.3	40.4	0.35	0.29						
3	0	0	-	0.5	5	0.5	12.4	30.1	14.9	36.8	0.26	0.22						
4	+	-	-	1	0	0.5	10.4	26.3	12.5	31.2	0.19	0.15						
5	+	+	-	1	10	0.5	14.2	34.2	17.1	41.0	0.21	0.17						
6	-	0	0	0	5	1	14.6	35.5	18.3	43.0	0.3	0.24						
7	0	-	0	0.5	0	1	12.6	31.3	15.2	39.7	0.21	0.17						
8	0	0	0	0.5	10	1	15.2	36.6	18.4	44.5	0.22	0.18						
9	0	+	0	0.5	5	1	16.1	39	19.6	47.6	0.23	0.19						
10	+	0	0	1	0	1	15.5	37.1	18.7	45.4	0.16	0.13						
11	-	-	+	0	10	1.5	14.4	35.1	17.1	42.9	0.27	0.22						
12	-	+	+	0	5	1.5	18.4	44.9	22.4	54.3	0.29	0.23						
13	0	0	+	0.5	0	1.5	16.5	40.2	20.0	49.8	0.2	0.16						
14	+	-	+	1	10	1.5	15.3	37.1	18.3	45.7	0.14	0.11						
15	+	+	+	1	5	1.5	18.7	45.4	22.8	57.7	0.16	0.13						

Note: f_{com}^c – strength of concrete whose binder did not undergo mechanoactivation, MPa; f_m^c – concrete strength, MPa; G^c – abrasion of concrete whose binder did not undergo mechanoactivation, g/cm²; G^m – abrasion of concrete based on the mechanoactivated binder, g/cm²

The result of statistical processing of experimental data is the polynomial models (1), (2) for the dependence of compressive concrete strength on the independent factors X_1 , X_2 , X_3 :

$$f_{com}^c = 36,2 + 0,8X_1 + 0,3X_1^2 - 0,3X_1X_2 + 4,2X_2 - 0,9X_2^2 + 0,2X_2X_3 + 5,3X_3 - 0,4X_3^2, \quad (1)$$

$$f_{com}^m = 44,3 + 1,1X_1 - 0,1X_1X_2 + 0,5X_1X_3 + 5,2X_2 - 0,6X_2^2 + 7,2X_3 - 0,9X_3^2. \quad (2)$$

An analysis of mathematical model (1) shows that the decisive influence on the strength of concrete is exerted by its content of the admixture Relaxol-Super PC. When increasing the content of admixture from 0.5 to 1.5 % by weight of the binder, we observed an increase in the concrete strength of 30÷35 % on average. The next most important influence on the strength of concrete is exerted by the consumption of microsilica in the binder. Increasing its concentration in the Portland cement from 0 to 10 % leads to an increase in the concrete strength by 20÷25 %. As far as the dispersed reinforcement is concerned, the introduction to the composition of a concrete mix of 1 % of polypropylene fibers leads to an increase in the concrete strength by not larger than 5÷7 %. The role of the polypropylene fibers dramatically increases in terms of the impact resistance and abrasion of concrete. Graphical dependences, shown in Fig. 2, demonstrate that increasing the amount of the polypropylene fibers introduced to concrete leads to a decrease in the magnitude of abrasion from 0.29 g/cm² (fibers are absent) to 0.16 g/cm² (the amount of fibers is 1 %). For the self-compacting concrete, manufactured using IST, the abrasion reduces and ranges from 0.13 to 0.23 g/cm², which is almost 20 % lower compared with the concrete not exposed to mechanoactivation.

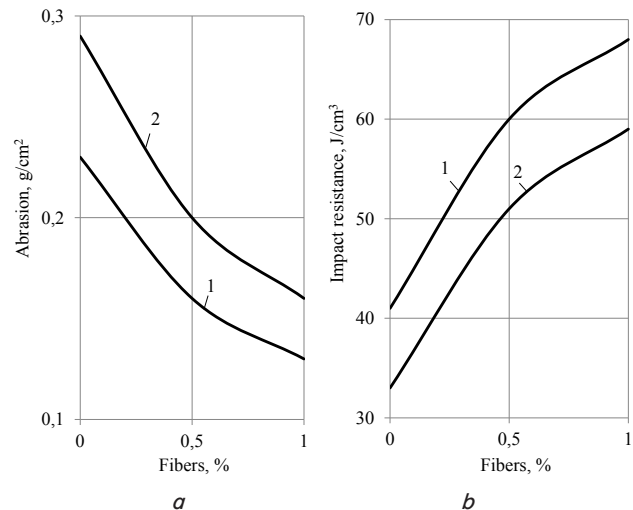


Fig. 2. Effect of the content of polypropylene fibers in the binder on abrasion (a) and impact resistance of concrete (b).

The levels of factors X_2 and X_3 are fixed at the level + 1:

- 1 – concrete based on the mechanoactivated binder;
- 2 – concrete on the binder not exposed to mechanical activation

The positive influence of polypropylene fibers is also observed for the impact resistance of concrete, Fig. 2, a. Enhancing its content from 0 to 1 % leads to the increased impact resistance, from 41 to 69 J/cm³, that is larger than by 40 %.

Joint introduction to the composition of the concrete mix of a water-reducing additive, microsilica, and fibers increases the strength of the self-compacting concrete by 40÷45 % compared with control (control is understood to be the strength of a self-compacting concrete without adding

microsilica and polypropylene fibers. The concentration of Relaxol-Super PC is 0.5 % by weight of the Portland cement.

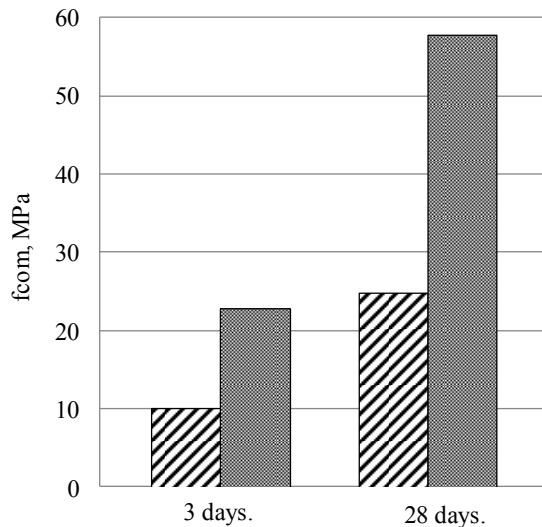


Fig. 3. Effect of the technology for preparing a concrete mix on strength of the self-compacting concrete: ▨ – control; ■ – separation technology, variation levels of factors X_1 , X_2 , X_3 are fixed at +1

We experimentally determined the strengthening of influence of the examined formulation factors on the strength of SCC for the case of preparing concrete mix using the separation technology with the application of the binder activation (model 2). The values of compressive concrete strength, shown in Fig. 3, indicate that the impact of the formulation factors (F , MS , Super PC) in combination with the binder mechanoactivation ensure a sharp increase in the concrete strength compared with control. Thus, the strength of concrete based on the mechanoactivated binder at the age of 3 days is 22.8 MPa, which is 2.2 times higher than that of control. At the age of 28 days, the strength of concrete based on the mechanoactivated binder exceeds the strength of control samples by 2.3 times.

6. Discussion of results of studying the effective viscosity of suspensions and compressive strength of the self-compacting concretes

It is known that the main structure-forming component of a concrete mix is the highly concentrated cement suspensions (cement slurry), characterized by the highly developed interface between the solid and liquid phases [10–18]. It is known that the strength of interaction between particles of the dispersed phase in coagulation structures is on average $10^{-9} \dots 10^{-12}$ N per contact [19, 20]. The binding energy of coagulation contacts drops sharply when the particles are covered by a monolayer of the surface-active substance (SAS). When adsorbed, SAS push the particles apart at a distance of a minimum of two molecular layers, shielding at the same time the most energetically active sites of their surface [12, 18–21]. Coagulation structures are distinguished by the pronounced dependence of structural-mechanical characteristics on the impact of mechanical factors. The limit of destruction for the original coagulation structure is

characterized by the minimum value of effective viscosity. Proof of this are the results of research into effective viscosity of the highly concentrated cement suspension ($W/C=0.3$), exposed to the activation at a high-speed mixer ($n=2,800$ rpm) with the water-reducing admixture Relaxol-Super PC, Table 1. High-speed mixing of the suspension in the presence of admixture makes it possible to reach the limit of destruction for the original structure of the cement-water composition, which is expressed by a decrease in effective viscosity by tens of times compared to the original.

Introduction of microsilica to the composition of Portland cement (up to 10 %) leads to an increase in the effective viscosity of cement-containing suspensions whose binder was not exposed to mechanoactivation. It was experimentally determined that the mechanoactivation almost eliminates the effect of microsilica on the water content of suspensions. This is especially evident in the cement-containing suspensions with the water-reducing admixture Relaxol-Super PC, Fig. 1.

The positive effect of the binder mechanoactivation is also expressed in the technology for preparing the self-compacting concrete mixes [16]. Along with the fact that mechanoactivation helps reduce the water content of a concrete mix, it also intensifies the process of concrete hardening, Fig. 3.

Accelerated acquiring of the strength of concrete based on the mechanoactivated Portland cement, in our view, is due to the intensification of forming the new hydration products of the hardening cement [9].

The structural features of the high-speed mixer-activator make it possible to create turbulent flows in a cement-containing suspension. When particles of the solid phase collide, there is, in the contact zone, a complex of physical, chemical, and mechanical phenomena and processes, which change their surface properties completely. Because such interactions occur in turbulent flows in the activation period, then, according to [22], products of the new phase are torn off the surface of the particles and pass into the volume of the dispersed medium. Thus, mechanoactivation creates all the necessary prerequisites for accelerating the processes of hydration, structure-formation, and the concrete strength acquisition.

With a range of positive qualities (increased activity of the binder and mineral admixtures in the process of their mechanical activation, a drastic reduction of effective viscosity in the highly concentrated suspensions, homogeneous distribution of finely dispersed components in the volume of a mix), the intensive separation technology requires further improvement. The range of the suspension activation modes should be expanded, which will make it possible to abandon the drying of finely dispersed mineral admixtures to constant weight, to utilize “stale” cements, to adjust temperature mode of the activation of cement-water compositions. Undertaking appropriate research would provide an opportunity to create multilevel resource-saving composite materials with the predefined physical-mechanical characteristics.

7. Conclusions

Mechanical activation of the highly concentrated suspension in the presence of water-reducing admixture Relaxol-Super PC, microsilica, and polypropylene fibers, leads to

the destruction of the primary structure, which is expressed by a decrease in effective viscosity from 1,500 to 148 cP, that is larger than by 10 times.

The intensive separation technology for preparing the self-compacting concrete mix using the mechanoactivated

cement-containing highly concentrated suspension ensures, at the age of 28 days, an increase in the strength of concrete at compression, compared with control, by 2.3 times; impact resistance – by 15 %. The application of IST reduces the abrasion of concrete by 20–25 %.

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