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Research article

Mechanical properties and brittleness of concrete made by combined fly ash, silica fume and nanosilica with ordinary Portland cement

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Abstract: This paper introduced a new concrete composites made by quaternary binder by partially replacing ordinary Portland cement (OPC) with different percentages of supplementary cementitious materials (SCMs). The motivation is to reduce our dependency on OPC to reduce CO_2 emission and carbon foot print. As the main substitute for the OPC, siliceous fly ash was used (FA). Moreover, silica fume (SF) and nanosilica (nS) were also used. This study utilized the following contents of SCMs used: 5% of nS; 10% of SF; 0, 15, and 25% of FA. During examinations the main mechanical properties of concrete composites, i.e. compressive strength (f_{cm}) and splitting tensile strength (f_{ctm}) were assed. The brittleness of these materials was also analysed. Based on the conducted studies, it was found that concrete composite based on quaternary blended cements, of series Mix3, has shown the best results in terms of good strength parameters, whereas the worst mechanical parameters were characterized by concrete of series Mix4. On the other hand, concrete including only SF and nS (Mix2 series) were characterized by the greatest brittleness. It was observed that f_{cm} of concrete composites for series Mix2, Mix3, and Mix4 increase of 41%, 48%, and 31% respectively compared with the concrete without additives, i.e. series Mix1. In addition, f_{ctm} also increase of 39%, 47%, and 30%, respectively, for the three series mentioned above, compared with the control concrete. Concrete of series Mix3, with high mechanical properties and demonstrating the features of quasi-plastic material, i.e. having lower brittleness, can be used in concrete and reinforced concrete structures subjected mainly to dynamic and cyclic loads. Therefore, it can be used, in the construction of foundation structures for machines and other types of structures in which the above-mentioned loads are dominant.

Keywords: siliceous fly ash (FA); silica fume (SF); nanosilica (nS); quaternary binder concrete; mechanical properties; brittleness; sustainable construction

1. Introduction

Nowadays, concrete is undoubtedly the most used construction material in the world. In addition, the construction of buildings and structures made of concrete, as well as the subsequent maintenance and renovation of concrete structures, catalyze the development of world economies, thus significantly contribute to their economic progress and the increase of gross domestic products. Therefore, from the economic and social point of view, the dynamics of the development of the concrete industry is still highly desirable [1–3].

However, when looking at the production process of this very useful composite, it should be realised that its production—despite the fact that it provides safe living conditions for billions of people around the world and has a significant impact on the well-being of many global economies—is unfortunately against the principles of sustainable development, generating definitely negative effects on the natural environment.

The impact of the lack of ecological production of this construction material concerns mainly the cement matrix of the concrete composite [4,5]. Ordinary Portland cement (OPC), which is the basic binder for the production of concrete, is formed as a result of burning Portland clinker, which generates significant amounts of harmful greenhouse gases, mainly CO_2, during this process. It is estimated that approx. 7 to 9% of the total annual CO_2 emissions in the world is related to the production of OPC and, interestingly, in terms of the global emission of this harmful oxide, this places OPC production processes above aviation fuel (2.5%) and only slightly lower than agriculture (12%) [6–8]. If you add to this the fact that the cement production process consumes significant amounts of energy, both thermal and electrical [9], then after summing up all the above aspects, concrete in its natural form becomes a definitely non-ecological material [10–12].

Therefore, in order to reduce the negative environmental impact of the only OPC-based concrete production, measures have been taken to reduce the share of pure Portland clinker in the composition of cements by replacing it with other mineral components in the form of additives [13], and recently also nanoadditives [14,15]. More and more often, modern construction concretes are based on multi-component cements, with a more or less diversified composition, containing one or more substitutes for cement binder [16,17]. Such materials are referred to as Supplementary Cementitious Materials (SCMs) [18,19].

The use of multi-component cements containing SCMs allows to improve the efficiency of OPC production related to the possibility of using large amounts of mineral additives and nanoadditives, including often problematic or even harmful waste, and meets the guidelines of sustainable development [20]. It should also be emphasised that increasing the share of SCMs in OPC contributes to a significant reduction of CO_2 emissions and energy consumption during the production process.

For these reasons, the use of multi-component cements with a diversified composition is justified from an economical and ecological point of view. In addition, the synergistic effect of the interaction of several mineral additives has a more favorable effect on the properties of multi-component cements compared to cements containing only one mineral additive [21]. This allows, among others, for preparing concrete composites resistant to significant static as well as impact [22], dynamic [23–25] and fatigue loads [26–28]. Therefore, on an increasingly larger scale, laboratory tests are carried out and then implementation works on the use of multicomponent cements [29,30] in concrete technology are realized [31,32].

The main SCMs, used in composites of this type, are fly ash (FA)—both low volume FA and high volume FA, silica fume (SF) [33]. Other frequently used SCMs are also: ground-granulated blast-furnace slag, waste glass, limestone powder and crumb rubber. More and more often, matrices of such concretes also contain nanomaterials, most often in the form of nanosilica, nanocellulose or nanotube [34,35]. In addition, the possibilities of nanotechnology are also used in the field of production of active nanomaterials that are able to purposefully modify the structure of concrete composites, e.g. as a result of the use of nanoseeds of the C–S–H phase affecting the reduction of the unfavorable, brittle CH phase in concrete in order to create a larger amount of dense and permanent phase C–S–H. Unfortunately, a significant factor inhibiting, so far, the development of concrete production based on multi-component cements is the lack of practical experience related to the use of binders of this type in concrete technology [29,30].

Therefore, this article proposes a solution involving modification of the concrete material with the main SCMs, i.e. FA and the two types of silica-based additives. For this purpose, the composition of the binder in the composites by non-compacted silica fume (SF), and nanosilica (nS) were modified [36].

In addition, with regard to the principles of sustainable construction, the proposed solution allows for:

- Effective reduction of CO_2 emission [37],
- Significant reduction in the carbon footprint created in the production of OPC for ordinary concrete [37].

In fact, concretes made with applying the quaternary binder were tested for evaluation of their basic mechanical parameters as well as brittleness.

The conclusions resulting from the research undertaken—in terms of the synergy of the impact of SCMs on the main mechanical parameters as well as brittleness in new cementitious composites with a diversified binder composition—may contribute in the future to a more conscious use of such materials in composite structures. Undoubtedly, this will positively affect the reduction of CO_2 emissions into the atmosphere, which will be a significant step towards the further development of sustainable construction.

2. Materials and methods

2.1. Materials

2.1.1. Parameters of raw materials

The constituting materials of concrete used in the present study are: OPC (CEM I 32.5R), natural gravel as coarse aggregates (with specific gravity 2.65 and aggregate size 2.0–8.0 mm) and natural sand as fine aggregates (specific gravity of 2.60 and maximum size of 2.0 mm). The specific gravity of both aggregates (in oven-dried particle conditions) was measured with using pyknometer method based on the European Standard EN-1097-6:2013 [38].

The SCMs, which were used as partial replacement of OPC to produce quaternary mixes, are FA, SF and nS and the relevant properties of these materials were determined in the laboratory as per relevant codes of practice. In addition, superplasticizer (SP) STACHEMENT 2750 based on polycarboxylates (1.8% of binding material weight) was used in order to improve the flowability of the concrete. The laboratory pipeline water for preparation all mixtures was also used.

The chemical composition and physical properties of the cement and SCMs are listed in Tables 1 and 2, respectively.

Table 1. Chemical composition of the OPC and SCMs used (mass%).

Material/constituent	SiO_2	Al_2O_3	CaO	MgO	SO_3	Fe_2O_3	K_2O	P_2O_5	TiO_2	Ag_2O
OPC	15.00	2.78	71.06	1.38	4.56	2.72	1.21	-	-	-
Class F FA	55.27	26.72	2.35	0.81	0.47	6.66	3.01	1.92	1.89	0.10
Non-condensed SF	91.90	0.71	0.31	1.14	0.45	2.54	1.53	0.63	0.01	0.07
Konasil K-200 nS	>99.8	-	-	-	-	-	-	-	-	-

Table 2. Properties of binders used.

Material/parameter	Specific gravity (g/cm^3)	Blaine's fineness (m^2/g)	Particle diameter (μm)
OPC	3.11	0.33	40
Class F FA	2.14	0.35	30
Non-condensed SF	2.21	1.40	11
Konasil K-200 nS	1.10	200	0.012

2.1.2. Mix proportions

It should also be noted that, based on previous studies, the effect of the modification of concrete composites FA in the amount of 20 and 30% of cement weight was recognized quite well. On the other hand, it is also known that the most beneficial effects are brought by modification of the concrete structure SF in the amount of 10% [39], while nS 5%, e.g. [40].

For above reasons, and that it was possible to draw additional conclusions regarding the influence of the main SCMs, i.e. FA, on the analysed parameters it was assumed that the content of FA in each of the three concretes based on quaternary blended cements was different (0%, 5% and 15%). Moreover, in all series of concrete, the constant amount of the binder substitute at 10% and 5% was for SF and nS respectively.

Based on the above the mix proportions for all mixes are given in Table 3.

Table 3. Mix proportions used in the studies (kg/m^3).

Mix	Mix No.	OPC	FA	SF	nS	Water	SP	Sand	Gravel
100% OPC	Mix1	352	0	0	0	141	0	676	1205
85%OPC+0%FA+10%SF+5%nS	Mix2	299.2	0	35.2	17.6	141	6	676	1205
80%OPC+5%FA+10%SF+5%nS	Mix3	281.6	17.6	35.2	17.6	141	6	676	1205
70%OPC+15%FA+10%SF+5%nS	Mix4	246.4	52.8	35.2	17.6	141	6	676	1205

2.1.3. Mixing procedure

The full mixing process along with duration of each mixing stage is shown in Figure 1. Then, the fresh mixture was poured into the molds and vibrated on the vibrating table. The concrete cubes were cast for compressive strength and splitting tensile strength testing.

After finishing, the specimens were covered with wet fabric and stored in casting room at 20 ± 2 °C. In the next step, specimens were demolded after 48 h and kept in a water tank for the first 14 d. For the next 2 weeks, the specimens were cured in a laboratory conditions and then examined after 28 d of their preparation.

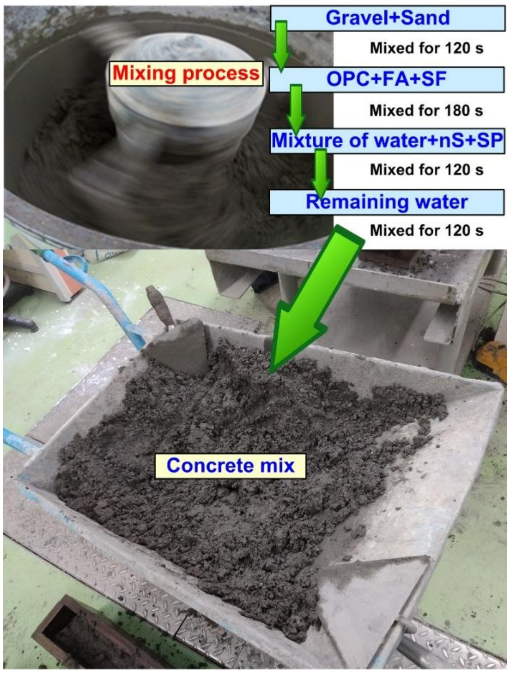


Figure 1. Full mixing process of concrete made by combined SCMs with OPC.

2.2. Methods

2.2.1. Mechanical property tests

Mechanical property tests were carried out according to the European Standards EN 12390-3:2011+AC:2012 [41] and EN 12390-6:2009 [42]. Compression strength—f_{cm} and splitting tensile strength—f_{ctm} were investigated during the studies. In order to ensure the repeatability of test results, six specimens for all composites and both mechanical tests were prepared and reported after 28 d of curing.

Cube specimens (150 mm × 150 mm × 150 mm) were used for both type of tests, which were conducted in a hydraulic servo testing machine with a maximum bearing capability of 3,000 kN. During the experiments the specimens were loaded statically. The view of cubes during the tests is shown in Figure 2.

Figure 2. Specimens during the compressive strength (a) and splitting tensile strength (b) tests.

2.2.2. Brittleness

Modification of concrete by mineral additives and nanoadditives also affects the brittleness of composites with a cement matrix. Moreover, this property, similarly as the susceptibility to fracture toughness (due to internal primary structure defects like pores, voids and initial cracks etc.) [43–45], provides important information about the material in terms of its durability [46].

Therefore, in addition to a detailed analysis of the strength parameters, the presented tests investigated changes in the brittleness of the concrete being the subject of the experiments. For this purpose, one of the available methods was used, i.e. tension-compression strength ratio. Thus, the brittleness indices were calculated based on the results of basic strength tests, i.e. f_{cm} and f_{ctm}. According to [47], it is an effective method to estimate this parameter in the case of cured concretes, but unsuitable for concretes analyzed at an early ages.

Furthermore, the method of determining the brittleness of concretes based on the analysis of the Q index, developed by Jeng and Shah [48] and presented in [47], requires knowledge of the modulus of elasticity of the analyzed composites and their parameters of fracture toughness—in both linear and non-linear terms [48]. For this purpose, additional complex studies would have to be carried out.

In addition, it should be stated that the method based on the analysis of proportions of concrete strength parameters is also beneficial due to the fact that the strengths of compressed and tensile strength of concrete samples are closely related. However, this relationship is not proportional.

As f_{cm} increases, f_{ctm} also increases, but the rate of this increase is decreasing. The ratio of the two strengths depends on many factors, such as the composition of the concrete mix, the age of the concrete, the shape of the samples, the method of making and curing the materials, and the methods of testing tensile strength. These dependencies are described in detail in their works, e.g. Raphael [49] and Oluokun [50]. They recognized that the mutual relations of the results of f_{cm} and f_{ctm} have a close relationship for a given material. Therefore, it can be written with mathematical relations [49,50].

Considering the above, in own research the brittleness index (BI), in percents for particular composites was determined based on the Eq 1:

$$BI = \frac{f_{ctm}}{f_{cm}} \cdot 100\%. \tag{1}$$

3. Results and discussion

3.1. Mechanical properties

The results of tests of basic strength parameters of concretes with a variable structure of the cement matrix are shown in Figure 3. It shows that the proposed material modification resulted in a very clear improvement in both f_{cm} and f_{ctm} for all concretes including SCMs. Additionally, the upward trends for both analyzed parameters were strictly consistent with each other between the individual materials.

The highest compressive and tensile strength was obtained for concrete containing the addition of 3 different SCMs in the composition of the cement matrix. The increases in both strength parameters in the case of Mix3 were almost 50% higher when compared to the values obtained for the reference concrete, i.e. Mix1 (48% and 47% for f_{cm} and f_{ctm}, respectively). Slightly lower, although still quite high, increase in the value of strength parameters was recorded for concrete with silica-based additives only, i.e. Mix2. For this composite, both parameters increased by 40%. The significantly weakest effect of material strengthening was observed in the Mix4 containing the greater amount of the FA modifier, i.e. 15%. For this material, increases of f_{cm} and f_{ctm} by only 30% were visible. Nevertheless, these results also appear to be very advantageous in comparison to the values obtained for concrete based only on OPC (Figure 3).

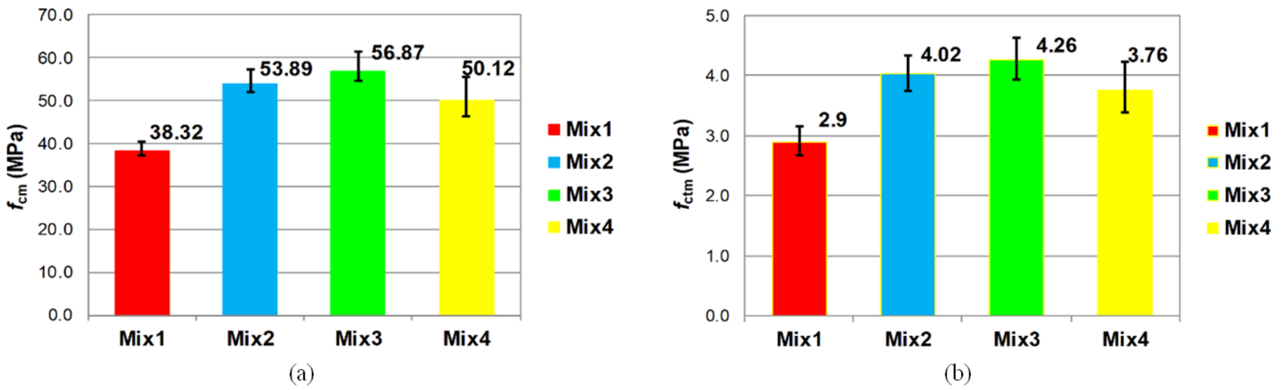


Figure 3. Effect of modification of the cement matrix on compressive strength (a) and splitting tensile strength (b) of concretes containing SCMs.

In addition, when analysing the obtained results of strength parameters, it should also be noted that a more complex modification of the structure of the cement matrix results in a smaller convergence in the obtained results; to a greater extent on f_{ctm}. The graphs in Figure 3 show larger dispersions of the obtained results in the case of quaternary binder composites, i.e. from Mix2 to Mix4, compared to the values obtained for the reference concrete, i.e. Mix1. Based on the size of error bars, placed on individual graphs, it can also be observed that the largest dispersion of results were for concrete of series Mix4, i.e. with 3 SCMs in its composition and a higher content of FA.

The FA additive, used in small amounts, is able to additionally produce a larger amount of more compact C–S–H phase, which makes the material more airtight by filling the pores [51,52]. However, the presence of FA in the composition of the cement matrix implies a slight reduction in the strength parameters of the composite through heterogenization of its structure and reduced pozzolanic activity in the initial curing period of materials with these additives [53,54]. As a consequence, the smallest effect of improving the composite strength parameters and the increase of heterogeneity in the obtained results were observed in the concrete of series Mix4 (Figure 3). The effect appeared despite the presence of two other more active SCMs in the concrete composition—SF and nS.

Mix2, which contained only two SCMs, showed a much higher value of both compressive and tensile strength compared to Mix4. Nevertheless, the results for this material were clearly lower than the values obtained for the matrix-based composite composed of 80%OPC+5%FA+10%SF+5%nS (Figure 3). Therefore, it can be concluded that supplementing the composition of the cement binder with three pozzolanic active additives (one of which is FA in the amount of several percent) causes the occurrence of a strong synergy between all components in the material structure, which clearly increases the material strength parameters (Figure 3). This phenomenon is confirmed by the results of tests on the microstructure of composites of this type presented in papers [55,56].

3.2. Brittleness

Figure 4 summarizes the calculated brittleness indexes for all analyzed composites with error bars. Due to slight differences between the results in particular series of concrete, the obtained values were rounded to 3 decimal places. Moreover, it should be noted that the obtained brittleness results are quite convergent, i.e. characterized by low levels of error bars (Figure 4). This proves the small dispersion of the obtained values of strength parameters (Figure 3). Thanks to this, the results can be considered representative and the conclusions resulting from the conducted research are significant.

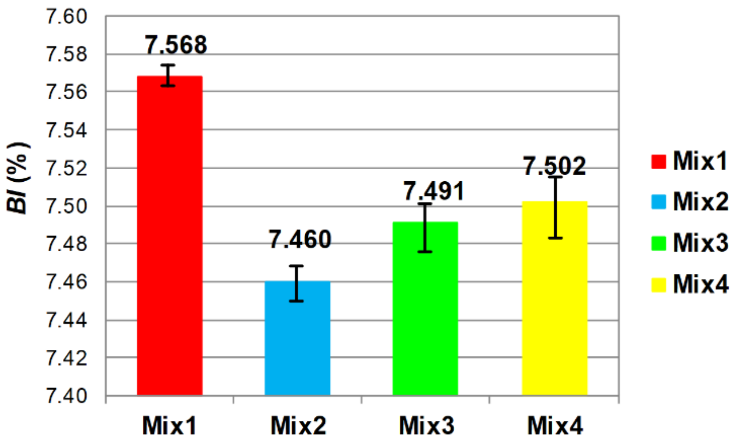


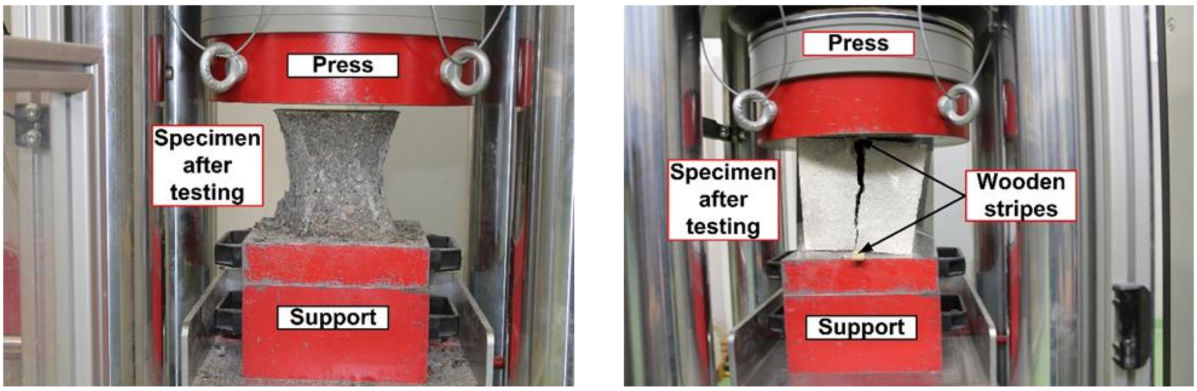
Figure 4. Brittleness index (*BI*) of analyzed composites.

When analyzing the data in Figure 4, it can be seen that concrete of series Mix2 was characterized by the highest brittleness due to the use of highly active pozzolanic additives—SF and nS. On the other hand, the substitution of OPC by FA results in a gradual decrease in the brittleness of the material, which also changed the behaviour of these composites in the destruction process during investigations of mechanical parameters (Figure 5). This is emphasized in the next part of this subsection.

Reactive FA grains are characterized by a different stiffness and a higher modulus of elasticity than the cement matrix [57]. In addition to this, they positively change the structure and porosity of Interfacial Transition Zone (ITZ) in modified concretes [58]. On the other hand, FA grains, due to their lower specific surface area and reactivity compared to silica materials, do not react as quickly as SF, especially nS. As a result, some of the FA grains in the structure of such concretes appear in the first months of curing as micro-aggregates more or less related to the matrix structure. Some of the grains are well integrated with the leaven while there are also grains with microcracks in the ITZ area [59]. In such composites, spots of FA grain separation are also visible [21,22,54]. All these factors mean that composites with FA additive are characterized by lower brittleness than ordinary concretes or concretes modified with other materials such as SF and nS. Such a phenomenon was observed in tests of ordinary concretes containing FA and other SCMs [60] as well as special concretes, e.g. roller compacted concretes [61] and high-performance concretes [62].

However, it should be noted that in the case of the analyzed composites, although the brittleness of Mix3 and Mix4 was lower than in the case of Mix2, it is still higher than in the control concrete, i.e. Mix1. It was undoubtedly the effect of the participation in the matrix composition of these SF and nS composites. In the case of concretes modified only with the FA additive, a significant reduction of the brittleness level is observed usually with an increase of FA in their composition of the concrete mix.

A view of exemplary specimens after the conducted strength tests is shown in Figure 5. The destruction of most of the specimens took place in a typical way for this type of tests. On the other hand, the differences clearly identified the level of brittleness of individual mixtures. In most cases, they were consistent with the results presented in Figure 4.



(a) (b)

Figure 5. A view of specimens after conducted tests compressive strength (a) and splitting tensile strength (b).

In the case of the most brittle concretes, i.e. Mix2 and partly Mix3, the specimens failure, both in compression and in tension, occurred rapidly. During compression tests, the side portions of the cubes completely detached from the core of the specimens, leaving a distinct cone. However, during splitting tensile tests, a strong bang was audible and the specimens clearly cracked in their half.

On the other hand, in less brittle concretes, i.e. Mix1 and Mix4, the compression cone was less pronounced and the failure of specimens less spectacular (Figure 5). In addition, the tensile failure effect in these composites was clearly less audible. It can be clearly stated that the visible destructive schemes of investigated specimens fully confirm the BI results obtained on the basis of the calculations (Figure 4).

4. Conclusions

In this paper, the effect of using SCMs with diversified composition as a partial replacement of OPC on the main strength properties and brittleness of plain concrete was investigated. In the course of the experiments, the composition of concrete binder was modified with three different materials, including two mineral additives, i.e. FA and SF and a nanoadditive in the form of nS.

Based on the results obtained from the presented studies, it was possible to draw conclusions regarding the impact of individual SCMs compositions on the obtained measurements of analyzed parameters and the possibility of using some of the tested materials in specialized concrete and reinforced concrete structures.

Therefore, the main conclusions from the presented studies are as follows:

(1) The substitution of OPC with the FA+SF+nS combination causes a clear change of mechanical parameters and brittleness in quaternary binder concrete.

(2) Modification of the binder composition with three pozzolanic active materials resulted in an increase in the analysed mechanical parameters for each of the combinations compared to the results obtained for the control concrete (Mix 1) by approx. 40% for Mix2, approx. 50% for Mix3 and approx. 30% for Mix4 (Figure 3).

(3) Concrete including the total addition of siliceous materials without FA (Mix2 series) are characterized by the greatest brittleness (Figure 4).

(4) Supplementing the composition of the binder with SF and nS with the 5% FA additive causes an increase in all mechanical parameters by approx. 10%. Such action causes a slight change in the behaviour of the material in the process of its destruction from clearly brittle to quasi-plastic (Figure 4).

(5) An increase in the FA content in the concrete mix by another 10% causes a significant decrease in the strength parameters by 10% compared to concrete with the addition of silica modifiers only (Figure 3). In addition, concrete of series Mix4 is clearly less brittle (Figure 4). In general, as content of FA rises throughout each of quaternary binder series, material becomes more ductile and shows less brittle failure.

(6) Concrete of series Mix3, with high mechanical properties and demonstrating the features of quasi-plastic material, i.e. having lower brittleness, can be used in concrete and reinforced concrete structures subjected mainly to dynamic and cyclic loads. Therefore, concretes with the binder proportions 80%OPC+10SF+5%nS+5%FA can be used, for example, in the construction of foundation structures for machines and other types of structures in which the above-mentioned loads are dominant.

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Conflict of interest

The author declare no conflict of interest.

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