doi: 10.18720/MCE.72.9

Fiber fine-grained concretes with polyfunctional modifying additives

Дисперсно-армированные мелкозернистые бетоны с полифункциональными модифицирующими добавками

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Key words: fine-grained concretes; modifiers; dispersive fiber; mineral additive; optimization; limit of tensile strength in bending; limit of compressive strength

Ключевые слова: мелкозернистый бетон; модификаторы; дисперсное волокно; минеральная добавка; оптимизация; предел прочности на растяжение при изгибе; предел прочности при сжатии

Abstract. The purpose of the experimental study was to research the efficiency of dispersive reinforcement and modifying of compositions of cement fiber fine-grained concretes with active mineral and chemical additives, and to optimize the developed compositions according to the strength efficiency criteria. Multicriteria optimization of compositions and properties of modified fiber fine-grained concretes is an urgent task in connection with the complexity of their formulations. The experimental study was planned on the basis of a D-optimal plan containing 15 experiments. Upon the experimental results, experimental and statistical models were built to reflect the dependencies of the limit of compressive strength and tensile strength in bending of fiber fine-grained concretes on the type and concentration of modifiers (mix I) and dispersive fibers (mix II). Analysis of study results of saturated D-optimal plan was carried out on triangular diagrams Gibbs-Roseboom, they was built with use polynomial models of "mixture I, mixture II, technology – properties" and allowed to trace influence of 6 to variable factors in two-dimensional space. Optimum fields of variation of fine-grained modified fiber concrete components are identified with use method of experimental-statistical modeling. The efficiency of modifying fine-grained concretes with polyacrylonitrile fiber, astralene-modified basalt microfiber as well as with highly active metakaolin has been established. Use of these modifiers in the compositions allowed to obtain cement composites with a wide range of strength characteristics: from 30 to 53 MPa at compression, from 3.7 to 6.6 MPa for bending tensile.

Аннотация. Целью экспериментального исследования являлось изучение эффективности дисперсного армирования и модифицирования активными минеральными и химическими добавками составов цементных дисперсно-армированных мелкозернистых бетонов, а также оптимизация разработанных составов по прочностным критериям эффективности. Многокритериальная оптимизация составов и свойств дисперсно-армированных модифицированных мелкозернистых бетонов является актуальной задачей в связи с многокомпонентностью их рецептур. Планирование экспериментального исследования осуществлялось на основе D-оптимального плана, содержащего 15 опытов. По результатам эксперимента производилось построение экспериментально-статистических моделей зависимости предела прочности при сжатии и на растяжение при изгибе мелкозернистых бетонов от вида и содержания модифицирующих добавок (смесь I) и дисперсных волокон (смесь II). Анализ результатов исследования осуществлялся по треугольным диаграммам Гиббса-Розебома, построенным по полиномиальным моделям типа «смесь I, смесь II, технология – свойства», позволяющим проследить влияние 6 варьируемых факторов в двухмерном пространстве. С помощью метода экспериментально-статистического моделирования выявлены оптимальные области варьирования компонентов модифицированных мелкозернистых дисперсно-

Низина Т.А., Балыков А.С., Володин В.В., Коровкин Д.И. Дисперсно-армированные мелкозернистые бетоны с полифункциональными модифицирующими добавками // Инженерно-строительный журнал. 2017. № 4(72). С. 73–83.

армированных бетонов. Установлена эффективность модифицирования мелкозернистых бетонов полиакрилонитрильным волокном, модифицированной астраленами базальтовой микрофиброй, а также высокоактивным метакаолином. Использование данных модификаторов в рецептуре позволило получить цементные композиты с широким диапазоном прочностных характеристик: от 30 до 53 МПа – при сжатии, от 3,7 до 6,6 МПа – на растяжение при изгибе.

Introduction

The primary task in designing plans of experimental studies developed in order to obtain compositions of construction materials is an opportunity to provide multi-criteria optimization and to reveal the most reasonable concentration of binding agents, fillers and aggregates, modifying additives, etc.

An increased number of components, e.g., an increased total number of formulation and process factors of cement compositions, results in the need to overcome difficulties caused by so called curse of dimensionality [1]. Furthermore, the optimization of compositions must guarantee high number of operational and process properties of the material, including resource saving criteria. As a rule, optimum coordinates of the quality system criteria under study do not overlap. Solving these multi-criteria tasks is possible in case of integrated realization of reasonable pre-requisites and upon theoretical pre-requisites, as well as by carrying out physical and calculation experiments, and when optimizing their results [2] when the issues related to taking compromise decisions arise.

Currently, a complicated concrete composition that includes 6-7 and more components is becoming a necessary reality [3–7]. Multiple additives and modifiers into concrete (hydrophobic and hydrophilic organic surfactants – super- and hyper- plasticizers [8, 9], fine fillers [5, 10], chemical and active minerals additives of natural and technogenic origin [11–14], dispersive fibers [15–21], including with the use of carbon nanoparticles [22, 23], etc.) are a unique key to solving many process tasks. Multifunctionality and complexity of applied modifiers allows efficiently controlling the structure formation processes at various stages of concrete production [24] and producing composites having high performance characteristics [3–7, 25]: High Performance and Ultra-High Performance Concretes [26–28], High Strength and Ultra-High Strength Concretes [29], Reactive Powder Concretes [30, 31], Self-Compacting Concretes [32], etc.

This causes some complications primarily associated with the cement and additive compatibility and additives' inter-compatibility [6, 33], which is a subject of many studies and discussions at global forums. Reputable Canmet forums distinguish the task of quantitative assessment of complex additives component compatibility between each other and cement/additive compatibility as the primary task [33]. When assessing compatibility, all factors are important, epically the type and concentration of additives in the mix. In various dosages, an additive can be either cure or poison, as the great German doctor Paracelsus once referred to drugs. Increased number of concrete mix components puts the Primum Non Nocere (do no harm) principle to the foreground.

Multi-criteria optimization of compositions and properties of modified fiber fine-grained concretes is undoubtedly an urgent task which requires using mathematical modeling and analysis methods to solve it competently. We believe that the method of experimental and statistical (ES) modeling proposed by V.A. Voznesenskiy [34, 35] and being actively developed at the moment [36, 37] is the most interesting one.

The purpose of this paper was to study the efficiency of dispersive reinforcement and modifying of compositions of cement fiber fine-grained concretes (CFFGC) by active mineral and chemical additives, and to optimize the developed compositions according to the strength efficiency criteria.

Materials and Methods

The experimental study was planned on the basis of a D-optimal plan containing 15 experiments [38]. Two groups of factors varied – the type and concentration of used additives: v_1 (condensed compacted silica fume (CCSF) by Keznetskiye ferrosplavy OJSC); v_2 (white highly active metakaolin (WHAM)); v_3 (Penetron Admix (Admix) sealant for concrete mixes), as well as the type and concentration of the applied fiber: w_1 (polypropylene multi-filament fiber (PP) with the cutting length of 12 mm, diameter of 25÷35 microns, density of 0.91 g/cm^3); w_2 (specially treated polyacrylonitrile synthetic fiber FibARM Fiber WB (PAN) with the cutting length of 12 mm, diameter of 14÷31 microns, density of 1.17 ± 0.03 g/cm^3); w_3 (astralene-modified basalt microfiber Astroflex-MBM (MBM) with the length of

100÷500 microns, average diameter of 8÷10 microns, bulk density of 800 kg/m^3, astralene concentration of 0.0001 ÷ 0.01 % of the fiber weight). The variance levels of the factors under study are given in Table 1.

In designing the experimental study plan, the following conditions were fulfilled:

$$\begin{gathered} 0 \le v_i \le 1;\ \Sigma v_i = 1;\ i = 1,2,3; \\ 0 \le w_i \le 1;\ \Sigma w_i = 1;\ i = 1,2,3. \end{gathered} \tag{1}$$

In experimental studies, several series of sample prisms 40 x 40 x 160 mm were manufactured from fiber-concrete mixes whose compositions included the following modifiers (apart from those mentioned above): Portland cement grade CEM I 42.5R; fine aggregate – natural quartz sand from the Novostepanovsk pit (Smolniy settlement, Ichalovskiy region, Republic of Mordovia) with the grain size below 5 mm – 65 % of the solid phase weight; superplasticizer Melflux 1641 F – 0.5 % of the binder weight. The changes in the compression strength (Russian State Standard GOST 310.4) and tensile strength in bending (Russian State Standard GOST 310.4) were studied after aging for 28 days.

Table 1. Levels of variation of experimental research factors (% by weight of cement)

Variable factors			**Levels of variation**		
			0	0.5	1
Type of additive	v_1	CCSF	0	10	20
	v_2	WHAM	0	3	6
	v_3	Admix	0	0.75	1.5
Type of fiber	w_1	PP	0	0.5	1
	w_2	PAN	0	0.75	1.5
	w_3	MBM	0	2.5	5

Results and Discussion

Upon the experimental results, experimental and statistical models were built [38, 39] to reflect the dependencies of the studied physical and mechanical quality indicators of fiber fine-grained concretes on the type and concentration of modifiers (mix I) and dispersive fibers (mix II). The generalized ES model was defined as a reduced polynomial $M_IM_{II}Q$ "mix I, mix II – property" in the following form:

$$\begin{aligned} \hat{y} = {} & b_{12} \cdot v_1 \cdot v_2 + b_{13} \cdot v_1 \cdot v_3 + b_{23} \cdot v_2 \cdot v_3 + d_{12} \cdot w_1 \cdot w_2 + d_{13} \cdot w_1 \cdot w_3 + \\ & + d_{23} \cdot w_2 \cdot w_3 + k_{11} \cdot v_1 \cdot w_1 + k_{21} \cdot v_2 \cdot w_1 + k_{31} \cdot v_3 \cdot w_1 + k_{12} \cdot v_1 \cdot w_2 + \\ & + k_{22} \cdot v_2 \cdot w_2 + k_{32} \cdot v_3 \cdot w_2 + k_{13} \cdot v_1 \cdot w_3 + k_{23} \cdot v_2 \cdot w_3 + k_{33} \cdot v_3 \cdot w_3. \end{aligned} \tag{2}$$

Two types of models "mix I (modifiers) – property" $(\hat{y}(v_1, v_2, v_3)$; M_IQ) and "mix II (dispersive fibers) – properties" $(\hat{y}(w_1, w_2, w_3)$; $M_{II}Q$) were distinguished from the $M_IM_{II}Q$ model with recording the respective group of composition factors [7]. For each type of the models and each physical and mechanical characteristics under study, seven triangle Gibbs-Rosebom diagrams were built in the form of 2D maps of level lines by using Statistica 10.0.1011 software.

To further analyze the impact of modifiers on the properties of cement composites, a generalizing indicators was introduced – a numerical characteristic of the property field in the form of an absolute values of the studied indicator corresponding to its maximum $\hat{y}_{\max}$. ES-models "mix I – property maximum" ($\hat{y}_{\max}(v_1, v_2, v_3)$; $M_IQ_{\max}$) and "mix II – property maximum" $(\hat{y}_{\max}(w_1, w_2, w_3)$; $M_{II}Q_{\max}$) reflecting the connection between the varied factors and the maximums of the properties under study represent polynomial equations of the following form [37, 40]:

$$\hat{y}_{\max} = b_1 \cdot v_1 + b_2 \cdot v_2 + b_3 \cdot v_3 + d_{12} \cdot v_1 \cdot v_2 + d_{13} \cdot v_1 \cdot v_3 + d_{23} \cdot v_2 \cdot v_3 + k_{123} \cdot v_1 \cdot v_2 \cdot v_3; \tag{3}$$

$$\hat{y}_{\max} = b_1 \cdot w_1 + b_2 \cdot w_2 + b_3 \cdot w_3 + d_{12} \cdot w_1 \cdot w_2 + d_{13} \cdot w_1 \cdot w_3 + d_{23} \cdot w_2 \cdot w_3 + k_{123} \cdot w_1 \cdot w_2 \cdot w_3. \quad (4)$$

By using the data obtained, for each of the studied characteristic, two triangle Gibbs-Rosebome diagrams were built to reflect respective systems $\hat{y}_{\max}(v_1, v_2, v_3)$ and $\hat{y}_{\max}(w_1, w_2, w_3)$ [37, 40]. For each of the analyzed parameters, the pairs of models $\hat{y}_{\max}(v_1, v_2, v_3)$ and $\hat{y}(w_1, w_2, w_3)$ as well as $\hat{y}_{\max}(w_1, w_2, w_3)$ and $\hat{y}(v_1, v_2, v_3)$ are then synthesized. Secondary models $\hat{y}_{\max(v)}(w)$ and $\hat{y}_{\max(w)}(v)$ were formed representing a triangle (Figs. 1, 2) sliding along the bearing triangle and fixed in seven centroid points (3 corners + 3 side centers + center of gravity) [37, 39, 40].

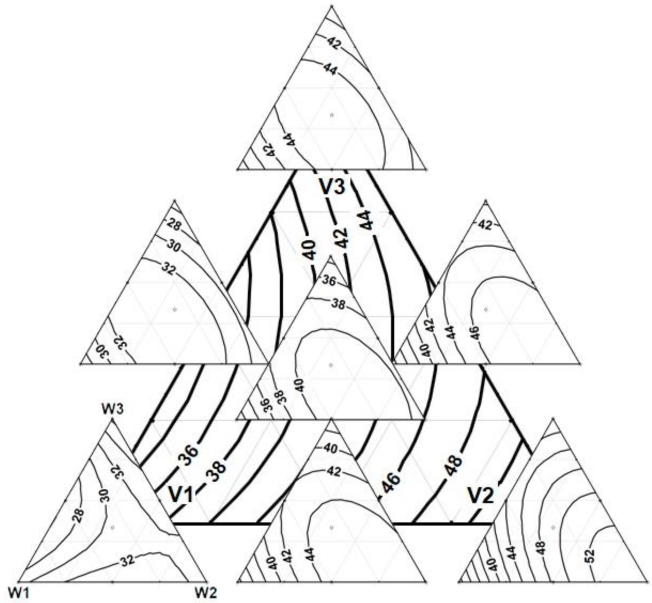


Figure 1. Diagrams "dispersive fibers – property" and contours of maximum values of the limit of compression strength of cement fiber fine-grained concretes (CFFGC) on triangle "modifying additives – property" [39, 40]

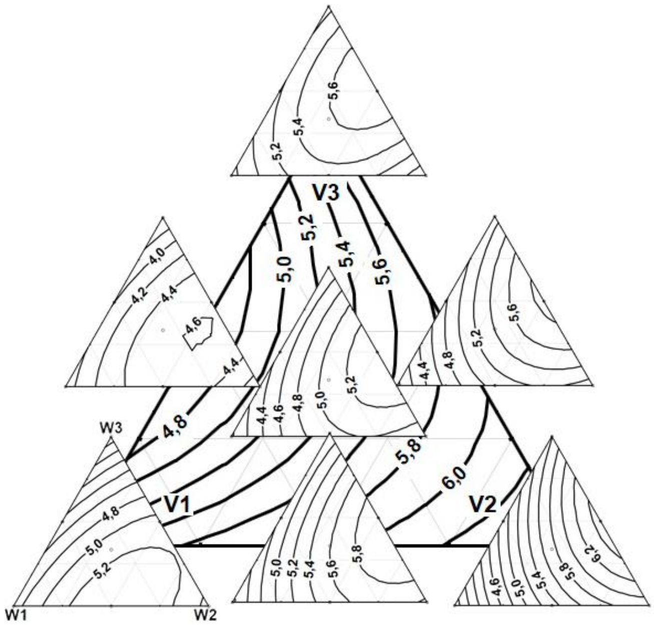
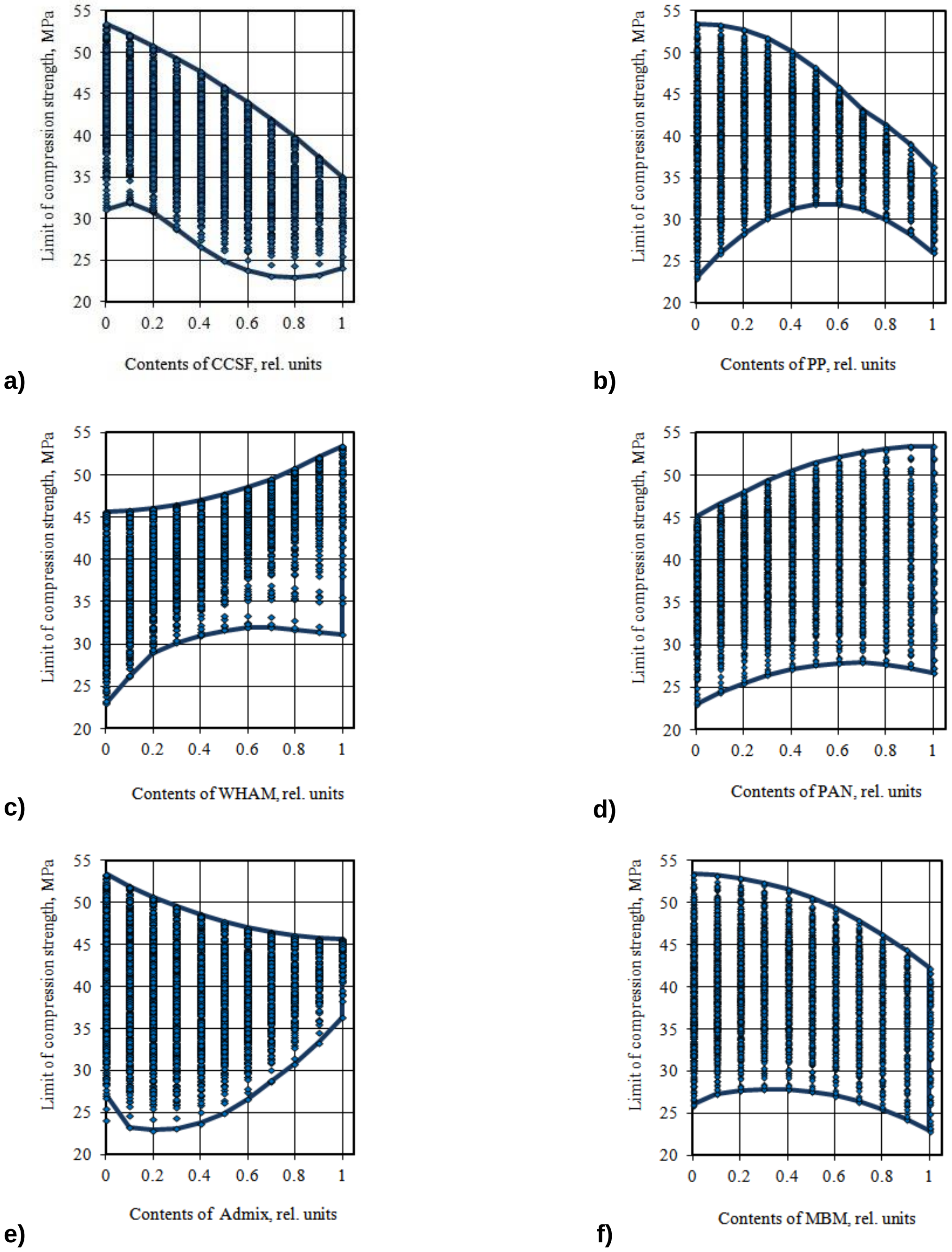


Figure 2. Diagrams "dispersive fibers – property" and contours of maximum values of the limit of tensile strength in bending of cement fiber fine-grained concretes (CFFGC) on triangle "modifying additives – property" [40]

The resulting secondary models $\hat{y}_{\max(v)}(w)$ and $\hat{y}_{\max(w)}(v)$ are highly informative multi-factor experimental and statistical models comparable to well-known analogues that are widely represented in the work [38]. These ES-models enable establishing a link and quantitative relations between investigated strength characteristics of the cement composites, process and operational factors with simultaneous minimization of labor costs and getting maximum information concerning the studied object.



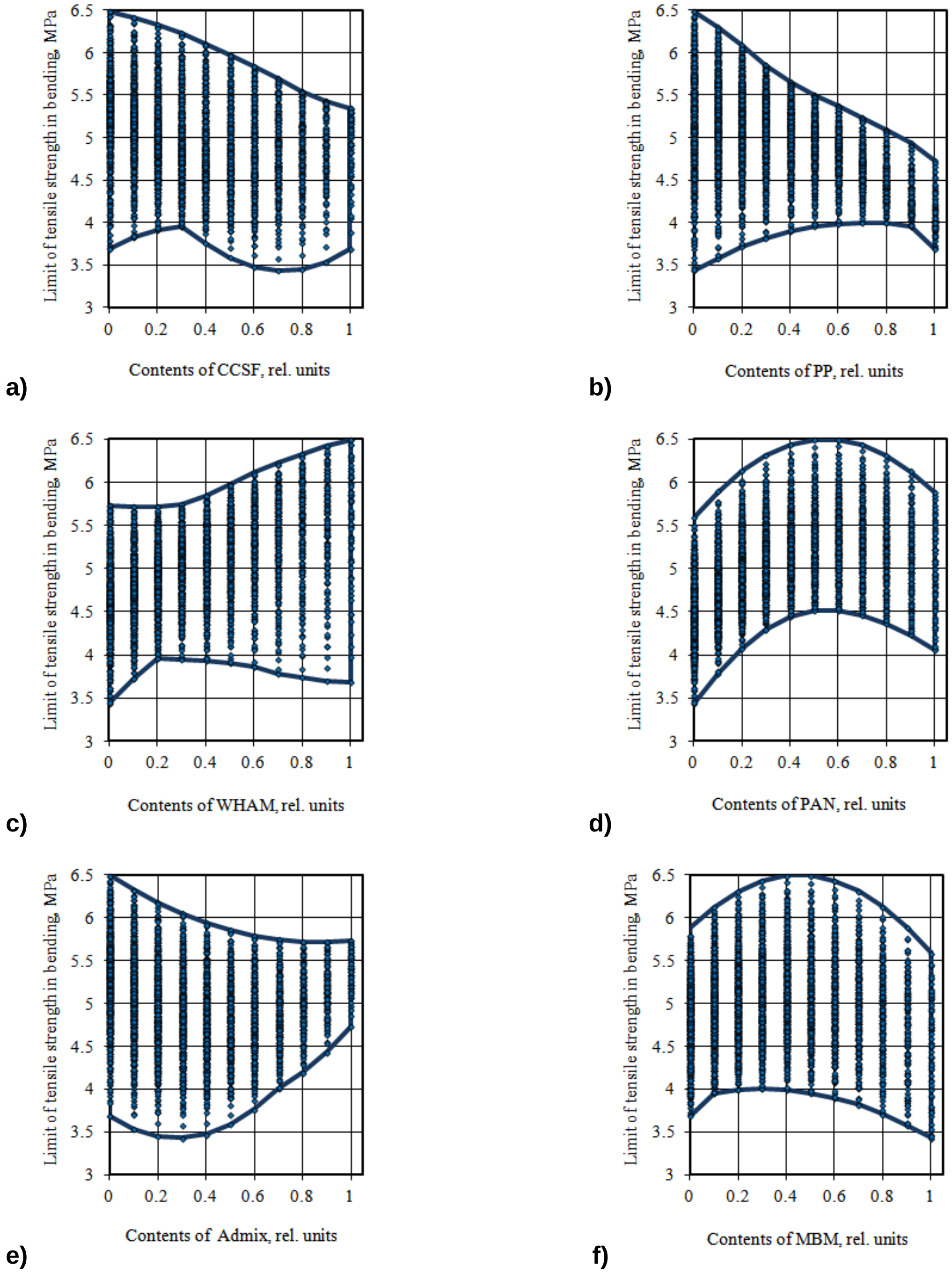
a) b) c) d) e) f)

Figure 3. Field of allowable values of the limit of compression strength of cement fiber fine-grained concretes (CFFGC) based on content of modifying additives (a, c, e) and dispersive fibers (b, d, f) in compositions

At the final stage of experimental studies, the compositions of modified fiber fine-grained concretes were optimized. By using an ES-model as a polynomial equation (2), for each of the six formulation modifiers, generalizing values of the characteristics under study were obtained – the areas of permitted solutions describing the technology stability (Figs. 3, 4). These areas are confined by minimal and maximum possible values of controlled properties depending on the concentration of the modifier analyzed.

Figures 3 and 4 represent the areas of permitted solutions for the studied mechanical indicators of cement composites – compression strength and tensile strength in bending of fiber fine-grained concretes. It has been found that the increased share of metakaolin in the total mass of active mineral additives causes the compression strength (Fig. 3, c) and the tensile strength in bending (Fig. 4, c) of cement composites to icnrease. For its maximum concentration in composites (6 % of the Portland cement weight), we can produce fiber concretes with a wide range of strength characteristics: 30 to 53 MPa for compression strength and 3.7 to 6.6 MPa for tensile strength in bending.

Introducing condensed compacted silica fume into concrete mixes causes the strength of cement composites to decrease, which shows the CCSF negative impact on the structure formation processes of cement composites as compared to other types of applied additives (Figs. 3, 4, a).



a) b) c) d) e) f)

Figure 4. Field of allowable values of the limit of tensile strength in bending of cement fiber fine-grained concretes (CFFGC) based on content of modifying additives (a, c, e) and dispersive fibers (b, d, f) in compositions

Mounted according to the study results the WHAM efficiency as compared to CCSF is confirmed by data of the work [13]. This is explained by: higher (about 2–2.5 times) pozzolanic activity of metakaolin; different chemical nature of additives (silicate in CCSF and aluminosilicate in WHAM); accelerated reaction between WHAM and lime as compared to CCSF, which ensures its effective binding during the first day of setting; higher plasticity and performance of concrete and mortar mixes, no surface adhesiveness of concrete with WHAM typical of concretes with CCSF; lower water demand of mixes with WHAM, meaning lower consumption of superplasticizers needed to achieve the same mobility of concrete mixes.

When increasing the concentration by means of Admix mineral modifier, the maximum values of strength are somewhat decreased, and the minimum possible values are increased (Figs. 3, 4, e); the area of permitted values is substantially reduced for the maximum filling of this additive (1.5 % of the Portland cement mass) – 36 to 46 MPa for compression strength and 4.7 to 5.7 MPa to tensile strength in bending.

This effect can be explained by newly formed calcium hydrosulfoaluminates and hydrocarboaluminates when Penetron Admix components interact with cement hydration products. These new formations appearing when the volume grows, along with the initial thickening of the structure, may cause negative internal stresses in case of non-optimal use. This is confirmed by the study results of other authors, in particular [14]. Thus, taking into account that it is necessary to accurately select the dosage of this mineral additive in order to rationally control the crystallization process and form the structure of cement composites.

By analyzing the areas of permitted solutions when studying the effect of dispersive fibers on the tensile strength in bending, we can make a conclusion on the efficiency of reinforcing fine-grained concretes with PAN-fibers and MBM-fibers (Figs. 4, d, f), with the highest strength of 6.5 MPa obtained when using a complex of PAN+MBM fibers with equal (50 %) shares of these fibers. This proves the appropriateness and efficiency of the multi-level reinforcement of fine-grained concretes by using carbon nanostructures (applying polyacrylonitrile fiber is reinforcement on the macroscale structural level; applying astralene-modified basalt microfiber is reinforcement on the upper macroscale level).

Presented in [17–23] the study results confirm the efficiency of the multi-level reinforcement (including the use of carbon nanoparticles), this is based on the hypothesis of the proportionality of the reinforcing elements to the "blocked" cracks of the corresponding level of structure (micro-, meso-, macro-).

Increasing the percentage of polypropylene fiber when reinforcing composites results in decreased maximum possible strength (Figs. 3, 4, b), with the area of permitted solutions of this indicators decreased – 3.6 ÷ 4.8 MPa for tensile strength in bending and 26÷36 MPa for compression strength.

Conclusions

To produce materials of various functional purpose with a wide range of properties, a systemic approach is needed to be applied when selecting initial materials, composite production technologies, planning and analyzing methods for experimental studies. An important role is played by informative multi-factor experimental and statistical models that enable establishing a link and quantitative relations between material quality indicators, its structural parameters formulation, process and operational factors with simultaneous minimization of labor costs and getting maximum information concerning the studied object.

As result of experimental studies:

1. The efficiency of modifiers and dispersive fibers was assessed for a number of physical and mechanical properties (the limit of compression strength (Russian State Standard GOST 310.4) and the limit of tensile strength in bending (GOST 310.4) after aging for 28 days) in order to produce concretes of various functional purpose.
2. Secondary models $\hat{y}_{\max(v)}(w)$ and $\hat{y}_{\max(w)}(v)$ were formed representing a triangle (Figs. 1, 2) sliding along the bearing triangle and fixed in seven centroid points (3 corners + 3 side centers + center of gravity).
3. Thanks to optimization of compositions of fiber fine-grained concretes, the areas of permitted solutions were defined, reflecting the possible range of changes in the quality indicator under study depending on the formulation and the percentage of each of the applied modifiers.

4. By analyzing the areas of permitted solutions when studying the effect of modifying additives on the physical and mechanical properties, we can make a conclusion on the efficiency of modifying fine-grained concretes with white highly active metakaolin (WHAM) (Figs. 3, 4, c). For its maximum concentration in composites (6 % of the Portland cement weight), we can produce fiber concretes with a wide range of strength characteristics: 30 to 53 MPa for compression strength and 3.7 to 6.6 MPa for tensile strength in bending.

5. By analyzing the areas of permitted solutions when studying the effect of dispersive fibers on the tensile strength in bending, we can make a conclusion on the efficiency of reinforcing fine-grained concretes with PAN-fibers and MBM-fibers (Figs. 4, d, f), with the highest strength of 6.5 MPa obtained when using a complex of PAN+MBM fibers with equal (50 %) shares of these fibers.

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