materials proceedings

Proceeding Paper

High-Strength Concrete Using Ash and Slag Cements †

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† Presented at the 10th MATBUD'2023 Scientific-Technical Conference "Building Materials Engineering and Innovative Sustainable Materials", Cracow, Poland, 19–21 April 2023.

Abstract: The article discusses the possibility of improving the properties of concrete made with composite ash-containing cement (CC) by introducing complex chemical additives. The pozzolanic activity of CC and its degree of hydration increases with the increasing dispersion of cement resulting from the introduction of a complex additive in the form of a polyfunctional modifier (PFM) during grinding, including a grinding intensifier-propylene glycol and a superplasticizer. The analysis of mathematical models of water demand and strength of concrete made with the PFM additive showed that a significant reduction in water demand and an increase in the strength of concrete based on the ash-containing CC is possible with the introduction of the PFM additive. For concrete based on CC with the addition of PFM, it has been demonstrated that it is possible to design concrete mixes on the basis of the obtained models. Relevant examples are provided.

Keywords: high-strength concrete; composite cement; fly ash; slag; multifunctional modifier; planning of experiments

1. Introduction

As is known, high-strength concrete currently includes concrete with a compressive strength of 60–100 MPa and higher at the age of 28 days [1,2]. With the use of classic technology, obtaining high-strength concrete is possible by ensuring the high quality of raw materials, low values of the water-cement ratio, and sufficient compaction of the concrete mixture. In practice, these requirements are achieved by reducing the water content of the concrete mixture when using stiff mixtures, or by introducing plasticizing additives, increasing the activity and specific surface of cement, introducing hardening accelerators, reducing water consumption, and optimizing the grain composition of aggregates with high physical and mechanical properties [1–6].

The technology of high-strength concrete of the new generation involves the introduction of superplasticizer additives with a high (20–40%) water-reducing effect and dispersed active mineral fillers into the composition of concrete mixtures. These components provide both important individual effects, namely achieving extremely low values of the water-cement ratio while maintaining ease of installation, an increase in the volume of hydration products, the degree of cement hydration, and a significant synergistic effect. With the simultaneous introduction of superplasticizer additives and a dispersed active filler, the rheological potential of the plasticizing additive is fully realized on the one hand, whereas on the other hand, in the conditions of a limited water-cement ratio, the positive effect of the dispersed active filler on the structure formation of cement stone and concrete is manifested [7–10].

In hydration systems, the physical interaction of cement particles and the filler is significantly affected by the so-called "compressed conditions" [11], which are characterized by a sharp increase in the concentration of the solid phase and the transition of part of the volume water into film water. At the same time, the change in free surface energy

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Citation: Dvorkin, L.; Zhitkovsky, V.; Marchuk, V.; Makarenko, R. High-Strength Concrete Using Ash and Slag Cements. *Mater. Proc.* **2023**, *13*, 16. https://doi.org/10.3390/materproc2023013016

Academic Editors: Katarzyna Mróz, Tomasz Tracz, Tomasz Zdeb and Izabela Hager

Published: 14 February 2023

between the solid and liquid phases contributes to the intensive formation of crystal nuclei of hydration products, which is in accordance with the Gibbs–Volmer theory [12]. With the optimal concentration and dispersion of the active additive, a fine-grained structure of the binder is formed, which has a positive effect on the technical properties of the artificial stone.

The structuring effect of dispersed additives in concrete increases when the physicochemical activity of their surface increases. Methods of mechanical activation of additives include grinding them with cement in the presence of plasticizers and, if necessary, other chemical admixtures. This method of activation is the basis for obtaining composite cement with low water consumption [13].

An important indicator of the quality of concrete as a structural material is the specific consumption of cement per unit of concrete strength. In traditional concretes, this indicator is on average close to 10 kg/MPa, in new generation concretes it decreases two or more times [7–9].

The best active fillers for high-strength concrete, as demonstrated by many studies and by practice, are microsilica additives [14,15]. At the same time, the use of microsilica is complicated due to the need for its granulation or briquetting for transportation and dosing, as well as to significant fluctuations in its composition and properties.

As regards active fillers for high-strength concrete, the use of dispersed siliceous and aluminosilicous materials based on local raw materials and industrial waste is a practical interest [14,16]. Among such materials, the most common is coal fly ash and granulated blast furnace slag. The main task of our work was to figure out the technological parameters for obtaining high-strength concrete using composite ash and slag-containing cement and to research their complex structure and technical properties.

2. Materials and Methods

The main studies were performed using (EN 197-1):

Composite CEM V/A with a fly ash content of 27.4%, blast furnace slag—18.3%, clinker—45.7%, and gypsum—8.6%;

Portland cement clinker with mineralogical composition was used in the research: C_3S—63.95%; C_2S—15.15%; C_3A—7.42%, C_4AF—12.48%.

Fly ash and blast furnace granulated slag have the following chemical composition:

- fly ash: (SiO_2 + Al_2O_3 + Fe_2O_3)—85.8%, SO_3—2.3%, CaO_{free}—2.8%, MgO—2%, Na_2O + K_2O—1.2%, LOI—5.1%;
- blast furnace slag: SiO_2—39.5%, Al_2O_3—6.4%, Fe_2O_3—0.2%, CaO—47.2%, MgO—3.1%, MnO—1.1%, SO_3—1.7%.

The Samples of composite cement for research were obtained by joint grinding of the components in the laboratory ball mill. To regulate the properties of the composite cement and the concretes made with the use thereof, the influence of naphthalene formaldehyde (SP-1) and polycarboxylate (Sika VS 225) superplasticizers [17] and grinding intensifier-propylene glycol (PG) [18] was used.

For mineral additives, the pozzolanic activity was determined by the CaO uptake method [19,20]. Additives (ash, slag, and their mixture) before the test were milled to the required specific surface area (Sssa) (Table 1).

Table 1. Pozzolanic activity of fly ash and ash of slag composition.

Material	Sssa, m^2/kg	Absorption of CaO mg/g		
		7 Days	28 Days	60 Days
Fly ash	350	15	52	78
Fly ash	450	18	65	97
Fly ash	550	25	90	135
Fly ash + slag (1:1)	350	20	70	83
Fly ash + slag (2:1)	350	17	63	81
Fly ash + slag (1:1)	450	21	83	105
Fly ash + slag (2:1)	450	19	73	101

For the obtained composite cements, the grain composition (Table 2) was determined by the sedimentation method [21]. Also, the normal consistency, the change in the degree of hydration of the cement paste [22], and the standard strength of cement-sand samples over time were determined (Table 3) [23].

Table 2. Composite cements grain composition.

No.	Additives	Content of Fractions, %				
		<10 µm	10–20 µm	20–40 µm	40–60 µm	>60 µm
1	PG—0.04%, Sika VC 225—0.5%;	35.5	33.1	15.5	12.4	3.3
2	PG—0.04%	31.2	36.4	14.2	14.6	3.6
3	PG—0.02%, Sika VC 225—0.5%;	28.2	36.1	16.7	15.1	3.9
4	PG—0.02 %	26.5	33.7	18.4	17.2	4.2
5	PG—0.04%, SP-1—0.5%	29.8	35.5	14.3	15.8	4.6
6	SP-1—0.5%	22.8	35.1	19.6	17.3	5.4
7	Sika VC 225—0.5%	17.4	36.6	21.8	17.5	6.7
8	Without additives	15.6	35.5	22.5	18.2	8.2

Table 3. The main properties of composite cement (CC).

No.	Specific Surface Area S_{ssa}, m^2/kg	Additive PFM, %	Normal Consistency, %	Compressive/Bending Strength, MPa in Age, Days			
				1	3	7	28
1	350	without additives	27.8	15.5/2.5	22.4/3.2	31.6/4.1	41.5/5.8
2	450	–//–	28.3	19.3/3.1	25.5/3.6	39.7/4.8	52.3/6.2
3	450	PFM_1 (PG—0.04%, Sika VC 225—0.5%)	18.5	24.7/4.1	32.3/4.8	45.8/5.6	61.5/6.7
4	550	–//–	19.7	30.3/4.3	39.6/4.9	50.6/6.1	71.8/7.5
5	450	PFM_2 (PG—0.04%, SP-1-0.5%)	21.5	21.8/3.6	28.3/4.1	37.4/4.6	57.8/6.8
6	550	–//–	22.8	25.2/3.9	31.3/4.2	41.2/5.6	61.3/7.2

To analyze the influence of the composition of binders and concrete, as well as modifier additives under normal conditions of hardening and when subjected to heat treatment, experimental-statistical models were obtained using the method of mathematical planning [24–26]. When studying concrete mixtures and concretes, the three-level B4 plan was implemented [26]. According to plan B4, 24 series of experiments were performed. In each series, the water demand was determined until the specified slump was reached, and 6 concrete cubes 100 × 100 × 100 mm were made to determine the compressive strength [27]. To obtain concrete mixtures, quartz sand with fineness modulus $M_f = 1.95$

and crushed stone with D_{max} = 20 mm were used. Samples were tested after 1 and 28 days. The conditions for planning the experiments are given in Table 4. After the implementation and statistical processing of experiments, mathematical models of the water consumption and the concrete compressive strength were obtained.

Table 4. Conditions for planning experiments when obtaining models of the concrete mixture water consumption and the concrete at the CC strength.

No.	Factors		Levels of Variation			Interval
	Natural	Coded	–1	0	+1	
1	The content of PFM_1 additive in CC, %	X_1	0.4	0.7	1.0	0.3
2	Specific surface area of CC, S_{ssa}, m^2/kg	X_2	350	450	550	100
3	Water-cement ratio, *W/C*	X_3	0.25	0.35	0.45	0.1
4	Slump, Sl, cm	X_4	2	13	24	11

3. Results and Discussion

Experiments on laboratory ground cement showed that the pozzolanic activity of the ash-slag composition in the cement most significantly depends on the fineness of the grind, characterized by the specific surface area (S_{ssa}), and, to a lesser extent, on the ash:slag ratio (Table 1). However, decreasing the ratio of ash to slag significantly affects the kinetics of CaO bonding, increasing the amount of bound CaO, especially in the period from 7 to 28 days.

Achieving an increased composite cement's specific surface area while minimizing energy consumption is possible with the use of grinding intensifier additives [28,29]. The propylene glycol (PG) has become widespread as such an additive. Along with the propylene glycol, the addition of superplasticizers and their compositions with PG have a certain influence on the grinding kinetics and grain composition of cement. The experiment results are shown in Figure 1 and Table 2.

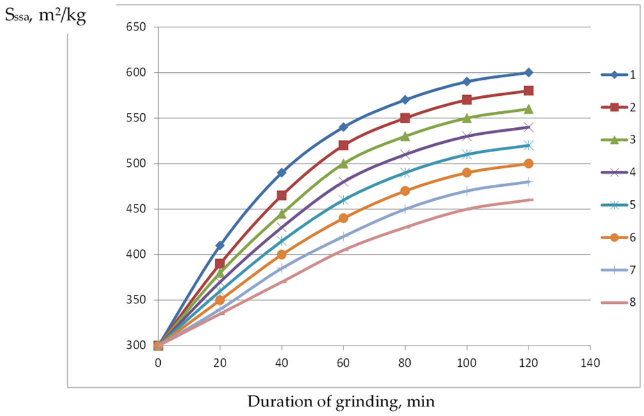

Figure 1. The influence of additives on the specific surface area of CC at different durations of grinding. 1—PG—0.04%, Sika VC 225—0.5%; 2—PG—0.04%; 3—PG—0.02%, Sika VC 225—0.5%; 4—PG—0.02%; 5—SP-1—0.5%, PG—0.04%; 6—SP-1—0.5%; 7—Sika VC 225—0.5%; 8—without additives.

The addition of PG practically doubled the content of the cement's finest fraction. The composite additive PG + Sika VC 225 provides a grain composition of cement that is slightly different from the grain composition of cement with a single PG additive. The naphthalene-formaldehyde superplasticizer introduction into the cement also to some extent intensifies the grinding of the cement. This effect is significantly lower than the propylene glycol effect.

Enrichment of composite cement during grinding with the finest fractions significantly accelerates its hydration. This is evidenced by the results of experiments on determining the content of hydrated water in samples of cement stone with $W/C = 0.3$ at different durations of their hardening (Figure 2).

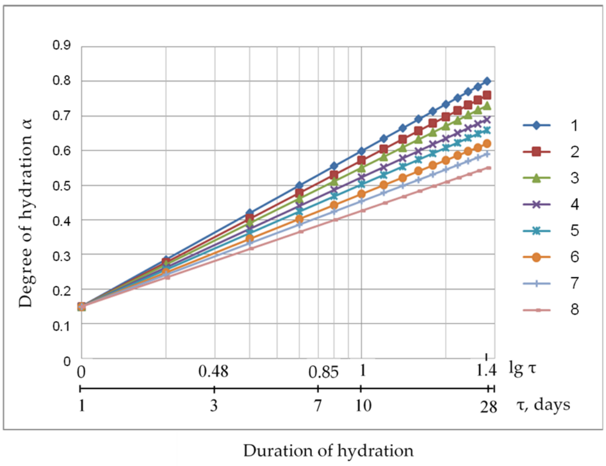

Figure 2. The influence of the hydration duration on the degree of hydration of composite cements (α) (line numbering is given for cements according to Figure 1).

The hydration kinetics of composite cements ground with PG additives, superplasticizers SP-1 and Sika VC 225 can be approximated by a general equation [18]:

$$\alpha = k \lg \tau + B \tag{1}$$

where τ—hydration duration; k i B—coefficients depending on the additives type and grain composition of cements.

Complex additives, including grinding intensifier (PG) and superplasticizers SP-1 and Sika VC 225 can be considered polyfunctional cement modifiers (PFM):

- PFM_1—PG + Sika VC 225;
- PFM_2—PG + SP-1.

The composite cements' main properties values, which were obtained by grinding in a laboratory ball mill, are given in Table 3. It follows from them that the strength of composite cement with a clinker content of 50% when adding PFM_1 and $\text{S}_{\text{ssa}} = 450\ \text{m}^2/\text{kg}$ reaches 60 at the age of 28 days, $\text{S}_{\text{ssa}} = 550\ \text{m}^2/\text{kg}$—70. When PFM_2 is introduced, it is 55 and 60 MPa, respectively. At the same time, at the 3 days, the strength of cement with PFM_1 reaches 50% of the 28-day strength.

The study of the concrete mixtures and concrete properties based on modified composite cement with the addition of PFM_1 using mathematical planning of experiments (Table 4) made it possible to obtain mathematical models of the concrete mixture water demand (W) and the concrete compressive strength at the 1 ($f^{1}{}_{\text{cm}}$) and 28 days ($f^{28}{}_{\text{cm}}$):

$$\begin{aligned} W = {} & 142.1 - 22X_1 + 4.17X_2 - 10.1X_3 + 17.1X_4 + 6X_1^2 + 1.5X_2^2 + 6.6X_3^2 - 1.5X_4^2 + 0.6X_1X_2 - 0.6X_1X_3 + \\ & + 1.3X_2X_3 - 0.3X_2X_4 + 0.4X_3X_4 \end{aligned} \tag{2}$$

$$f'_{cm} = 33.1 + 1.3X_1 + 8.9X_2 - 7.5X_3 - 1.2X_1^2 - 4.1X_2^2 + 2.8X_3^2 + 0.3X_1X_2 - 0.3X_1X_3 - 2.5X_2X_3 \tag{3}$$

$$f_{cm}^{28} = 70.9 - 0.3X_1 + 9.8X_2 - 13.8X_3 - 2.5X_1^2 - 4.28X_2^2 + 3.78X_3^2 + 3.9X_2X_3 \tag{4}$$

For the concrete compressive strength models, the influence of concrete mixture slump, provided that other varied factors are constant, was statistically insignificant. Factors can be arranged in the following sequence according to the decreasing influence on the studied properties:

$$W : X_1 > X_4 > X_3 > X_2$$
$$f'_{cm} : X_2 > X_3 > X_1$$
$$f_{cm}^{28} : X_3 > X_2 > X_1$$

When analyzing models of water consumption of a concrete mixture (Figures 3 and 4), attention is drawn to the practically identical values of water consumption of a concrete mixture in the interval $W/C = 0.45 - 0.35$ and its significant increase at $W/C < 0.35$. this confirms the well-known [1] rule of water consumption constancy at $W/C < W/C_{critical}$. At the same time, the results of the study show that the polycarboxylate superplasticizers addition makes it possible to shift the critical value $W/C_{critical}$ towards lower values. For concrete without superplasticizers, it is usually in the range of 0.43–0.4 [28]. The water demand model also reflects the increase in the concrete mixture water demand as the specific surface area of the cement increases.

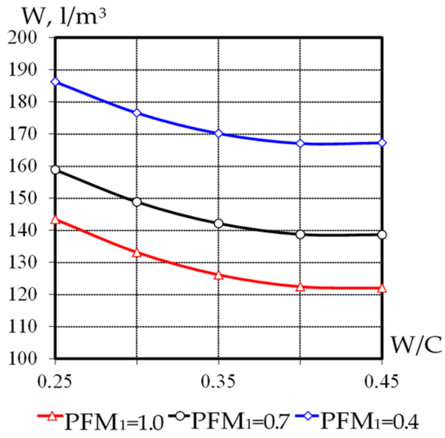

Figure 3. The influence of W/C and PFM_1 additives on the concrete mixture water consumption (S_{ssa} = 450 m^2/kg, Sl = 13 cm).

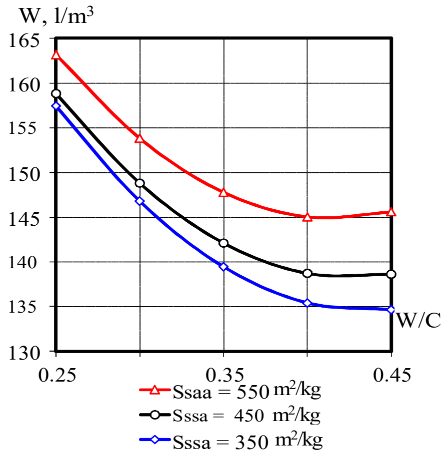

Figure 4. The influence of W/C and the specific surface area of the CC on the concrete mixture water consumption (PFM_1 = 0.7%, Sl = 13 cm).

Analysis of the strength mathematical models (Figures 5–7) shows the unconditional leading value of the factors W/C and Sssa. The influence of the specific surface area of the CC increases significantly for the one-day strength of the concrete. With increased values of

Spyt, the effect of increasing W/C on the early strength of concrete becomes less significant (Figure 5).

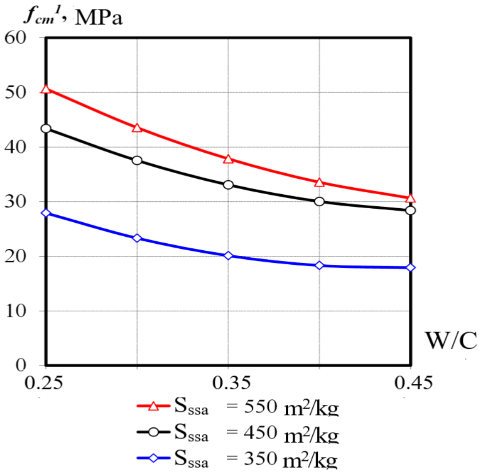

Figure 5. The influence of *W/C* and specific surface area on the concrete compressive strength at the 1 day (PFM_1 = 0.7%).

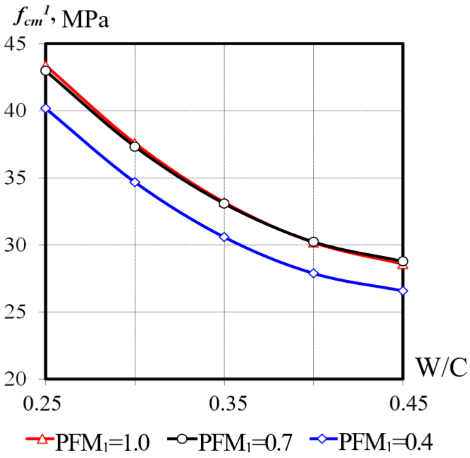

Figure 6. The influence of *W/C* and PFM_1 content on the concrete compressive strength at the 1 day (S_{ssa} = 450 m^2/kg).

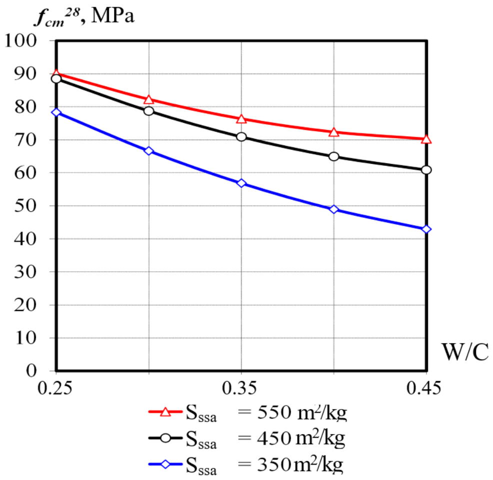

Figure 7. The influence of *W/C* and specific surface area on the concrete compressive strength at 28 days (PFM_1 = 0.7).

The models make it possible to design compositions of the concrete mixture on the CC with the PFM, specified workability, and strength.

Numerical Example

It is necessary to design the concrete class C 40/50 composition with compressive strength at a 1 day of not less than 30% 28 of the daily strength with the slump Sl = 13 cm when using composite cement with S_{ssa} = 350 m^2/kg and PFM_1 0.7% of the mass of cement. Materials: granite crushed stone fractions 5–20 mm with true density (ρ_{cs}) -2720 kg/m^3 and bulk density (ρ_{bcs}) -1350 kg/m^3, sand with true density (ρ_s) -2650 kg/m^3.

1. Let us find the required average strength of concrete class C 40/50, determined on sample cubes with a variation coefficient of 13.5% [1]. $f_c{}^{28}$ = 50: 0.778 = 64.3 MPa
2. With the consumption of the additive PFM_1 = 0.7% (X_1 = 0) and the specific surface area of the CC S_{ssa} = 350 m^2/kg ($X_2 = -1$), calculate the necessary *W/C* from model (4). *W/C* = 0.311 With *W/C* = 0.311 ($X_3 = -0.39$), S_{ssa} = 350 m^2/kg ($X_2 = -1$), as well as the slump Sl = 13 cm (X_4 = 0), the concrete at one-day strength from model (3) is 22.4 MPa, which exceeds 30% $f_c{}^{28}$. Thus, for further calculations, we accept *W/C* = 0.311.
3. By model (2), when $X_1 = 0$, $X_2 = -1$, $X_3 = -0.39$, $X_4 = 0$, we will find the concrete mixture water demand: *W* = 145 L/m^3
4. Cement Consumption: $C = \frac{W}{W/C} = \frac{145}{0.311} = 466\ \text{kg/m}^3$
5. Let us find the crushed stone and sand consumption, applying known formulas [1,2].

$$Cs = \frac{1000}{\alpha\frac{v_{cs}^{\text{II}}}{\rho_{bcs}} + \frac{1}{\rho_{cs}}} = \frac{1000}{1.375\frac{0.5}{1.35} + \frac{1}{2.72}} = 1140\ \text{kg/m}^3$$

where *α*—coefficient of displacement of coarse aggregate grains,

$V_{cs}{}^{p}$—the crushed stone intergranular voids.

$$S = \left(1000 - \left(\frac{C}{\rho_c} + W + \frac{Cs}{\rho_{cs}}\right)\right)\rho_s = \left(1000 - \left(\frac{466}{3.1} + 145 + \frac{1140}{2.72}\right)\right) \times 2.65 = 757\ \text{kg/m}^3.$$

Calculated composition of concrete, kg/m^3: *C* = 466 kg/m^3, *W* = 145 kg/m^3,

Cs = 1140 kg/m^3, *S* = 757 kg/m^3, PFM_1 = 466 × 0.007 = 3.26 kg/m^3, (PG = 0.0004*C* = 0.18 kg/m^3, SP = 0.0066*C* = 3.07 kg/m^3).

4. Conclusions

1. The rational ratio of fly ash and blast-furnace granulated slag in the composition of the composite additive provides its increased pozzolanic activity, which increases significantly with an increase in the cement-specific surface area.
2. The joint addition of propylene glycol and superplasticizers into the composite cement during its grinding ensures that its specific surface is achieved without a significant increase in the duration of grinding and, as a result, a significant increase in the degree of hydration and strength, especially in the early stages of hardening.
3. Using mathematical planning, experimentally obtained experimental-statistical models of water demand, 1 daily and 28 daily strength of concrete on composite cement containing a polyfunctional modifier, including a polycarboxylate superplasticizer and a grinding intensifier. The models made it possible to quantify the influence of the main factors of concrete compositions and their interaction, as well as to design compositions of high-strength concretes with given values of workability and strength.

Author Contributions: Conceptualization, L.D. and V.Z.; methodology, R.M.; software, V.Z.; validation, V.M. and R.M.; formal analysis, L.D.; investigation, R.M.; resources, R.M.; data curation, V.Z.; writing—original draft preparation, V.M.; writing—review and editing, L.D.; visualization, V.Z.; supervision, L.D.; project administration, R.M.; funding acquisition, R.M. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Acknowledgments: In this section, you can acknowledge any support given which is not covered by the author contribution or funding sections. This may include administrative and technical support, or donations in kind (e.g., materials used for experiments). The publication cost of this paper was covered with funds from the Polish National Agency for Academic Exchange (NAWA): "MATBUD'2023-Developing international scientific cooperation in the field of building materials engineering" BPI/WTP/2021/1/00002, MATBUD'2023.

Conflicts of Interest: The authors declare no conflict of interest.

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