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Engineering of green cementitious composites modified with siliceous fly ash: Understanding the importance of curing conditions

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A B S T R A C T

Siliceous fly ash can be applied as a cement replacement material for mortars used in cement-based floor overlays. At present, there is no knowledge of the appropriate curing conditions that need to be applied on concrete floors. Moreover, previous research related to curing conditions has left research gaps related to the conditions in which such floors are cured. This study focuses on understanding the importance of the curing conditions that are typical for concrete overlays on the physical, chemical, mechanical, and microstructural properties of siliceous fly ash-modified mortars. The results indicate that curing conditions have a significant impact on these properties (particularly long-term effects). Differences were observed between siliceous fly ash-modified mortars cured in different conditions. In this research, we investigated the volume porosity, water absorption, microstructure, hydration process, pore segmentation, and strength of composites that have had cement partially replaced with the addition of fly ash. It should be noted that the use of siliceous fly ash in properly cured mortars that are applied in floor overlays could lead to a better use of green cementitious composites.

1. Introduction

Fly ash (FA) is commonly used in the construction industry [1,2,3]. While initially considered a waste that is recycled in cementitious composites, its importance has grown significantly in recent years, and it has become a desirable product [4,5]. The promising effects of replacing cement with the addition of fly ash in cementitious composites, such as enhanced workability, higher strength, lower shrinkage, or slower heat evolution, make it the most widely applied mineral replacement for Portland cement in cementitious mixtures [6,7].

Siliceous, calcareous and fluidized bed combustion are the most widely utilized types of fly ash. They differ in chemical composition, depending on the combustion process used in their formation. Usually, this process also influences the physical and chemical properties of the ash, such as fineness, phase composition, or pozzolanic activity. The type of fly ash also determines how it affects the properties of cementitious composites. Giergiczny [8] developed a general classification of fly ash types and compared their properties. Golewski [9] stated that the addition of fly ash in high strength concrete not only makes the material more sustainable, but also improves its mechanical properties. Golewski [10] observed, when fly ash is used as a cement replacement of 20%, that the Interfacial Transition Zone (ITZ) changes: the replacement of cement with the addition of FA results in smaller cracks in the ITZ. Yu and Ye [11] studied the pore structure of cement paste blended with fly ash, and observed a linear relationship between the total porosity and curing age. Yang et al. [12] reported a positive impact of the wet-milling of fly ash on the sustainability and properties of fly ash-modified with cementitious composites. Furthermore, the desired influence of fly ash on the properties of cementitious composites is the pozzolanic reaction, which leads to the balanced evolution of heat.

Cementitious mortars are commonly used in floor overlays. Floors are one of the key elements of buildings, the properties of which

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determine the potential of their usage [13–15]. The application of appropriate additives to the cementitious mixture may significantly improve its properties, such as workability, compressive strength, brittleness, or volume porosity [16,17,18]. Siliceous fly ash is commonly used for this purpose: it improves the workability and balances the heat evolution of the mixture. Han et al. [19] observed that heat released in the hydration process (during the first 72 h) is significantly lower in composites with the partial replacement of cement with fly ash than in the case of mixes made of 100% Portland cement. Yang et al. [20] investigated the effect of steam curing on the compressive strength and microstructure of high volume ultrafine fly ash (UFA) cement mortar, and found that it affects the early and late compressive strength, as well as the chemical composition of steam cured composites. De Matos et al. [21] noted that different types of fly ash have different effects on cementitious mixes. They confirmed that the heat release during the hydration of FA-modified mixes is reduced when compared to that of the reference mix. They also proposed an indicator of this reduction, which is connected with the compressive strength of the composite (°C/MPa). Wang et al. [22] concluded that fly ash, when compared to other Supplementary Cementitious Materials (SCMs), is more conducive to reducing the long-term hydration term (overall heat evolution) and shrinkage. They observed that the addition of fly ash has an impact on the amount of hydration products in composites. Yu et al. [23] noticed that the reacted hollow fly ash particles provide additional space for the accommodation of reaction products. They reported that the total porosity of blended cement pastes decreases only slightly over a long-time period (more than 1 year). In floor applications, slower strength development and the reduction of shrinkage are highly beneficial, and can increase the durability of the floor [24]. These properties make siliceous fly ash a useful cement replacement material in mortars applied in floor overlays.

An improperly prepared floor often leads to damage such as chipping, cracking, and delamination. To prevent this type of damage, it is essential to ensure that the cementitious overlay has appropriate curing conditions [25]. There are currently no proven curing conditions for floors. Therefore, recommendations for curing conditions are limited to general information known from descriptions of cementitious mortar technology. Termkhajornkit et al. [26] investigated the effect of water curing conditions on the hydration degree and compressive strength of fly ash–cement paste. They stated that curing conditions have a strong impact on the hydration of cementitious composites, which is not due to changes in the hydration rate of fly ash, but instead to the effect of the curing conditions on cement hydration (especially belite). They also observed that the water/binder ratio has an impact on the hydration degree of fly ash. Poon et al. [27] stated that fly ash has a significantly different influence on the strength, porosity, and durability of cementitious materials when subjected to different curing conditions. Ramezanianpour and Malhotra [28] noted that continuous moist curing is essential in order to achieve the highest strength, the lowest porosity, and the highest durability of composites. They also stated that fly ash-modified cementitious composites are characterized with better properties when compared to composites with the addition of other SCMs. Moreover, other researchers in [26,27,28] stated that the optimal curing conditions for cementitious composites are water-curing (90–100% humidity) at an elevated temperature (60–80 °C). Nevertheless, the specificity of floor overlays (large space, access to the element from only one side - the top side) means that it is not possible to ensure such conditions. Consequently, two curing conditions are usually used in the production of floors: 1) Air-curing (natural conditions in a space closed by envelopes: Humidity: 20–55% (variable) and Temperature: 16–30 °C (variable)), 2) Air-humid curing (natural conditions in a space closed with external partitions with cyclic pouring with water and covering with foil: Humidity: 55–90% and Temperature: 23 °C). These curing conditions are normally proposed based on experience, and there is a lack of scientific research on the influence of these types of curing conditions on the physical, chemical, and mechanical properties of mortars used to make floor overlays.

Considering the above, this study aims to understand and explain the influence of different curing conditions on the properties of siliceous fly ash-modified mortars used in floor overlays. In these studies, siliceous fly ash is used as a cement replacement material. We examine the effect of typical curing conditions on the mechanical, physical, chemical, and microstructural properties of mortars modified with the addition of siliceous fly ash. The selected conditions are typical for engineering practice when making cementitious floors. Due to the fact that the seleted conditions are based on experience, they have not yet been studied in detail, which makes this study both novel and practically relevant. Moreover, the use of siliceous fly ash in mortars applied in cementitious floors has not been studied before, with research primarily focusing on high-calcium fly ash. Therefore, research concerning the influence of siliceous fly ash and the different curing conditions of floors on the properties of mortars significantly contributes to fulfilling the current research gap. Another important novelty of the paper is the use of the latest techniques of microscopic analysis, as well as the processing of its data, in order to assess the impact of curing conditions on fly ash-modified cementitious materials. The article allows the phenomenon of curing conditions to be understood in a comprehensive way (to date, the influence of curing conditions on the mechanical and microstructural properties has mainly been analyzed).It should also be emphasized that the conducted research has a direct practical applicability, since the selected methods of curing correspond to those occurring on a construction site.

2. Materials and methods

2.1. Materials used in the research, the mix design of mortars, and the preparation of samples

The research consisted of investigating the influence of various curing conditions of cementitious mortars on their properties. For this purpose, four different series of cementitious mortars were made, which differed only in the amount of cement replaced with siliceous fly ash. Their compositions are presented in Table 1. Ordinary Portland cement CEM I 42,5R (Górażdże, Poland, Blaine specific surface area = 3650 cm^2/g), fine aggregate (Byczeń, Poland), and siliceous fly ash (Kogeneracja S.A., Poland, Blaine specific surface area = 4120 cm^2/g) were used in all the mixtures. In order to avoid the influence of chemical agents on the microstructural properties and chemical composition of the cementitious materials, no chemical agents (water reducers, air entraining agents) were used in the mixtures [29,30].

To map the conditions in which floor overlays may be cured on a construction site, three types of curing conditions are used: CC1 - Air cured, CC2 - Air-humid cured, and CC3 - water cured. The details of each condition are presented in Table 2. Samples were cured for 7, 28 and 90 days, depending on the test the sample was subjected to.

The mixing procedure was as follows. The air-dried ingredients were weighed and then mixed in a mixer (volume of mixer = 50 L), where they were mixed for 90 s. Then, water was added and mixed for 90 s. Finally, excess material was scraped off the sides of the mixer and the mixing was continued for 90 s. The mixture was then placed in standard moulds (40x40x160 mm). After 24 h, the samples were demoulded and placed in a storage area according to the curing conditions described in

Table 1
Mix design proportions of the mortars.

Series	CEM I 42.5	FA	Water	w/c	w/b	Sand
(-)	(kg/m^3)			(-)		(kg/m^3)
REF	280	0	140	0.50	0.50	840
FA10	252	28	140	0.56		840
FA20	224	56	140	0.63		840
FA30	196	84	140	0.71		840

Table 2
Curing conditions of the prepared samples.

Description	Stored	Humidity	Temperature
(-)	(-)	(%)	(°C)
CC1	air cured	20	23
CC2	air-humid cured	70	23
CC3	water cured	100	23

Table 2. Conditions CC1 simulate cementitious floors cured in air and which are unsecured by foil; conditions CC2 simulate floors secured by foil and which are cured by pouring water; and conditions CC3 simulate floors cured under ideal water conditions (underwater curing), which in practice is actually impossible for floors.

2.2. Granular analysis of the ingredients and properties of the fresh mortars

A test involving the sieving of powders through a set of gauges was performed to determine the particle size distribution of the dry ingredients. The investigations of the fresh mortar consisted of determining its setting time and consistency. The setting time was specified using Vicat apparatus according to [31]. The consistency of the mixes was tested using the slump flow method according to [32]. The chemical composition of the ingredients was measured using the JED-2300 energy dispersive X-ray (EDX/EDS) detector (Table 3).

Fig. 1a presents the particle size distribution of the ingredients used in the study. The particle size distributions (PSD) of the Portland cement and siliceous fly ash used in the research are very similar. A similar PSD composition facilitates the mixing of ingredients and prevents segregation. Moreover, a properly selected siliceous fly ash PSD allows the consistency of the cement mixture to be modified. The grains of siliceous fly ash (a) and Portland cement (b) reveal slightly different shapes: siliceous fly ash is characterized by spherical grains, while Portland cement grains resemble parallelograms (Fig. 2). It can be observed that siliceous fly ash contains some larger particles that can be easily seen, whereas Portland cement has significantly finer particles.

2.3. Determination of the principal properties of the hardened mortars

The volume porosity and water absorptivity of the hardened mortars were investigated according to [33]. Samples with dimensions of 40 × 40 × 160 mm were dried for at least 24 h at a temperature of 110 °C with the use of a Zyle Professional dehydrator until a constant weight was obtained.They were then measured and weighed. After that they were fully soaked in water and weighed until they reached a constant weight. The volume porosity (V_p) and water absorptivity (W_a) of the mortars was determined according to equations (1) and (2).

The volume porosity (V_p) was determined on the basis of the ratio of the air pore volume (V_{ap}) in the tested material to the volume of the tested sample (V).

$$V_p = \frac{V_{ap}}{V} * 100\%(\%) \tag{1}$$

Table 3
Measured chemical compositions of the ingredients used in the research.

Ingredient	Cement CEM I 42,5R	Siliceous fly ash
Chemical compound	[wt. %]	
CaO	63,94 ± 1,17	3,34 ± 1,82
SiO_2	18,86 ± 1,37	54,33 ± 2,18
Al_2O_3	5,73 ± 1,25	30,65 ± 1,63
FeO	2,75 ± 2,59	4,78 ± 3,60
MgO	1,54 ± 1,25	2,45 ± 1,57
SO_3	5,89 ± 1,26	0,00
K_2O	1,30 ± 0,83	2,51 ± 1,38

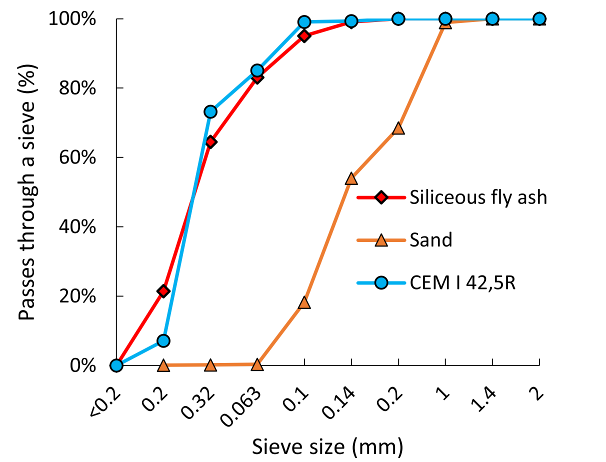

Fig. 1. Particle size distribution of the materials used in the research.

The water absorptivity (W_a) was determined on the basis of the dry sample mass (m_d) and the wet sample mass (m_w).

$$W_a = \frac{m_w - w_d}{m_d} * 100\%(\%) \tag{2}$$

The principal mechanical properties of the hardened mortars were determined after 7, 28, and 90 days of curing. For this purpose, compressive and flexural strength tests were carried out. The compressive strength was tested according to [34] on samples with dimensions of 40 × 40 × 80 mm. The flexural strength was tested according to [34] on samples with dimensions of 40 × 40 × 160 mm. In total, 10 specimens from each series were used for the compressive strength testing, and 5 for the flexural strength testing.

2.4. Microstructural analysis

Microstructural analysis was performed using the JEOL JSM-6610A Scanning Electron Microscope (SEM). A beam current of 38nA, an accelerating voltage of 20 kV, the material contrast mode (backscattered electrons), the topography contrast mode (secondary electrons), and a working distance of 10 mm were applied. The observations were performed on the samples' cross-sections with dimensions of 20x20 mm. The preparation of the samples for the microstructural observations consisted of them being immersed in a non-conductive epoxy resin, ground by sandpaper with a number of 120–1200, and polished by a diamond medium with a size of 6 µm and 1 µm. In order to obtain electrical conductivity, as well as to avoid the electrical charging of the samples, a conductive path made of copper tape was used. Afterwards, the prepared samples were coated with carbon. A conductive carbon layer with a thickness of 40 nm was obtained from graphite electrodes in a vacuum of 2 × 10^{-5} Pa (2 × 10^{-6} Torr) using the thermal sputtering method.

The chemical element composition was measured using the JEOL JED-2300 energy-dispersed X-ray spectrometer (EDS). The detector's dead time did not exceed 15%, and the count rate was set to approximately 4000 cps. Quantitative analyses of the chemical composition were carried out with the use of JEOL software (JEOL JED-2300 Analysis Station) using the integrated ZAF method. The quantitative results of the chemical composition were performed with the assumption that the identified chemical elements form oxides. The chemical composition of the samples were performed for an area of 445x300 µm for each sample (magnification of 300x). The size of the analyzed area allowed for the analysis of places where there were no grains of sand, and it was therefore possible to avoid obtaining uncertain results that would be influenced by such grains of sand.

The analysis of the near-surface area porosity of the samples was performed for three reference samples (REF CC1, REF CC2, and REF

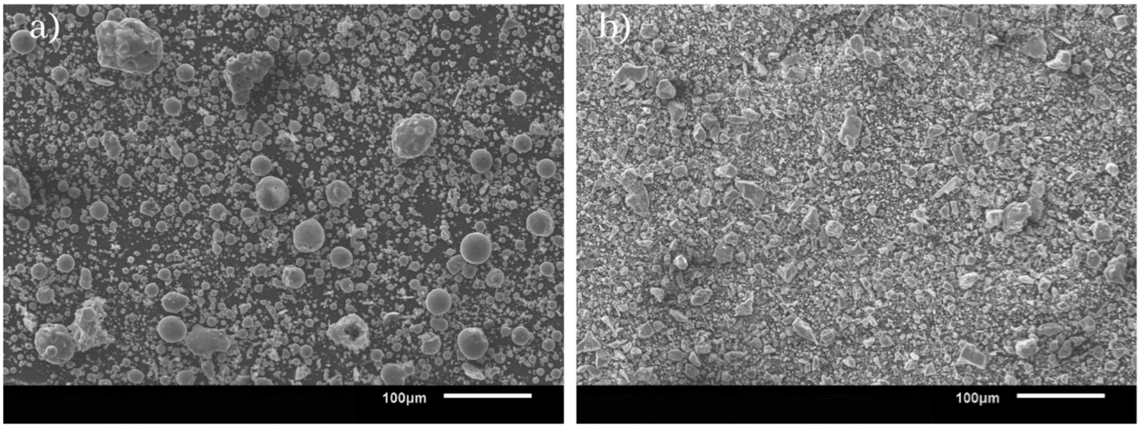

Fig. 2. The morphology of a) siliceous fly ash, and b) cement CEM I 42,5R. Images taken using SEM and a SE detector.

CC3) and for three samples with the addition of fly ash (FA30 CC1, FA30 CC2, and FA30 CC3). The images of the microstructure of the samples at 500x magnification were analysed using the ImageJ program following the methodology developed in [35]. First, the optimal "threshold" value on the cumulative grayscale histogram was determined for each image separately. The "threshold" value means that all the pixels in colors on the greyscale histogram from 0 to the "threshold" value are considered as pores. All the pores were assigned a black color using the ImageJ program, with porosity images thus being created. From the images of the segmented pores, pore size distribution plots were made for each sample using ImageJ and Excel. First, each image was properly scaled in ImageJ ("Set Scale" function), and then, using the "Analyze Particles" function, a list of pores with their area in μm^2 was generated. The obtained data was then grouped by pore size, and the size of each set was counted in Excel.

For all the microstructural analyses, a minimum of 10 images of each sample were used. The analyses were performed after 90 days of curing.

3. Results and discussion

3.1. Properties of the fresh mortars

The setting time of mortars depends on the amount of added fly ash (Fig. 3). The results of the experimental research are presented in the graph using solid colours. It can be seen that as the amount of FA in the mix increases, so does the initial and final setting time. The addition of fly ash (30%) caused the extension of the initial setting time by 65 min (i.e. by 25%), which can be seen to be significant. A similar result was also obtained for the final setting time (the time increased by 19%). Giergiczny [8] described a similar effect of adding fly ash on the setting time of mixes. He observed that the addition of most SCMs (as fly ash, slag) results in a longer setting time. The graph also shows the results of setting time tests from the literature [36,37]. It should be noted that there are significant differences in the values of the results obtained by the authors (therefore, it was decided to use the normalized method of comparing results). However, a trend that is similar to the one obtained in the experimental studies can be noticed. In this study, it was observed that the replacement of cement with fly ash in the cement mixes delayed the initial time of setting. It was noticed that cement replacement with the use of fly ash results in a delay in the final setting time. No significant changes between the initial and the final setting times were observed as a result of adding fly ash to the mixes when compared with the reference series. The results obtained by Yilmaz and Olgun [36] show a smaller influence of fly ash on the changes of setting times when compared with the current research. The different values of setting time obtained by Yilmaz and Olgun [36] can be related to the type of fly ash used in their study – low-calcium fly ash from Turkey and the addition of limestone to mixes. Guneyisi et al. [37] also reported a similar (as obtained in this research) variability of setting time results. This is related to the use of the same type of fly ash in the two studies – siliceous (sourced in Turkey). However, the values are somewhat different to those reported herein. Yilmaz and Olgun [36] showed that the addition of 30% of fly ash causes a 14% extension of the initial setting time and a 31% extension of the final setting time. The greatest variability was obtained in the study of Guneyisi et al. [37], where for the addition of 30% of fly ash, the initial setting time was longer by about 64%, and the final setting time by 63%. This can be attributed to the use of concrete in their study instead of mortar.

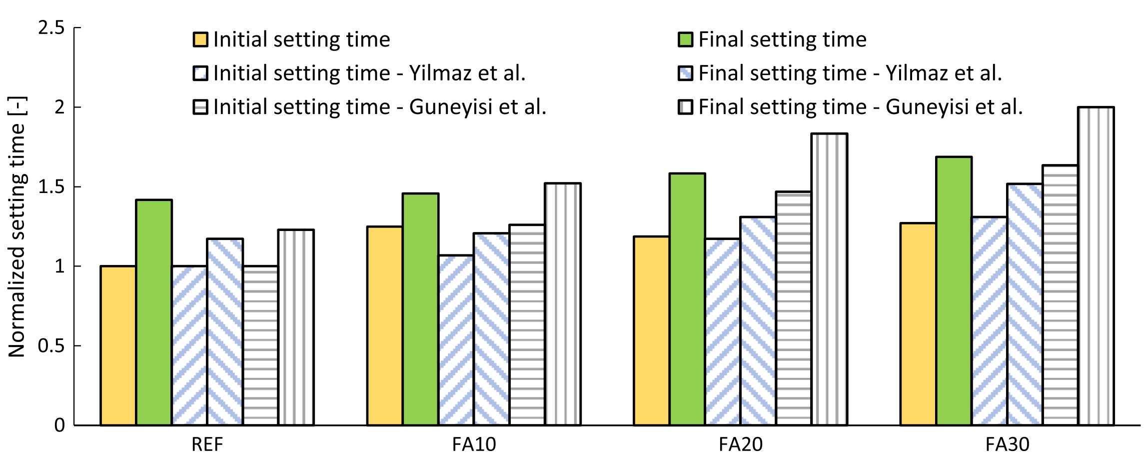

Fig. 3. Normalized setting time of the fresh cementitious mixes modified by siliceous fly ash when compared with similar mixtures based on published research.

The slump flow diameter that was measured in the current research (solid colour) is significantly different than the findings from literature [37] (grey hatched columns) - see Fig. 4. It can be seen that as the amount of the fly ash supplement increases, so does the diameter of the slump flow. The research series modified with the addition of 30% of fly ash have a larger slump flow diameter (14% more) when compared to the reference series. A similar dependence has been reported elsewhere [37]. It is widely known that the addition of fly ash in mixtures results in better workability and an increase of the slump flow diameter [38,39]. This is mainly due to the morphology of fly ash grains (spherical grains), which have a different structure than Portland cement or aggregate particles (angular grain structure that resembles a parallelogram). The fly ash used herein is characterized by a higher specific surface area of grains (4120 cm^2/g) than Portland cement, which results in a higher slump flow diameter in the mortars. Both binders have a similar particle size development of grains, and therefore differences in workability should only be assosciated with the morphological properties of the grains. A Mora et al. [40] studied the workability of fly ash-modified mortars.They observed that the specific surface area of the ingredients affects the slump spread diameter. They concluded that in the case of the finest fractions, the lubricant effect of the surfaces of the fly ash particles counteracted water adsorption. They observed that the particle sieve size development of fly ash has a significant impact on the workability of mortars. Giergiczny [8] concluded that the glassy phase of the spherical shape of FA grains allows more liquid mixes (with the same, or lower amount of water in the mix when compared to the reference mix) to be obtained.

The differences in the values obtained herein and the values obtained in the literature result from the differences in the cement used for the tests and the differences in the storage conditions of the material before the test [36,37]. Güneyisi and Gesoğlu [37] performed tests for self-compacting concrete and obtained results that are different to those obtained herein. However, the goal of the comparison was to analyse the trends, and not the exact values.

3.2. Physical and mechanical properties of the hardened mortars

3.2.1 vol. porosity of the mortars

The volume porosity (V_p) of the mortars with a partial replacement of cement with the addition of fly ash depends on the curing conditions of the composite (Fig. 5). In fact, the volume porosity is influenced by the curing conditions and the amount of fly ash in the mortars. Air-cured mortars have the highest porosity. Air-humid cured mortars have a lower porosity than air-cured mortars, and a higher porosity than water-cured mortars. It can also be stated that their volume porosity decreases with an increase in the amount of fly ash in the mortars. Therefore, the method of curing and the amount of fly ash have a significant impact on the volume porosity of mortars modified with fly ash. This is consistent with previous studies [27,28,41]. Ramli et al. [41] concluded that the porosity of cementitious composites depends on the curing conditions and modifiers (temperature, humidity) during the curing of samples. They also stated that the curing conditions have an impact on the shape and volume of the air pores in composites. Ramezanianpour and Malhotra [28] studied the effect of different curing conditions of mortars modified with supplementary cementitious materials (SCMs) on their porosity. They concluded that it is important, for SCM-modified composites, to test the porosity after more than 28 days (due to the pozzolanic activity of SCMs). They also concluded that the lowest porosity of motars modified with SCMs is achieved for moist curing conditions, while in room-curing conditions composites exhibit a higher porosity. Poon et al. [27] studied the effect of curing conditions on the pore structure of fly ash cement pastes and mortars. They stated that the porosity of fly ash-modified composites after 28 days was slightly higher than composites without the addition of FA, while after 90 days, the FA activity was visible – the porosity of FA-modified composites is significantly lower than that of composites without FA. There was also a difference in the mean pore size and pore size distribution. The volume porosity of mortars also has a significant impact on the durability of cementitious floors [42,43]. A lower porosity should, in principle, result in more durable floors.

3.2.2. Compressive strength

After 7 days of curing, the highest compressive strength was demonstrated by the REF water-cured mortars (Fig. 6). The strength decreased as the amount of cement in the mortars decreased (i.e. with an increasing percentage of fly ash). After 28 days of curing, the REF mortars still outperformed the FA samples. The strength decreased when the amount of cement in the mortars decreased, but the differences were smaller than in the case of the samples cured for 7 days. After 90 days of curing, there were significant differences when compared to 7 and 28 days (Fig. 6). The FA30 water cured series showed the highest strength, and there was an increase in compressive strength with an increase in the amount of fly ash in the composites. Similar observations were reported by Poon et al. [27]: a significant influence of curing conditions on the mechanical properties of mortars modified with the addition of fly ash was observed (even a 30% increase in compressive strength for water cured mortars when compared to air-cured mortars). Danish et al. [44] also reported an increase in compressive strength for composites

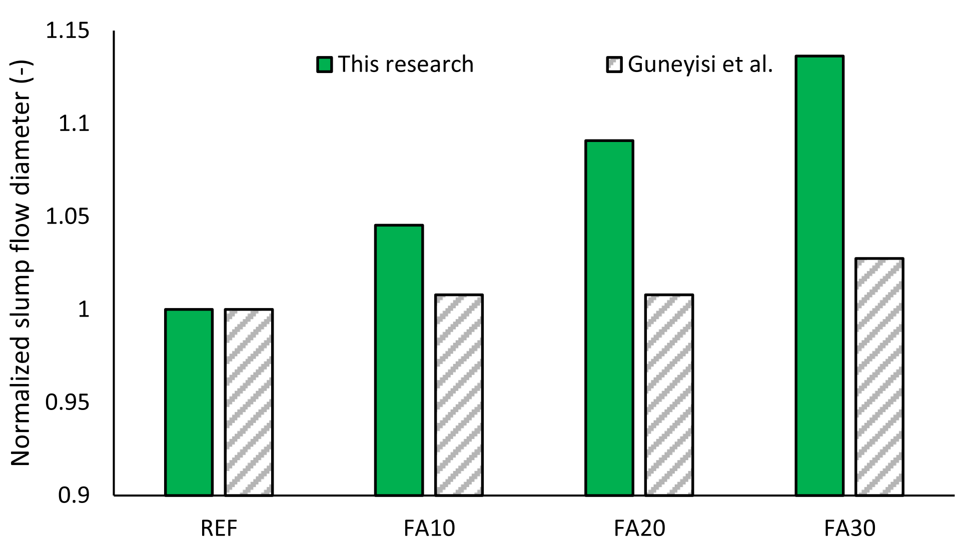

Fig. 4. Normalized slump flow diameter of the fresh cementitious mixes modified by siliceous fly ash when compared with similar mixtures from the literature.

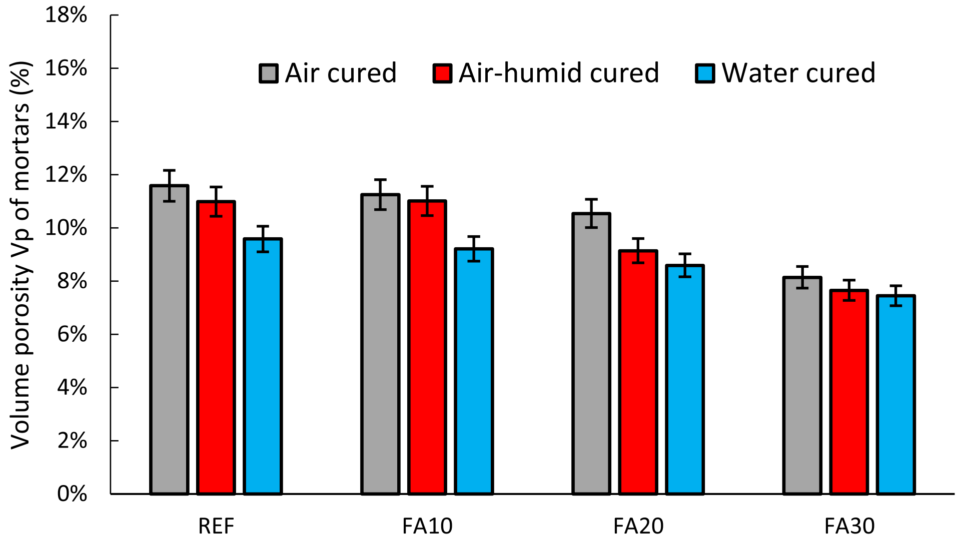

Fig. 5. Volume porosity (V_p) of the mortars with the partial replacement of cement with the addition of fly ash after 90 days of curing.

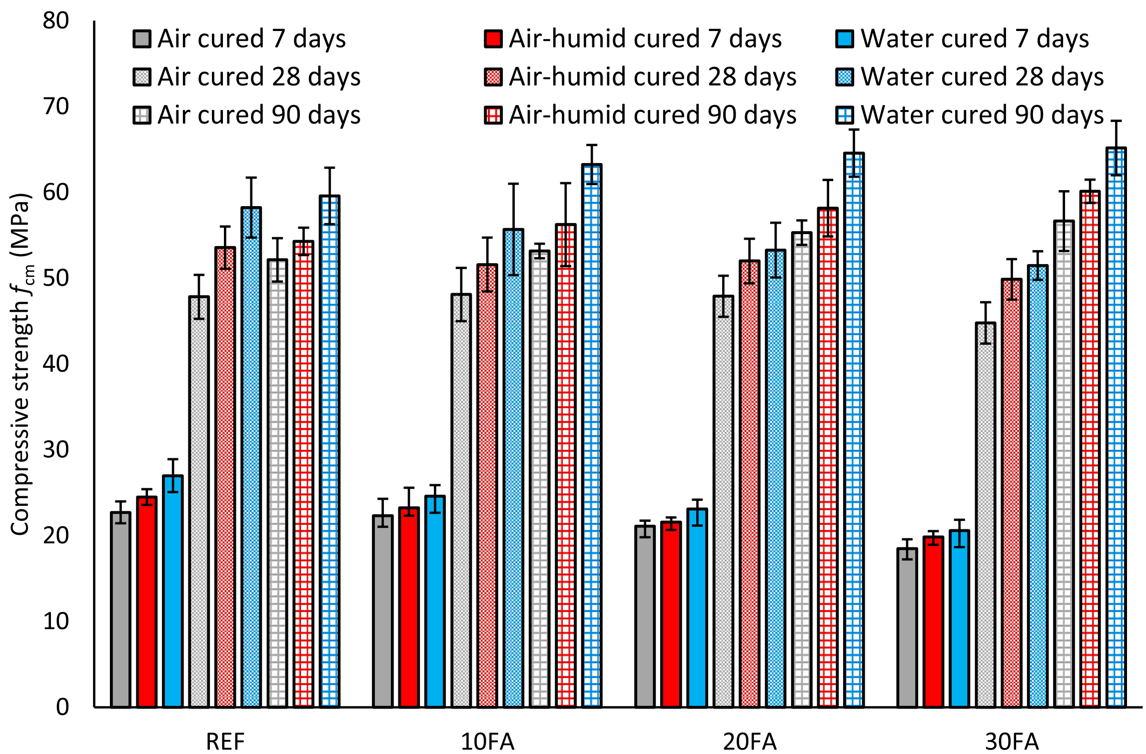

Fig. 6. Compressive strength (f_{cm}) of the cementitious mortars modified with siliceous fly ash after 7 days, 28 days and 90 days of curing.

cured in water (70% higher strength). They observed that the curing temperature has an important impact on the mechanical properties of cementitious composites. The research allows the authors to conclude that the mechanical properties of composites are connected with their microstructural properties. Other researchers observed similar trends [45,46].

3.2.3. Flexural strength

The flexural strength results vary depending on the curing conditions and the amount of cement replaced by fly ash. The highest flexural strength was demonstrated by the water cured REF mortars. The strength decreased as the amount of cement in the mortars decreased. Fig. 7 shows the flexural tensile strength of the mortars after 28 days. Moreover, the highest strength was also demonstrated by the water cured REF mortars at 28 days. The strength decreased as the amount of cement in the mortars decreased. The water cured samples showed the highest strength (almost 20% higher than the air-cured mortars). Fig. 7 shows the graph of the flexural strength after 90 days. There are significant differences between 7 and 28 days, because the water cured FA30 series has the highest strength. There was an increase in flexural tensile strength with an increase in the amount of fly ash. Similar observations were reported by Danish et al. [44]. They observed an increase in flexural strength for composites cured in water (70% higher strength). They stated that fly ash-modified composites cured in water and at a high temperature (80 °C) show the best mechanical properties.

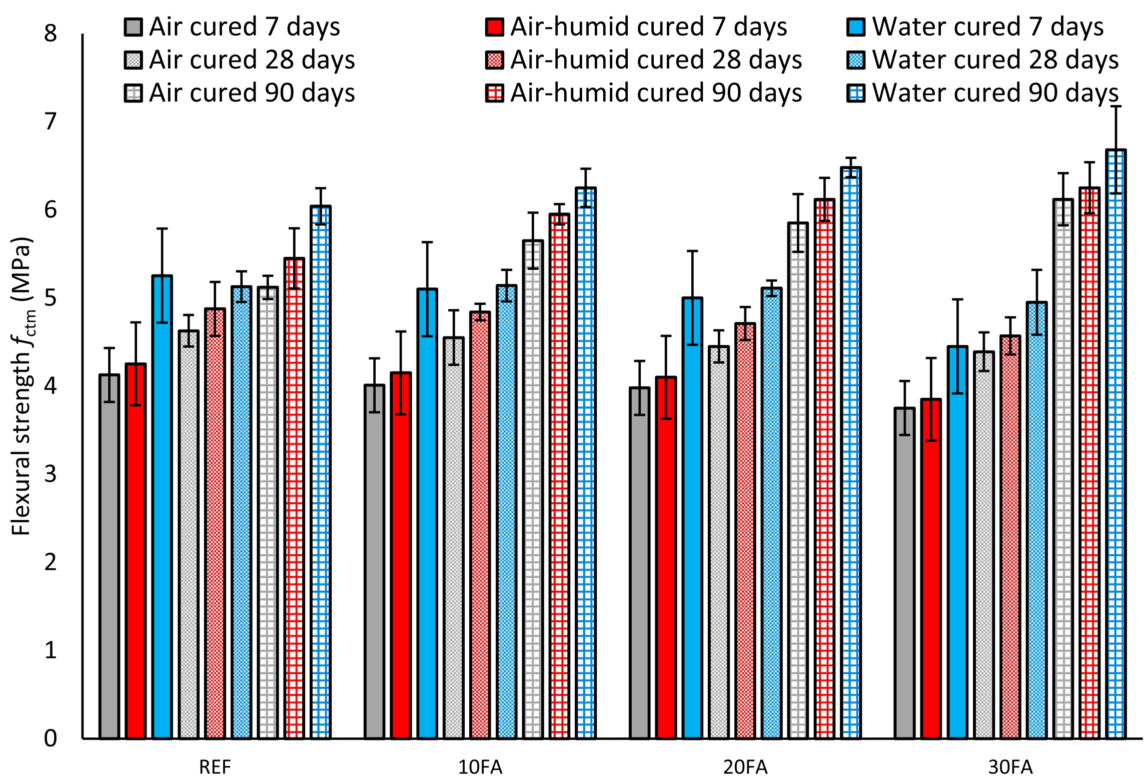

Fig. 7. Flexural tensile strength (f_{ctm}) of the cementitious mortars modified with siliceous fly ash after 7 days, 28 days and 90 days of curing.

3.3. Physical, chemical and microstructural impact of the curing conditions

3.3.1. Physical effect

The mechanical properties of the cementitious mortars modified with the addition of siliceous fly ash are clearly strongly connected with the curing conditions. However, it should be investigated why curing conditions have a significant impact on mechanical properties. To solve this issue, we decided to investigate the connection between the mechanical properties of mortars and their physical, chemical, and microstructural properties. To achieve these goals, we investigated the physical, chemical, and microstructural properties of the mortars.

Physical properties, such as porosity and water absorptivity, have a significant impact on the durability of cementitious floors. Fig. 8 presents the relation between the mechanical (compressive strength and flexural strength) and physical properties: a) volume porosity (V_p), and b) water absorptivity (W_a).

The mechanical properties of the mortars are clearly related to their

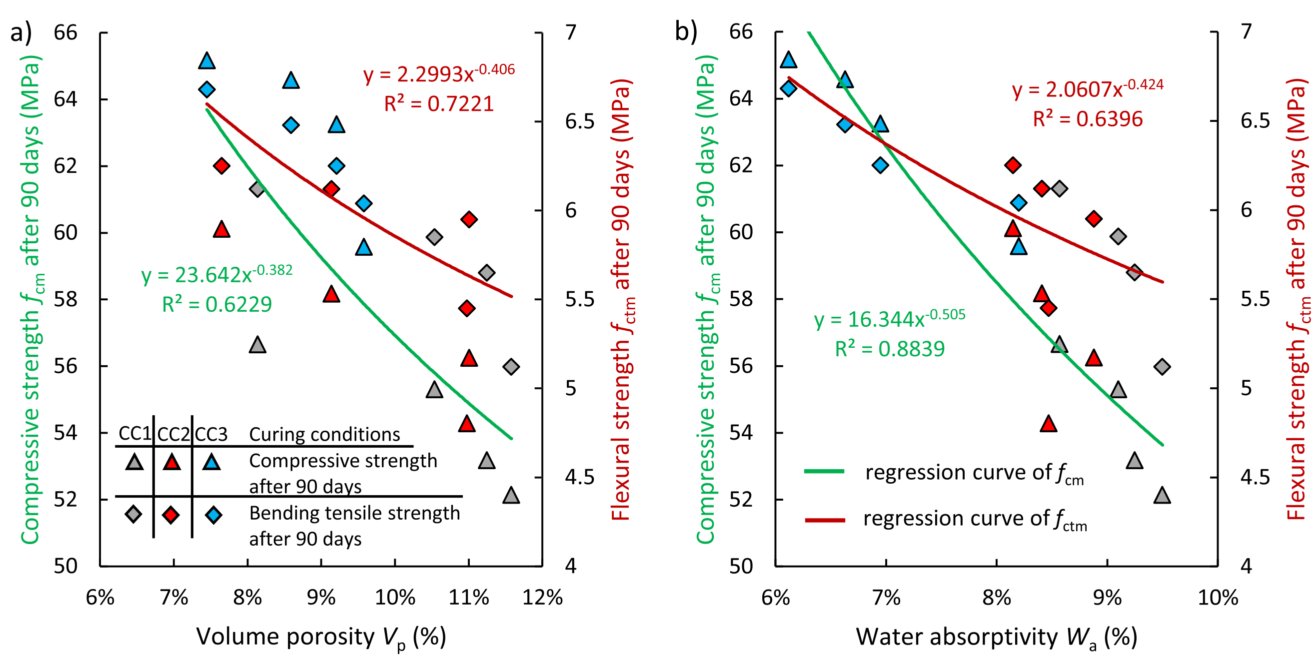

Fig. 8. Relationship between the mechanical properties of the mortars after 90 days and a) volume porosity (V_p) and b) water absorptivity (W_a).

physical properties (Fig. 8). As expected, an increase in volume porosity was correlated with a decrease in the mechanical properties of the mortars. The curing conditions had a significant impact on this relation. The water-cured samples (CC3) were characterized by improved mechanical properties and a lower volume porosity. This was not observed in the samples cured in air cured (CC1) and air-humid cured (CC2) conditions. Fig. 8b presents the realationship between the mechanical properties (compressive strength and flexural tensile strength) and the water absorptivity of the mortars. Similar to Fig. 8a, it can be observed that mechanical properties are related with physical properties. Moreover, the water-cured samples (CC3) showed better mechanical properties and lower water absorptivity. It was observed that the samples cured according to CC2 conditions had better properties when compared to the samples cured according to CC1 conditions (i.e. higher mechanical properties and lower water absorptivity). Danish et al. [44] observed similar effects. They stated that the curing conditions of fly ash-modified mortars have an impact on the chemical composition and microstructure of composites, as well as on the hydration process. This was also reported in other research [47,48].

3.3.2. Chemical effect

The chemical compositions of composites after curing time are crucial, as they enable changes caused by different curing conditions to be determined. The chemical composition of the mortar has a significant impact on the mechanical properties of the composite. The FA30 series, after 90 days of curing, exhibited the highest mechanical properties. On the other hand, after 7 days of curing, the REF series was characterized by the best mechanical properties. To compare the impact of fly ash and different curing conditions, we decided to investigate these two series of samples. Both series were tested after 90 days of curing (chemical composition and microstructural properties). Table 4 presents the chemical compositions of the investigated mortars.

It can be observed that the analysed compound proportions for the REF series are significantly lower when compared to the FA30 series. It can also be seen that all of the investigated proportions are higher for the CC3 curing conditions (blue colour). Calcium oxyde is the main compound occurring in Portland cement. The majority of the properties of cementitious composites depend on the amount of calcium oxyde. To analyze the chemical composition of the specimens cured in different conditions, we decided to investigate the proportions of Al_2O_3/CaO and SiO_2/CaO. The Al_2O_3/CaO ratio is often related to the synergistic effect of fly ash in cementitious materials. This tendency was also observed in the results obtained in these studies. The properties of fly ash-modified mortars are widely known [49,50]. The water used in the curing of the cementitious samples in the CC3 conditions may have had some impact on the chemical composition of the composite. Nevertheless, in order to determine the chemical composition of each research series, 10 × 10 × 10 mm samples were taken from the center of the larger 40 × 40 × 160 mm samples (which were cured according to curing conditions CC1-CC3). Due to this, a distance of about 15 mm (between the edge of the smaller sample and the edge of the bigger sample) was obtained. Jiang et al. [51] studied the impact of acid corrosion on the depth of changes in cementitious composites. They concluded that after 90 days exposition to acid corrosion, the depth of changes to the structure and properties of cement pastes (w/c = 0.4 (-)) was about 10 mm. Similar conclusions were obtained by other researchers [52–54]. Based on this, it can be concluded that the effect of calcium hydroxide corrosion (leaching of calcium by the water used during the curing of the samples (CC3)) is negligible with regards to the obtained results presented in Fig. 9a. For a higher Al_2O_3/CaO ratio, the highest synergistic effect is observed. Fig. 9b presents the chemical–mechanical analysis of the investigated specimens. It can be observed that the chemical composition is clearly connected with the mechanical properties of the specimen. The samples with higher proportions of Al_2O_3/CaO and SiO_2/CaO showed better mechanical properties. When analyzing these results, it can be stated that the SiO_2/CaO proportions are dependent on the curing conditions. In this study, the synergistic effect can be observed. Moreover, it can be noted that curing conditions have an impact on the elevated synergistic effect in fly ash-modified cementitious composites. The synergistic effect and curing conditions are very important in the case of cementitious floors. Other researchers emphasized the relationship between the chemical composition and durability of cementitious floors [42,55,56].

Table 4
Chemical composition of the mortars modified with the addition of siliceous fly ash cured in different conditions after 90 days - determined using the EDX/EDS method.

Series	REF	FA30	REF	FA30	REF	FA30
Curing conditions	CC1 Air cured		CC2 Air-humid cured		CC3 Water cured	
Chemical compound	(wt. %)					
CaO	64,42 ± 1,01	49,73 ± 0,75	59,58 ± 0,65	45,19 ± 0,84	59,47 ± 0,86	44,46 ± 0,75
SiO_2	22,59 ± 0,74	29,21 ± 0,88	25,63 ± 0,75	34,87 ± 0,62	25,61 ± 0,63	34,88 ± 0,88
Al_2O_3	5,92 ± 0,71	12,46 ± 0,76	6,25 ± 0,68	12,21 ± 0,56	7,15 ± 0,62	12,31 ± 0,76
FeO	2,78 ± 2,40	3,59 ± 1,60	2,90 ± 1,43	2,81 ± 1,95	3,08 ± 2,05	2,59 ± 1,60
MgO	1,37 ± 0,82	2,03 ± 0,76	3,67 ± 1,25	1,99 ± 0,86	1,75 ± 0,96	1,93 ± 0,96
SO_3	2,94 ± 0,55	2,00 ± 0,84	1,96 ± 0,72	2,10 ± 0,55	2,94 ± 0,55	2,62 ± 0,84
K_2O	0,00	0,98 ± 0,54	0,00	0,84 ± 0,69	0,00	1,20 ± 0,54

3.3.3. Microstructural effects

Fig. 10 presents the Scanning Electron Microscope (SEM) images of the specimens of siliceous fly ash-modified mortars cured in different conditions. Some important properties are marked in different colours: green – unhydrated cement particles, blue – siliceous fly ash particles, orange – microstructure cracks.

Based on the SEM observations, it can be noted that the microstructure of the fly ash-modified composites is different to the reference specimens. Moreover, the reference samples are characterized by more microcracks.There are sometimes undeveloped grains among the fly ash grains, which reduce the reactivity of the ash in the cement composites [57]. The unreacted fly ash particles are especially visible in Fig. 10d, and may indicate that the material is of poor quality. However, it should be mentioned that the fractions of the unreacted particles are relatively small. It can also be seen that there are fewer unhydrated cement particles for the CC3 curing conditions when compared to the CC1 curing conditions. To analyse the composites' microstructure in more depth, surface analysis was performed. Fig. 11 shows images of the segmented pores of the investigated samples.

The most numerous group of pores are those up to 2 μm^2 (from 900 to 1,450 units) (Fig. 12). When comparing the obtained results, the unequivocal effect of the curing method on the number of pores up to 2 μm^2 was not noticed. On the other hand, the results show an increase in the number of pores of up to 2 μm^2 in the samples with the addition of fly ash when compared to the corresponding reference samples. For the remaining pore sizes, a tendency was observed that the air-cured samples most often have the largest number of pores of a certain size; the air-humid cured samples have an intermediate number; and the samples cured in water have the lowest number of pores of a certain size. This tendency is visible for both the reference samples and the samples with the addition of fly ash.

For each image of the segmented pores, the percentage of black pixels in the image was calculated using Mathematica, with the average porosity for each sample thus being obtained (Fig. 12). When comparing the images of the segmented pores for the reference samples (Fig. 11 a, b, c), it can be seen that the REF CC1 sample has the highest average

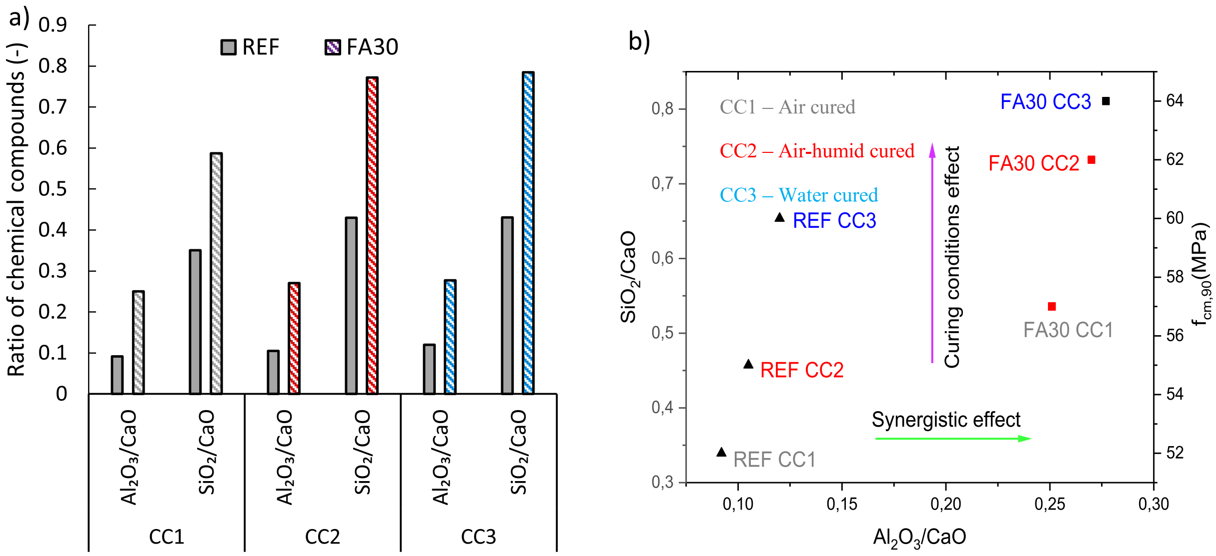

Fig. 9. Analysis of the chemical composition of the mortars: a) chemical analysis determined using the EDX/EDS method, b) chemical–mechanical analysis.

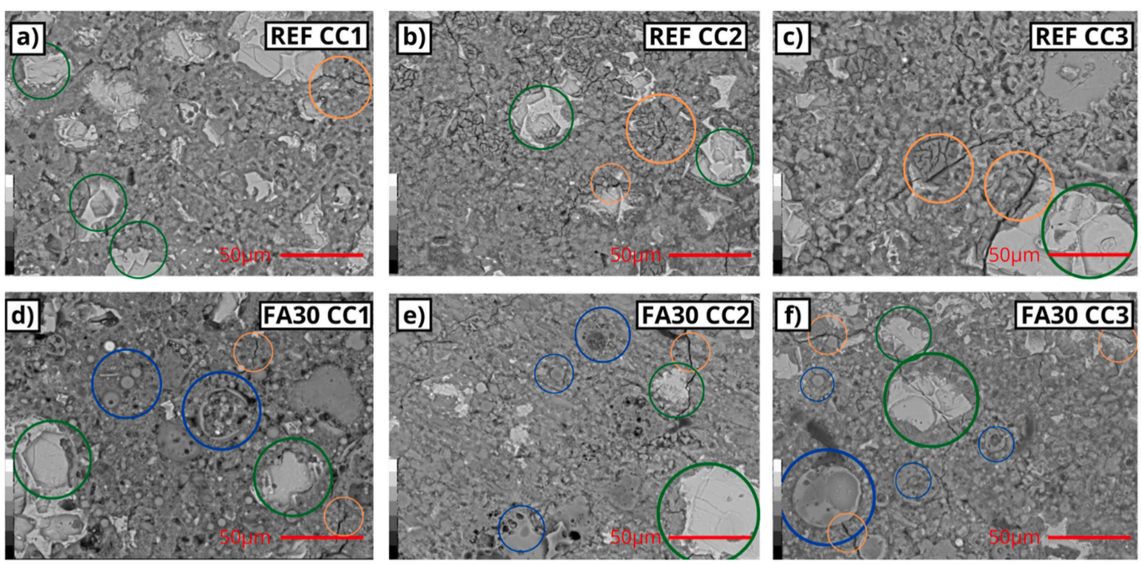

Fig. 10. The microstructure of the tested samples after 90 days of curing: a) REF CC1, b) REF CC2, c) REF CC3, d) FA30 CC1, e) FA30 CC2, f) FA30 CC3; Scanning Electron Microscopy with the use of a BSE detector; markings: green – unhydrated cement particles, blue – siliceous fly ash particles, orange – microcracks, (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

porosity (9,3%) when compared to the REF CC2 (av. porosity 8,5%) and REF CC3 (av. porosity 7,6%) samples. Similarly, the FA30 CC1 sampe has a higher average porosity (10,5%) when compared to the FA30 CC2 (av. porosity 9,3%) and FA30 CC3 (av. porosity 8,1%) samples (Fig. 11 d, e, f).

On this basis, it can be concluded that the wetter the curing conditions of the mortar, the lower the number of pores of a certain size, except for pores below 2 μm^2 (Fig. 12). Furthermore, the addition of fly ash significantly increases the number of pores with a diameter of up to 2 μm^2 (Fig. 12), whereas it has a lower effect on the number of pores with a diameter of above 2 μm^2.

The analysis of the Scanning Electron Microscope (SEM) images, with particular emphasis on the frequency of pixels on the images, is presented in Fig. 13. According to the procedure described in [35,58], an analysis of hydration of cementitious mortars cured in different conditions was performed. The grey value hystograms show visible peaks that are related to the number of pores, hydration products (HP), and the amount of calcium hydroxide (CH)..

Fig. 13a presents the frequency of the pixels of the SEM images for the REF series samples. It can be seen that the samples cured in the CC1 conditions show the lowest amount of pores (grey value 50–80), but the differences are not very significant. However, for the hydration products (HP), there is a difference in the frequency of the pixels – the samples cured according to the CC3 conditions present the highest frequency of pixels for the grey value of 100–150 (connected with the grey value of the hydration products). Similar results were obtained for the CH (grey value 175–190). Fig. 13b presents the frequency of the pixels of the SEM images for the FA30 series samples. The samples cured according to the CC1 conditions show the highest amount of pores. The samples cured according to the CC3 conditions have the highest frequency of pixels in the grey value of 100–150 (related with the amount of hydration products in the microstructure). The frequency of pixels in the grey scale, which is associeted with the CH for the samples cured according to the CC2 and CC3 conditions, are similar. Moreover, it should be stated that the FA30 series shows a higher frequency of pixels in the grey scale (100–150). These pixels are directly associated with the hydration

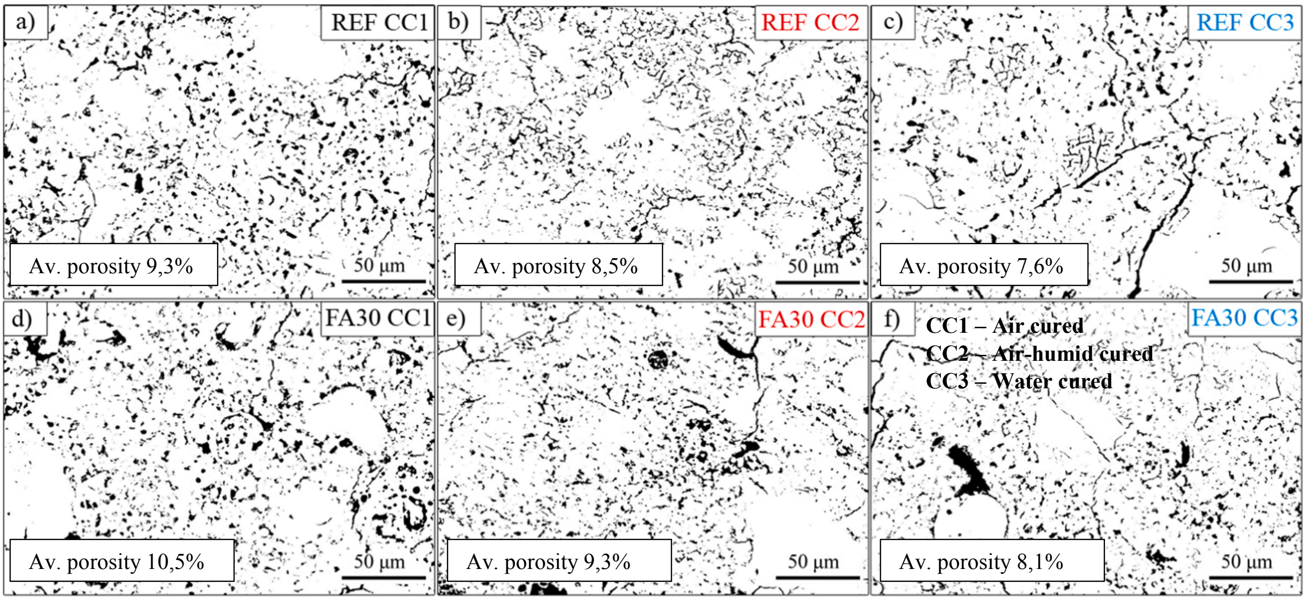

Fig. 11. Segmented pores (black pixels) of the samples after 90 days of curing: a) REF CC1, b) REF CC2, c) REF CC3, d) FA30 CC1, e) FA30 CC2, f) FA30 CC3.

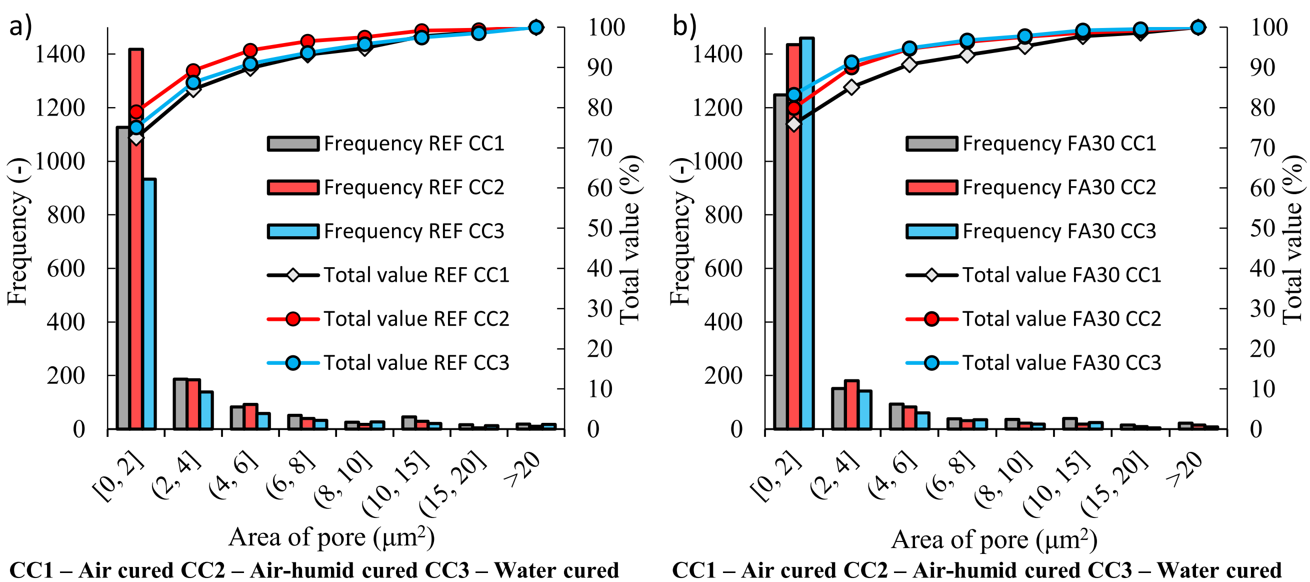

Fig. 12. Pore size distribution plots for the samples after 90 days of curing: a) REF samples, b) FA30 samples.

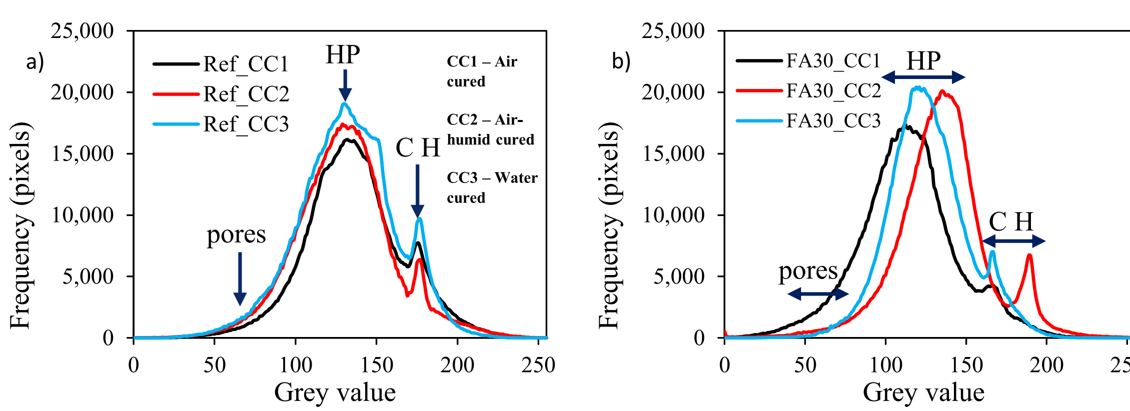

Fig. 13. Analysis of the SEM-based hydration process of the cementitious mortars cured in different conditions after 90 days of curing: a) REF series, b) FA30 series.

products.

Fig. 14 presents examples of the images obtained for the REF CC1 series from the performed analysis concerning the degree of hydration.

An estimation of the degree of hydration was performed according to Powers and Brownyard [59] using the average results obtained when analysing a minimum of 10 images of each sample. This approach is based on the assumption that the volume of the fractions of the hydration products (F_{hp}) is 2.1 times higher than the volume of the unhydrated grains (F_{up}):

$$\alpha = \frac{F_{hp}/2,1}{\frac{F_{hp}}{2,1} + F_{up}}(-) \tag{4}$$

The results presented in Fig. 15 confirm the effect of the curing conditions on the cementitious composites. As expected, the curing conditions had a significant impact on the hydration process. In the case of the samples investigated after 90 days of curing, a higher impact (when compared to the fly ash-modified series) of the curing conditions on the microstructure of the reference composites can also be observed. Thus, curing conditions are of particular importance for improving the microstructure of cementitious composites. The additional reactivity of the fly ash can also be observed, which is confirmed by the higher degree of hydration of the fly ash-modified samples when compared to the reference series. Significant differences in the microstructure of the composite are observed with regards to curing conditions. It can be observed that the samples cured according to the CC1 conditions have a higher amount of anhydrous cement when compared with the samples cured in the CC2 or CC3 curing conditions. The lowest amount of anhydrous cement was observed for the samples cured in the CC3 conditions. In turn, if the amount of anhydrous cement decreases, the amount of hydration products in the microstructure increases.

For a more precise comparison, an analysis of the relation between the hydration degree (α) and the mechanical properties of the samples was performed (Fig. 16).

Based on the performed analysis, it is possible to present a relation between the degree of hydration and the mechanical performance of the samples. It can be observed that the relationship is almost linear (especially for Fig. 16a in which $R^2 = 0.747$ (-)), which confirms that there is a relation between the SEM approximate hydration analysis and the measured mechanical properties. The samples cured according to the CC3 conditions show better mechanical properties, which is due to the higher amount of hydration products in their microstructure.

3.4. Analysis of the literature results

The topic of the effect of curing conditions on the mechanical properties of fly ash-modified mortars is known in literature. However, siliceous fly ash has rarely been studied. Nevertheless, a comparison of the results obtained in these studies with those obtained by other researchers is presented in Table 5.

When analysing the collected data and the authors'own results, it should be stated that the result of the development or regression of the compressive strength of fly ash-modified cementitious composites is dependent on the type, fineness, and chemical composition of the fly ash used (Table 5). Generally, all the presented results show the clear effects of curing conditions and the synergistic impact of using fly ash on the mechanical properties of composites (good development of strength after a long curing time). Therefore, the results reported herein are comparable with the results of other researchers in terms of mechanical properties. However, due to the lack of results concerning the influence of curing conditions on the microstructural properties of siliceous fly ash-modified composites in the literature, it is not possible to directly compare the results obtained in these studies with the results of other researchers.

4. Conclusions

In this research, cementitious mortars modified with the addition of siliceous fly ash cured in different conditions were investigated. Curing conditions are one of the most important factors affecting cementitious composites, as they have a direct impact on the development of the mechanical properties of mortars. The obtained results allow for the conclusion that curing conditions affect the physical, chemical, and microstructural properties of mortars.

Therefore, based on the results of research concerning mechanical properties, it can be stated that siliceous fly ash leads to the development of the "late" compressive and flexural strength in mortars that are used in cementitious floors. These strengths are connected with the durability of floors, and are highly desirable. The samples cured in water were characterized by the highest mechanical properties, however, the specimens cured according to the CC2 conditions (air-humid cured) also showed an enhanced development of strength when compared to the air-cured mortar samples. Therefore, one should strive to obtain air-humid curing conditions on a construction site (note that water curing is unrealistic for floors due to a structure only being accessible from one side - from the top).

A more in-depth analysis of curing conditions highlighted their impact on the physical, chemical, and microstructural properties of mortars. Curing conditions have an impact on the volume porosity and water absorptivity of mortars. The samples cured in the CC3 conditions had a lower volume porosity and water absorptivity when compared with the samples cured in the CC1 conditions. Moreover, it was shown that mechanical properties are related to the physical properties of mortars. The chemical compositions of the samples were dependent on the curing conditions. A greater synergistic effect of using siliceous fly ash can be observed in samples that are cured with a bigger amount of water. The impact of curing conditions on the proportions of SiO_2/CaO

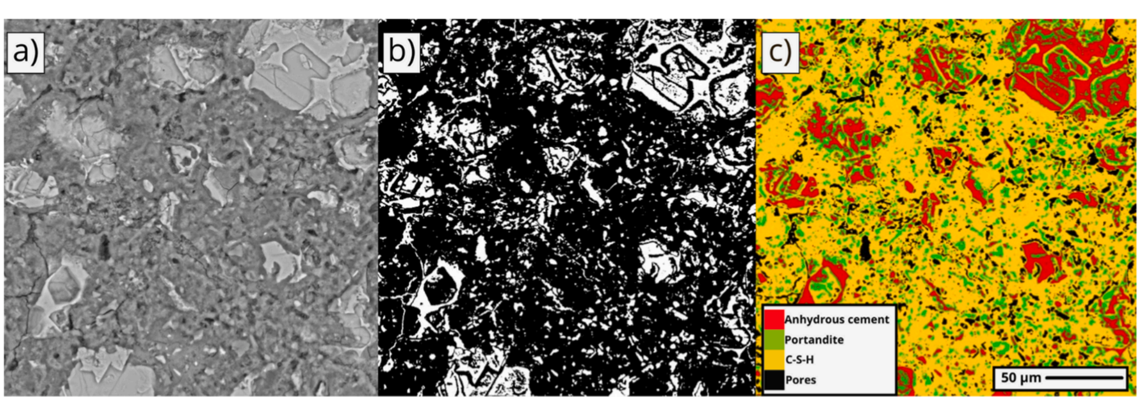

Fig. 14. Analysis of the hydration degree of the mortar specimens after 90 days of curing: a) BSE detector image, b) image after pore thresholding, c) image after phase segmentation.

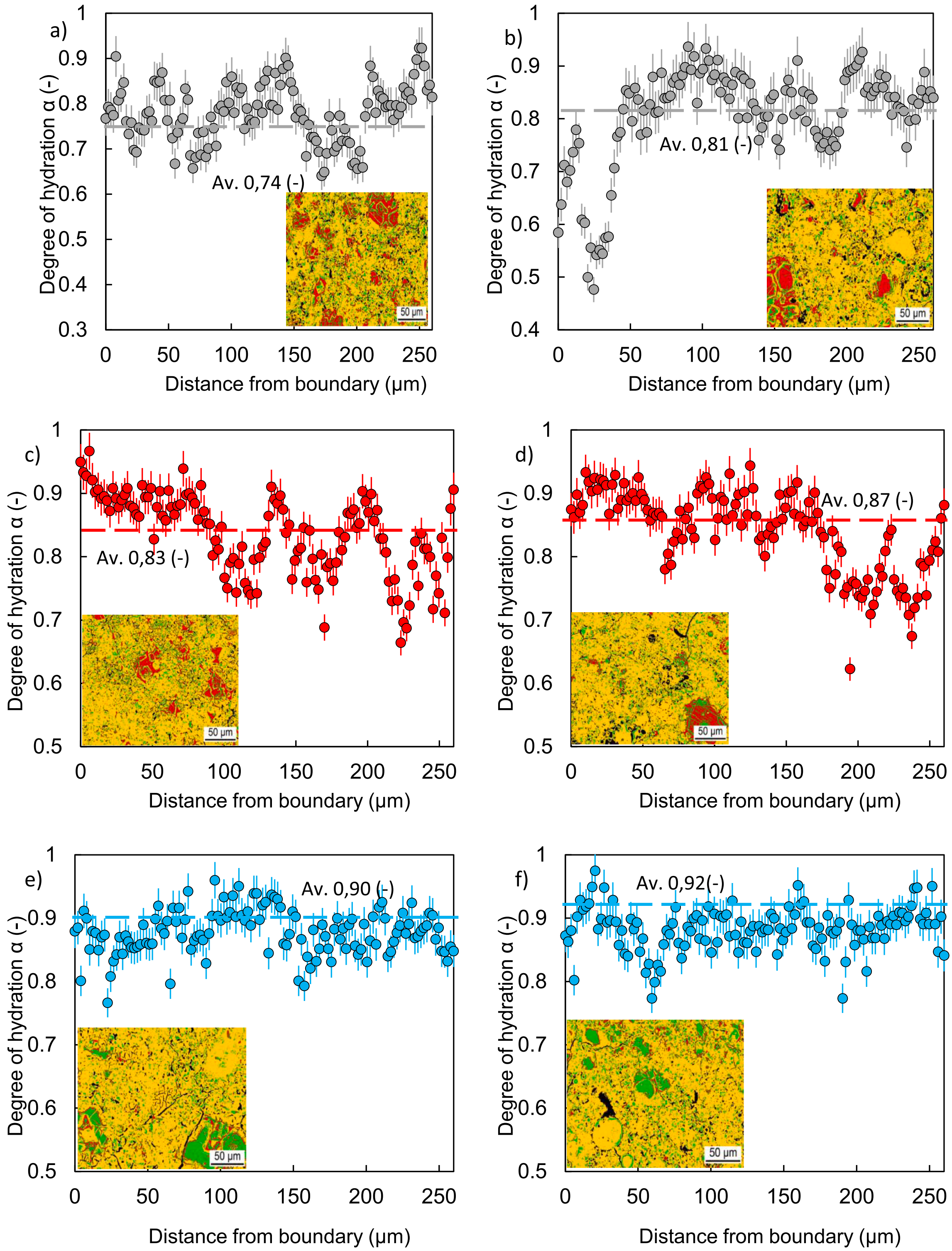

Fig. 15. Analysis of the hydration degree of the mortar specimens cured in different conditions after 90 days of curing: a) REF CC1, b) FA30 CC1, c) REF CC2, d) FA30 CC2, e) REF CC3, f) FA30 CC3.

(an increase of SiO_2 in the samples cured in a higher amount of water) was clearly observed. The authors refer this fact to the effect of the curing conditions. Moreover, it was observed that curing conditions have a direct impact on the microstructure of composites. The area of segmented pores is associated with the curing conditions. Moreover, the number of pores, hydration products (HP), and calcium hydroxide (CH) are related to the curing conditions.

To sum up, curing conditions have a direct impact on cementitious composites. They are especially important in floors due to the possibility of their durability and mechanical properties being increased. Very promising results were obtained for the mortars modified with the addition of siliceous fly ash, and therefore this cement replacement material should be used in cementitious mortars that are applied on floors. It should be in the interest of flooring contractors to provide adequate curing conditions of mortars. Ensuring the most humid curing conditions possible will increase the durability of cementitious floor

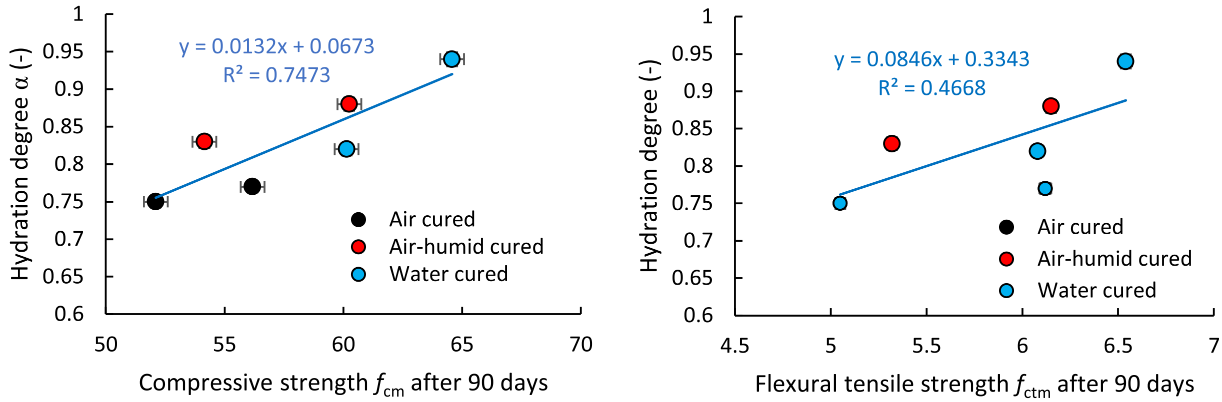

Fig. 16. Analysis of the connection between the hydration degree and the mechanical properties: a) compressive strength, b) flexural strength of the mortars cured in different conditions for 90 days.

Table 5
A comparison of the obtained results and the literature review in terms of the effect of different curing conditions on the compressive strength of fly ash-modified composites.

Reference	The scale of the tested composite	Chemical composition of the fly ash used in the research (wt.%)	Investigated after (days)	Compressive strength development or regression (%) Reference series		Fly ash – 20–40% mass of cement addition	
				Air cured	Curing conditions with the highest development of strength	Air cured	Curing conditions with the highest development of strength
This research	Mortars	CaO: 3,34 SiO_2: 54,33 Al_2O_3: 30,65 FeO: 4,78 MgO: 2,45 SO_3: 0,00K_2O: 2,51	28 90	100% 100%	122% 112%	94% 106%	108% 122%
C.S. Poon et al. [27]	Pastes	CaO: 5,69 SiO_2: 44,92 Al_2O_3: 35,39 FeO: 4,89 MgO: 1,29 SO_3: 0,71 K_2O: 0,64	28 90	100% 100%	137% 152%	67% 66%	112% 157%
Watcharapong et al. [60]	Mortars	CaO: 10,43 SiO_2: 45,37 Al_2O_3: 20,65 FeO: 12,31 MgO: 2,13 SO_3: 2,53 K_2O: 1,50	28 90	100% 100%	111% 118%	55% 52%	69% 81%
Termkhajornkit et al. [26]	Pastes	CaO: 1,30 SiO_2: 59,90 Al_2O_3:29,60 FeO: 4,80 MgO: 0,60 SO_3: nd K_2O: 0,70	28 90	100% 100%	128% 130%	78% 86%	87% 94%
Ogawa et al. [61]	Mortars	CaO: 2,41 SiO_2: 57,36 Al_2O_3: 28,68 FeO: 4,29 MgO: 1,28 SO_3: 0,35 K_2O: 0,83	28 90	100% 100%	117% 114%	73% 80%	129% 120%
Yang et al. [20]	Mortars	CaO: 5,20 SiO_2: 44,82 Al_2O_3: 31,69 FeO: 4,63 MgO: 0,66 SO_3: 1,50 K_2O: 1,34	28 56	100% 100%	105% 113%	77% 79%	105% 108%
Average:				100%	122%	76%	108%

overlays.

CRediT authorship contribution statement

Adrian Chajec: Conceptualization, Formal analysis, Investigation, Writing – original draft. **Agnieszka Chowaniec:** Formal analysis, Investigation, Writing – original draft. **Aleksandra Królicka:** Conceptualization, Methodology, Formal analysis, Investigation, Writing – original draft. **Łukasz Sadowski:** Writing – review & editing, Funding acquisition, Supervision. **Andrzej Żak:** Writing – review & editing, Funding acquisition. **Magdalena Piechowka-Mielnik:** Methodology. **Branko Šavija:** Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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