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Research Article

Long-Term Properties of Cement-Based Composites Incorporating Natural Zeolite as a Feature of Progressive Building Material

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This study is aimed at the utilization of natural zeolite as a prospective type of Supplementary Cementitious Material in connection with the innovations of construction solutions through the materials. The influence of zeolite on several properties of cement-based composites is studied. Basic characteristics of input powdery materials as well as the technological parameters of zeolite tested on mortar containing 50% of cement replacement are presented. The technical parameters of concrete containing 8, 13, and 25% of cement replacement by zeolite are presented as well. The paper is valuable due to the three-year testing period. The density of the hardened concrete was found to be decreasing with increasing zeolite content. While no significant differences in compressive strength were found after 28 days, differences between individual samples were clear after the long-term period. The presence of zeolite influenced the compressive strength positively in the case of the 8% and 13% replacement, while the higher proportion of natural zeolite (25%) caused lower compressive strength after 28 days but was similar to the reference concrete after three years. A major increase in strength was detected for all samples in the long-term period. It confirms the long-term potential of zeolite for strength improvement.

1. Introduction

The ordinary Portland cement is one of the most utilized binder materials for the production of building materials. However, its production has negative environmental impact due to the requirement of high amount of energy, production of greenhouse gases, and so on. Utilization of supplementary cementitious materials (SCMs) for the production of cement-based composites can help to significantly minimalize these adverse influences. Additionally, different SCMs bring several types of improvement to composites properties, for example, better workability or durability. Moreover, the costs of concrete can decrease. The incorporation of SCM is a feature of the current advanced concrete and is connected with the modern approaches to concrete production. Thus, it can be successfully involved in the modern methods of construction. Natural zeolite is one of the best-applicable SCMs, mainly due to its pozzolanic activity.

The quality and durability of concrete are strongly dependent on the kind, properties, and dosage of the basic components (cement, water, and aggregates) and the additives and admixtures. The alternative materials play an important role in current concrete technology such as SCMs. Due to the improvement of mechanical properties and durability of concrete, the trend of utilization of SCMs has increased [1–3]. SCMs improve the microstructure of concrete by minimizing its porosity and improving the resistance against the aggressive attack due to their chemical nature [2, 4–7]. In the standard EN 206 [8], three kinds of Type II additions—fly ash, blast furnace slag, and silica fume—are mentioned, and the conditions of their application to the concrete are specified.

The other kinds of addition can be used in specific cases of concrete structure exposure. Both zeolite and silica fume are recommended if a chemical attack occurs. The standard [8] defines the kind of addition in accordance with both the nature and intensity of chemical environment, but the recommended dosage is not specified in the standard. Therefore, the practical production of concrete can be uncertain. There are a number of studies investigating zeolite as cement supplement (pozzolanic additions) [9–17]. Some results of these studies are ambiguous, as shown below. There is a lack of information on the influence of specific dosage of additions on the long-term properties of the concrete and information about other relevant properties, such as length deformation and water absorption capacity.

Natural zeolites are hydrated aluminosilicates that have high amounts of reactive SiO_2 and Al_2O_3. Their reactivity is mainly attributed to their highly porous structure, which is related to the large external surface area giving interaction of zeolite with lime [11, 12], ability of ion exchange, and meta-stability, which supports the dissolution of zeolitic crystals and precipitation of hydrated calcium silicates and aluminates during interaction of OH^- ions available in the saturated lime solution [13, 14].

Researchers have found that the zeolites of different modifications act as pozzolanic additions in concretes; during cement hydration, CSH and CAH gel phases increase, which also increases the resistance of Portland cement compositions to acids and sulphate corrosion and increases its durability as well [11].

Regarding the early-age strength, the references are different. According to Yun-Sheng et al., zeolite added at 15% to the cement mix increases the early-age strength of concrete [15]. Turkish researchers carried out tests on concrete specimens containing 5, 10, 20, and 40% of zeolite. Compressive strength was measured after one, two, seven, and 28 days of curing. The results showed that the concrete specimens with zeolite additive had lower compressive strength compared to the control specimen after 24 hours of curing. The same trend was observed after two and seven days of curing. After 28 days of curing, the compressive strength of specimens containing 5% of zeolite increased by 6.8% compared to the control specimens; the compressive strength of the next specimens increased by 15.9%, 22.3%, and 4.1% for specimens containing 10%, 20%, and 40% of zeolite, respectively [16].

According to Ramezanianpour et al. [17], the addition of natural zeolite delays the strength development during the first seven days, after which the concretes containing 10% of natural zeolite provide almost similar compressive strength compared to that of the reference concretes. The strength of concrete mixtures containing 15% of natural zeolite, however, is marginally lower than that of the reference concrete. The pozzolanic activity of natural zeolite is significantly high, as most of the pozzolanic reactions occurred between seven and 28 days. The use of natural zeolite leads to considerable reductions in water permeability and capillary absorption for each of the selected water-to-cementitious material ratios. The highest improvements through the use of natural zeolite were observed in the rapid chloride permeability and electrical resistivity tests. The electrical resistivity of concrete samples containing natural zeolite was two to four times better than those of the reference samples. These improvements were more significant for concretes with higher water-to-cementitious materials ratios (w/cm). The electrical resistivity and rapid chloride penetrability of mixtures with 10 and 15% natural zeolite and w/cm of 0.50 was better than that of the reference concrete with w/cm of 0.35, indicating that using 10–15% of natural zeolite was more effective in improving permeability than reduction in w/cm from 0.50 to 0.35. The depth of carbonation increases with the use of natural zeolite. While the reduction of chloride penetration enhances the concrete resistivity against corrosion, the increased carbonation results in the opposite performance. Study of the SEM images shows significant improvements in porosity of the studied pastes through the use of natural zeolite. In addition, studying the images taken from transition zone reveals that natural zeolite enhances the structure of the transition zone favourably. The calcium hydroxide content is considerably reduced by the use of natural zeolite as an SCM, which results in the production of secondary C-S-H.

According to Vejmelková et al. [18], both the bulk and the matrix density of cement-based composites decrease with increase in the zeolite supplementary. Poon et al. [19] also state that 15% replacement of zeolite results in lower porosity, while a higher replacement level (25%) increases the porosity at all studied ages.

The drying shrinkage of products made with Portland-pozzolan cements is dependent on the hydration products and water demand of the mixtures. Although tests should be conducted to determine the drying shrinkage of Portland cement and natural pozzolans combinations, there is a scarcity of studies in this field [20]. Jana [21] investigates the effects of using 10, 20, and 30% of zeolite as an SCM. He observes that drying shrinkage of zeolite mixtures at 10 and 20% of Portland cement replacement levels is similar or slightly higher than that of the control mixture, whereas by using 30% of zeolite, the drying shrinkage is about 20% higher than the control mixture. Moreover, Kasai et al. [22] examined drying shrinkage for the mortars blended with clinoptilolite and mordenite. Both clinoptilolite and mordenite blended mortars experienced higher shrinkage than the control one.

It is reported that although natural zeolite reduces the slump of concrete, it can prevent bleeding and segregation. Regarding hardened concrete, natural zeolite increases compressive strength due to its pozzolanic property. Moreover, it enhances the durability of conventional concrete by reducing concrete permeability and, mainly, by improving resistance to alkali-aggregate reaction [23]. According to Najimi et al. [24], natural zeolite can be properly used as an SCM in normally consolidated concrete, considering the environmental protection and sustainable development. Chan and Ji [25] report that the pozzolanic reactivity of natural zeolite is between that of silica fume and fly ash.

This paper is aimed at the utilization of zeolite as a perspective kind of SCM and its influence on the long-term (up to three years) properties of concrete, since there is a lack of information about the long-term properties. Most reports are oriented towards the 28-day properties, or eventually up to one year, because of the challenges of long-term research. However, the pozzolanic additions need a longer time to develop their properties in the construction due to the slower progress of pozzolanic hydration processes. The short-term values of properties could be misleading.

Table 1: Chemical composition and particle size characteristics of binders.

Binder	SiO_2	CaO	Fe_2O_3 [% wt.]	Al_2O_3	MgO	CaO/SiO_2 [—]	$d(0.5)$ [μm]	$d(0.9)$ [μm]
OPC (CEM I 42.5 N)	20.34	64.1	2.99	2.97	9.03	3.150	26.68	67.87
Natural zeolite	78.75	3.50	1.73	11.6	1.16	0.040	20.68	91.97
Silica fume	97.00	0.70	1.50	1.10	—	0.007	0.226	8.647

Comprehensive results for zeolite, including basic characteristics (chemical composition and particle size distribution), technological parameters (mixing water demand, initial setting time, activity index, and relative linear deformation), and technical parameters of concrete incorporating zeolite (density, compressive strength, and total water absorption) are presented in the paper. For comparison, addition of silica fume is presented as well.

2. Materials and Methods

The main scope of the experiment is to observe the long-time technical parameters (density, compressive strength, and water absorption) of zeolite-based concrete and to assess the possibility of the practical utilization of natural zeolite for the production of progressive building material.

2.1. Materials Characteristics. Three types of mineral binders are used in the experiment. Ordinary Portland cement (OPC) class CEM I 42.5 N came from Turňa nad Bodvou, Slovakia, natural zeolite (NZ) came from Lehôtka pod Brehmi, Slovakia, and silica fume (SF) came from Istebné, Slovakia.

The chemical composition of ordinary Portland cement, natural zeolite, and silica fume was determined through X-ray fluorescence analysis (XRF). SPECTRO iQ II (Ametek, Germany) with silicon drift detector (SDD) with resolution of 145 eV at 10,000 pulses was used for the analysis. The primary beam was polarized using Bragg crystal and Highly Ordered Pyrolytic Graphite (HOPG) target. The samples were measured at 300 and 180 s at voltage of 25 kV and 50 kV and current of 0.5 and 1.0 mA in a helium atmosphere by using the standardized method of fundamental parameters for cements or liquids. The chemical compositions of binder solids are listed in Table 1.

The particle size distribution of binder solids was determined using a laser granulometric analyser (Mastersizer 2000; Malvern Instruments Ltd, UK). The parameters of grain size distribution are characterized by $d(0.5)$ and $d(0.9)$ medians, which are also listed also in Table 1. The curves of particle size distribution of binders are shown in Figures 1–3, respectively.

The differences in the particle size of binding materials are evident and in accordance with their character. Results also inform indirectly about the specific surface area in terms of mutual comparison: the smaller the particle size, the larger the specific surface area.

2.2. Technological Characterization of Natural Zeolite. Technological characterization (water demand for standard consistency, initial setting time, activity index, and relative linear deformation) of natural zeolite was investigated. Both the water demand and initial setting time were tested on pastes composed of 50% of OPC and 50% of NZ and pastes consisting of OPC only, while the water-to-binder ratio was 0.5. Both the strength activity index and relative linear deformation were tested on mortars consisted of 1350 g of normalized silica sand, 500 g of binder, and 225 g of water, while two samples with different binder composition were tested. The control mixture contained 100% of OPC and research mixture contained 50% of OPC and 50% of NZ.

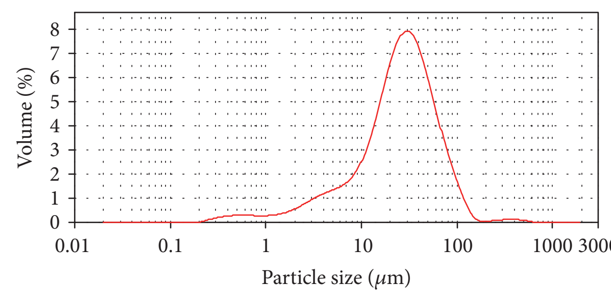

Figure 1: Particle size distribution of OPC.

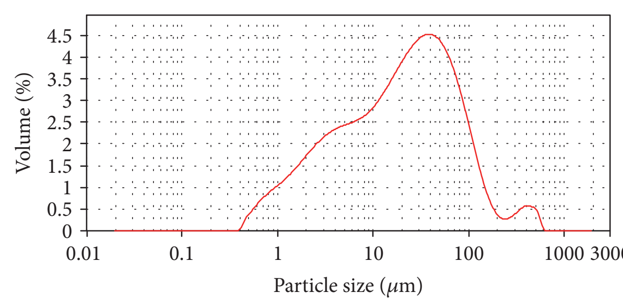

Figure 2: Particle size distribution of natural zeolite.

The water demand for standard consistency (the consistency that will permit the distance between plunger of Vicat apparatus and base-plate of 4–8 mm) and the initial setting time (the elapsed time, measured from zero to the time at which the distance between the needle and the base-plate is 3–9 mm) were measured using standard methods for testing cements (according to [26]).

The activity index was found in accordance with [27]. Here, the activity index refers to the ratio of the average compressive strength of SCM-based mortar and the reference cement-based mortar at the designated ages. Compressive strength was tested in accordance with [28].

The deformation changes of tested mortars were measured through active linear deformation [mm], using the

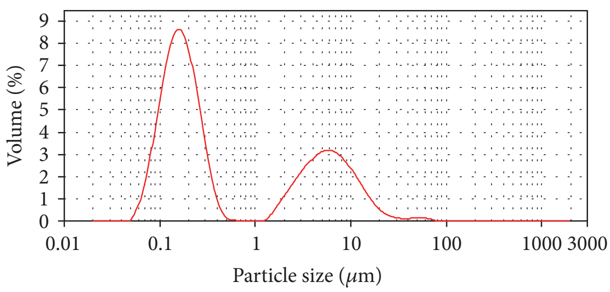

Figure 3: Particle size distribution of silica fume.

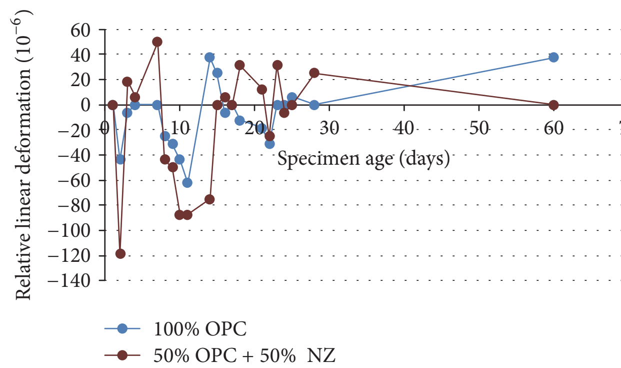

Figure 4: Relative linear deformation of the 100% OPC and 50% OPC + 50% NZ mortar.

length comparator, while three samples were measured for each recipe. The relative linear deformation was then calculated as the ratio of the active linear deformation [mm] to the actual length of the sample [mm]. Specimens of dimensions 40 × 40 × 160 mm were measured during 60 days at regular intervals. During this time, samples were kept in standard wet conditions (20°C temperature and water curing).

2.3. Physical-Mechanical Properties of Natural Zeolite-Based Concrete. The mineral binders (OPC, NZ, and SF) were used for concrete preparation. Natural normal-weight fine and coarse aggregates of fractions 0/4, 4/8, and 8/16 (NFA-0/4, NCA-4/8, NCA-8/16) were used. The superplasticizer based on polycarboxyl ether (PCE) was used to obtain optimal consistence together with tap water.

The experiment was focused on testing the concrete mixture designed by the acceptance of the standard recommendations for the composition for aggressive exposure. The recipe of the reference concrete was designed to achieve the compressive strength corresponding to C 35/45 strength class (according to [8]). Water-to-binder ratio (w/b) was 0.45 and cement content was 360 kg per 1 m^3 of fresh concrete. Natural zeolite was applied in varying replacement percentages to cement (8%, 13%, and 25% by weight). For directly showing binder modification by well-known silica fume, it was decided to use the combination of 8% of zeolite and 8% of SF. The materials and compositions of the tested concretes are given in Table 2.

The samples of cubes shape with dimensions 150 × 150 × 150 mm were prepared with standard methods. Specimens were released after one day and consequently cured in water under laboratory conditions until the tests execution. The standard tests (density, compressive strength, and water absorption) were executed after 28 days and the compressive strength after 365, 730, and 1095 days (one, two, and three years) according to [29–31].

3. Results and Discussion

3.1. Technological Characterization of Natural Zeolite. The water demand for standard consistency, initial setting time, and activity index of mortars are listed in Table 3. The consistent results comparing [17, 32, 33] have been found, in fact, to determine the expansion of water demand (probably due to a larger specific surface area) and to increase the setting time increasing of zeolite-blended mortars. The presented results for the zeolite paste also refer to the higher need for water to obtain the standard consistency. The initial setting time is practically three times longer than that of cement. Activity index after seven days (20.4) and 28 days (48.5%) shows low increase in strength at an early age and high cement replacement (50%).

Deformation changes in tested mortars are given through 60-day development of relative linear deformation, as shown in Figure 4.

Results of drying shrinkage presented worldwide are often not consistent because of nonuniform methods, apparatus, and curing conditions; these results are discussed in both positive and negative ways [21, 24, 34]. Here, the method of relative linear deformation is given for the direct comparison of two kinds of mortars cured under the same conditions, while the effect of zeolite is clearly visible. The relationship between the linear deformation changes and the risk of cracking is generally known.

The linear deformations of both samples are quite active up to 60 days, while the linear deformation of zeolite samples is somewhat larger than that of the cement-only samples; the values ranges of NZ and OPC based samples are −120 to +50 and −62 to +37, respectively. Jana [21] reports similar results when using 30% of zeolite as SCM; he obtained about 20% higher drying shrinkage than that of control mixture without zeolite.

3.2. Physical-Mechanical Properties of Natural Zeolite-Based Concrete

3.2.1. Density. The density of hardened concrete is referred as one of the durability-related properties, due to the porosity and permeability context. Concrete porosity is inversely proportional to density. Usually, the concrete permeability increases with an increase in porosity and decreases with an increase in density. The relation between permeability and porosity depends on the pore system properties. Lower concrete porosity leads to reduction in permeability and consequently in better durability. A very highly impermeable concrete reduces or eliminates the ingress of water and other aggressive chemicals and gases. This leads to improved concrete durability due to avoided expansive reactions, which can occur in the presence of these aggressive agents [35].

TABLE 2: The recipes of concrete mixtures. Components proportion was calculated for 1 m^3 of fresh concrete.

Mixture	OPC [kg·m^{-3}]	NZ [kg·m^{-3}]	NZ [%]	SF [kg·m^{-3}]	SF [%]	NFA-0/4 [kg·m^{-3}]	NCA-4/8 [kg·m^{-3}]	NCA-8/16 [kg·m^{-3}]	PCE [kg·m^{-3}]	w/b* [—]
V0	360	—	—	—	—	825	235	740	0.8	0.45
VZ1	330	29	8	—	—	825	235	740	0.8	0.45
VZ2	313	47	13	—	—	825	235	740	0.8	0.45
VZ3	270	90	25	—	—	825	235	740	0.8	0.45
VZ-SF	302	29	8	29	8	825	235	740	0.8	0.45

*Using the k-value = 2 for silica fume in accordance with EN 206.

TABLE 3: Technological parameters of zeolite comparing to cement.

Binder	Water demand [%]	Initial setting time [min.]	7-day activity index [%]	28-day activity index [%]
OPC	100.00	110	100	100
50% OPC + 50% NZ	126.62	320	20.4	48.5

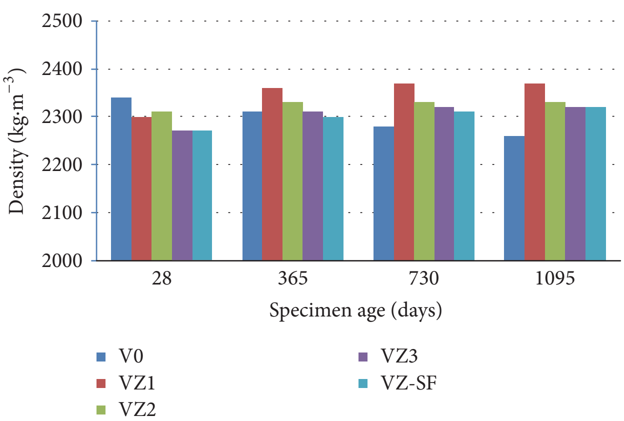

FIGURE 5: Density of hardened concretes after different time of hardening.

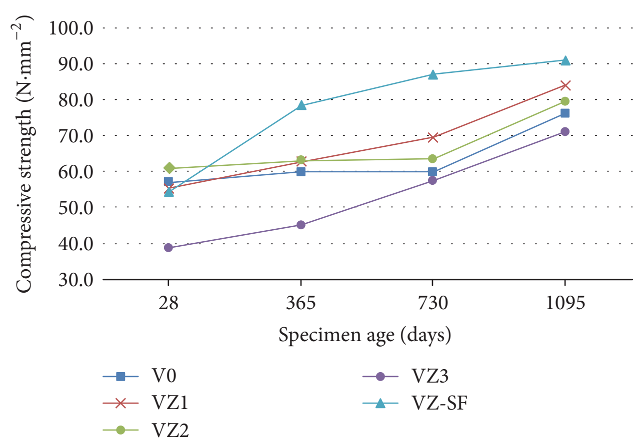

FIGURE 6: Time dependence of compressive strength of hardened concretes.

The results of density measurement are summarized in Figure 5. Values at 28 days and three years of setting and hardening ranged from 2270 to 2340 kg·m^{-3} and from 2260 to 2370 kg·m^{-3}, respectively. The density of all concretes increased during the time, excluding the concrete V0. The highest growth of density was found between measurements after 28 and 365 days—that is, during the first year of hardening. The density increased during the later stage only gradually. Concrete V0 obtained the highest density of all after 28 days but the lowest at the end of the experiment. Concrete VZ1 obtained highest density after one year and also in later stages. The amount of zeolite proportionally affected the density; thus, the higher dosage of zeolite caused the lower concrete density. This is in contrast to some research [36, 37], but, there, the zeolite content was up to 10%. According to Vejmelková et al. [18], both the bulk and matrix densities of the cement-based composites decrease with increase in the zeolite supplementary. Also, Poon et al. [19] observe that 15% replacement of zeolite resulted in lower porosity (affecting density), but a higher replacement amount (25%) increased the porosity at all the studied ages. Based on those comments and our results, the decrease in density can be attributed to the increase in zeolite amount.

The long-term results (one-, two-, and three-year) of all concretes incorporating the natural zeolite are higher than that of the OPC sample (V0), unlike the 28-day values. In this case, the results are lower and do not show the clear dependence on the amount of zeolite. This is clearly visible just after the long-term period. The presence of zeolite manifests itself differently over a longer period of setting and hardening of concrete.

Silica fume in combination with zeolite (concrete VZ-SF) decreased the density significantly compared to the reference concrete (V0) after 28 days. On the other hand, the density of VZ-SF was about 2.6% higher than V0 after three years.

3.2.2. Compressive Strength. The compressive strength was measured on 150 mm cubes after 28, 365, 730, and 1095 days of hardening. The time-development of compressive strength is shown in Figure 6. Generally, compressive strength of all concretes increased during the time, as also reported in previous research [17, 24]. The values after 28 days of setting and hardening ranged from 40 to 60 MPa, while values after three years ranged from 70 to 90 MPa. The initial reference strength (after 28 days) was almost the same for all concretes except VZ3. In the case of VZ3, the strength development was slower because of the high dosage of zeolite in the binder (25%). The early strength of concrete is commonly supplied by the hydration of CaO and silicates from cement clinker, while the pozzolanic activity of zeolite later influences the strength. The CaO/SiO_2 ratio of the zeolite used was under 0.5 (Table 1), which refers to its pozzolanic nature. It was in accordance with results from Ramezanianpour et al. and others [17, 36]. The amount of zeolite in total binder influenced the compressive strength, especially after two and three years of hardening. Concretes VZ1 and VZ2 with 8% and 13% of zeolite supplementary achieved about 16.4% and 6.2% higher compressive strength compared to the reference concrete after two years and 10.4% and 4.5% after three years, respectively. Concrete VZ3 with 25% zeolite supplementary obtained about 3.8% and 6.7% lower compressive strength compared to sample V0 after two and three years, respectively. According to Valipour et al. [38], the optimal content of zeolite to obtain highest compressive strength is 10%.

The significant increase in the compressive strength of all concretes was observed in the long term (three-year period). It confirms the long-term potential of natural zeolite to improve the mechanical properties. The positive change in strength was observed between 28-day and 3-year measurements, following the increase of about 33%, 53%, 30%, 83%, and 67% for concretes V0, VZ1, VZ2, VZ3, and VZ-SF, respectively. The concrete VZ3 achieved the most significant increase in compressive strength during the monitored period.

The silica fume significantly influenced the compressive strength of concrete. While the strength of VZ-SF sample after 28 days was similar to that of the other samples, the long-term strength values after forwarding periods were much higher. It is also evident that the strength development shows convex behaviour for all VZ concretes and concave behaviour for only VZ-SF concrete. Thus, the compressive strength of SF concrete increased faster than zeolite-based ones, but the strength of zeolite samples grew faster at the end of the monitored period.

The activity index was calculated as a relation between compressive strength of the related concrete and the reference concrete (VZ-/V0) after three years of hardening. This value characterizes the improvement or deterioration of zeolite-based concrete compared to the control one. It was 1.1, 1.0, 0.9, and 1.2 for samples VZ1, VZ2, VZ3, and VZ-SF, respectively. As expected, the higher zeolite content in binder caused lower compressive strength against the reference concrete.

With the exception of VZ-3, the long-term results (one-, two-, and three-year) of all concretes incorporating the natural zeolite are higher than that of the OPC sample (V0), except 28-day values. In this case, the results are close to each other and do not show the clear dependence on the amount of zeolite. This is clearly visible just after the long-term period.

3.2.3. Water Absorption. The water absorption of hardened concretes is summarized in Figure 7. Values after 28 days of setting and hardening ranged from 4.0 to 5.0%, while values after three years ranged from 4.2 to 5.7%. The long-term changes in water absorption of zeolite-based concretes were not significant. The relative differences in 28-day and three-year values were 12.5, 4.0, and 14.0% for VZ1, VZ2, and VZ3, respectively. The highest increase in water absorption (26.8%) was found for VZ-SF. Contrary to the control OPC sample (V0), values increase during the three-year period while the amount of zeolite in concrete influences the water absorption negatively. The higher content of zeolite leads to increase in water absorption. It is connected with the water absorption of natural zeolite, which is higher than that of cement [34]. Poon et al. [19] report that the porosity increases due to the higher replacement amount (25%) of zeolite at all studied ages; this may also be attributed to the higher absorption ability. It was recognized that the water absorption grew together with density. This result is unexpected, because several research works [17, 24] obtained adverse results.

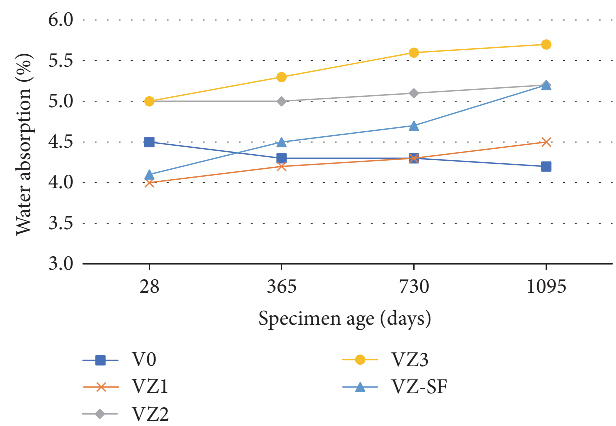

Figure 7: Time dependence of water absorption of hardened concretes.

The long-term results (one-, two-, and three-year) of all concretes including the natural zeolite are higher than that of the OPC sample (V0), unlike 28-day values. In this case, VZ1 and VZ-SF obtained lower values. The presence of zeolite manifests itself differently over a longer period of setting and hardening.

4. Conclusion

In this paper, the technological properties of natural zeolite and the long-term mechanical properties, density, and water absorption of concrete based on natural zeolite as SCM were evaluated. The results can be summarized as follows:

(i) At high cement replacement (50%) by the zeolite, the zeolite paste led to a higher demand for mixing water to obtain the standard consistency than that of OPC paste. The initial setting time was practically 2.9 times longer. Strength activity index after 28 days was 48.5%; this low increase in strength at an early age reflects the slow hydration process.

(ii) The long-term density of all zeolite-based concretes was higher than that of OPC concrete, unlike the

28-days values. Although the increase in zeolite amount caused the decrease in density, samples incorporating 25% zeolite achieved higher density than that of OPC in all later ages.

(iii) A major increase in strength was observed for all samples in the long term (three-year period). An increase in the zeolite amount caused a decrease in compressive strength, but the replacement of 8% and 13% of cement by natural zeolite brought about an improvement in long-term compressive strength compared to the only-OPC-based concrete. A higher replacement (25%) caused a decrease in strength after 28 days of setting and hardening; however, significant positive change (83%) was observed during the three-year period. This confirms the long-term potential of zeolite to improve the strength. Silica fume, in combination with natural zeolite, significantly improved the strength.

(iv) Long-term changes in water absorption of zeolite-based concretes were not significant, up to 14.0% in relative difference (26.8% for VZ-SF). Except for the V0 sample, values were increasing during the three-year period, while the higher amount of zeolite in concrete negatively influenced the water absorption.

(v) Generally, the long-term properties of zeolite-based concrete are different from that of 28-day properties, while both the density and compressive strength are influenced positively. The differences in concretes composition are manifested after a longer time, when the results are ordered more clearly and show a better sequence dependence on the sample's composition.

The results demonstrate the long-term potential of SCMs for the improvement of the concrete properties, which are important for the long-term role of concrete in the structure.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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